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
- ❒ 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
- ❒ Other ______________
- ❒ Consultant
- ❒ Educator
- ❒ Student

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	FAX	MAIL
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 this form to TMS Meeting Services **USA 724-776-3770** Fax registration requires credit card payment.	**Return this form with payment to** Meeting Services TMS 184 Thorn Hill Road Warrendale, PA 15086

TMS2002

131st Annual Meeting & Exhibition

AM02-PDF

Advance Registration Deadline: January 28, 2002

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: ________ COUNTRY ____ AREA/CITY ____ LOCAL NUMBER   Fax: ________ 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

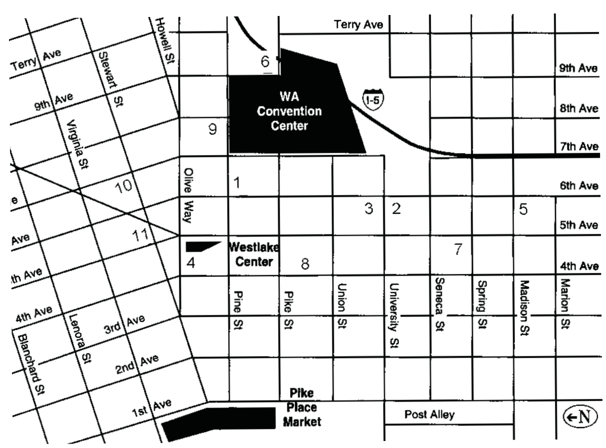
TMS has contracted a block of rooms at the headquarters hotel, Sheraton Seattle Hotel & Towers, along with each of the hotels, and therefore has assumed a financial liability for any and all rooms in that block that are not reserved. You are strongly encouraged to reserve your room(s) at the hotels listed 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 the 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 ______________________

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. ______________________

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

CALPHAD and Alloy Thermodynamics

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 Deformation: 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 Congress 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 Performance 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.

ISBN 0-87339-525-5

Approx. 600 pp., illus., index, hardcover

Order No. 5255

Member price: $70

	Monday-February 18 AM	Monday-February 18 PM	Tuesday-February 19 AM	Tuesday-February 19 PM	Wednesday-February 20 AM	Wednesday-February 20 PM	Thurs.-Feb. 21 AM
201	Computational Phase Transformations I	Computational Phase Transformations II	Computational Phase Transformations III	Computational Phase Transformations IV	Computational Phase Transformations V	Computational Phase Transformations VI	Computational Phase Transformations VII
203		Surface Engineering: Science & Technology - II I	Surface Engineering: Science & Technology - II II	Surface Engineering: Science & Technology - II III	Surface Engineering: Science & Technology - II IV	Surface Engineering: Science & Technology - II V	Surface Engineering: Science & Technology - II VI
204	Hume-Rothery Award Symposium: CALPHAD & Alloy Thermodynamics I	Hume-Rothery Award Symposium: CALPHAD & Alloy Thermodynamics II	Hume-Rothery Award Symposium: CALPHAD & Alloy Thermodynamics III	Hume-Rothery Award Symposium: CALPHAD & Alloy Thermodynamics IV			
205	Processing & Properties of Lightweight Cellular Metals & Structures The MPMD 3rd Global Symp. I	Processing & Properties of Lightweight Cellular Metals & Structures The MPMD 3rd Global Symp. II	Processing & Properties of Lightweight Cellular Metals & Structures The MPMD 3rd Global Symp. III	Processing & Properties of Lightweight Cellular Metals & Structures The MPMD 3rd Global Symp. IV	Processing & Properties of Lightweight Cellular Metals & Structures The MPMD 3rd Global Symp. V		
206				Fatigue and Creep of Metal Matrix Composites I	Fatigue and Creep of Metal Matrix Composites II	Fatigue and Creep of Metal Matrix Composites III	Fatigue and Creep of Metal Matrix Composites IV
208	David L. Davidson Symposium on Fatigue I	David L. Davidson Symposium on Fatigue II	David L. Davidson Symposium on Fatigue III	David L. Davidson Symposium on Fatigue IV			
209	Processing of Refractory Metals & Alloys	GA Nickel and Molybdenum	GA Composite Materials	Materials & Processes for Submicron Technologies - II I	Materials & Processes for Submicron Technologies - II II	Materials & Processes for Submicron Technologies - II III	
210	Second International Symposium on Ultrafine Grained Materials I	Second International Symposium on Ultrafine Grained Materials II	Second International Symposium on Ultrafine Grained Materials III	Second International Symposium on Ultrafine Grained Materials IV	Second International Symposium on Ultrafine Grained Materials V	Second International Symposium on Ultrafine Grained Materials VI	
211	GA Microstructural Phase Stability	GA Ferrous Materials: Carbon and Low Alloy Steels	GA Ferrous Materials: Alloy & Stainless Steels	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials I	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials II	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials III	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials IV
212	Advances in Metallic Glasses I	Advances in Metallic Glasses II	Advances in Metallic Glasses III	Advances in Metallic Glasses IV	Advances in Metallic Glasses V	Advances in Metallic Glasses VI	
213	High Performance Metallic Materials for Cost Sensitive Applications I	High Performance Metallic Materials for Cost Sensitive Applications II	High Performance Metallic Materials for Cost Sensitive Applications III	High Performance Metallic Materials for Cost Sensitive Applications IV	High Performance Metallic Materials for Cost Sensitive Applications V	High Performance Metallic Materials for Cost Sensitive Applications VI	
214	Creep Deformation Fundamentals and Applications I	Creep Deformation Fundamentals and Applications II	Creep Deformation Fundamentals and Applications III	Creep Deformation Fundamentals and Applications IV	Creep Deformation Fundamentals and Applications V	Creep Deformation Fundamentals and Applications VI	

Monday-February 18		Tuesday-February 19		Wednesday-February 20		Thurs.-Feb. 21	
AM	PM	AM	PM	AM	PM	AM	
	Shear Banding in Materials I	Shear Banding in Materials II	Deformation and Stresses in Small Volumes I	Deformation and Stresses in Small Volumes II	Deformation and Stresses in Small Volumes III	Deformation and Stresses in Small Volumes IV	303
		Modelling of Multi-scale Phenomena in Materials Processing I	Modelling of Multi-scale Phenomena in Materials Processing II	Modelling of Multi-scale Phenomena in Materials Processing III	Modelling of Multi-scale Phenomena in Materials Processing IV		304
	Water Vapor Effects on Oxidation of High-Temperature Materials I	Water Vapor Effects on Oxidation of High-Temperature Materials II	Water Vapor Effects on Oxidation of High-Temperature Materials III	Water Vapor Effects on Oxidation of High-Temperature Materials IV			305
	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium I	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium II	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium III	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium IV	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium V	Charles J. McMahon Interfacial Segregation and Embrittlement Symposium VI	307-308
GA Joining, Testing and Characterization	GA Al and Ti	Imaging of Dynamic Processes I	Imaging of Dynamic Processes II	Modelling of High Temperature Alloy Processing			310
		Commercial Mini-Sessions I	Commercial Mini-Sessions II	Commercial Mini-Sessions III	Commercial Mini-Sessions IV		401
Cast Shop Technology I	Cast Shop Technology II	Cast Shop Technology III	Cast Shop Technology V	Cast Shop Technology VI	Cast Shop Technology VII	Cast Shop Technology VIII	6A
Aluminum Reduction Technology I	Aluminum Reduction Technology II	Aluminum Reduction Technology III	Aluminum Reduction Technology IV	Aluminum ReductionTechnology V/ Carbon Technology	Aluminum Reduction Technology VI	Aluminum Reduction Technology VII	6B
General Pyrometallurgy I		GA Extractive Metallurgy	Teaching & Learning Hydrometallurgical Science & Engineering I	Teaching & Learning Hydrometallurgical Science & Engineering II	Teaching & Learning Hydrometallurgical Science & Engineering III	Teaching & Learning Hydrometallurgical Science & Engineering IV	601
Energy Issues in the Aluminum Industry – A Keynote Symposium	Carbon Technology I	Carbon Technology II	Carbon Technology III	(Room 6A) Carbon Technology/ Aluminum ReductionTechnology V			602-603
Flyash:Generation, Treatment, Metal Recovery and Disposal I	Flyash:Generation, Treatment, Metal Recovery and Disposal II	Recycling - General Sessions I	Recycling - General Sessions II	General Topics in Waste Treatment & Minimization I	General Topics in Waste Treatment & Minimization II		604
Magnesium Technology 2002 I	Magnesium Technology 2002 II	Magnesium Technology 2002 III	Magnesium Technology 2002 IV	Magnesium Technology 2002 V	Magnesium Technology 2002 VI	Magnesium Technology 2002 VII	606

	Monday-February 18 AM	Monday-February 18 PM	Tuesday-February 19 AM	Tuesday-February 19 PM	Wednesday-February 20 AM	Wednesday-February 20 PM	Thurs.-Feb. 21 AM
607	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" I	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" II	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" III	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" IV	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" V	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" VI	
608			Cast Shop IV		Aluminum Sheet & Plate Rolling & Finishing Technology & Applications I	Aluminum Sheet & Plate Rolling & Finishing Technology & Applications II	Aluminum Sheet & Plate Rolling & Finishing Technology & Applications III
609	Alumina & Bauxite I	Alumina & Bauxite II	Alumina & Bauxite III	Alumina & Bauxite IV	Alumina & Bauxite V	Third International Sulfide Smelting Symposium: "Sulfide Smelting '02" VII	
611		Automotive Alloys I	Automotive Alloys II	Automotive Alloys III		Materials Issues Related to the Collapse of the World Trade Center	
612		Lead-Free Solders and Materials Issues in Microelectronic Packaging I	Lead-Free Solders and Materials Issues in Microelectronic Packaging II	Lead-Free Solders and Materials Issues in Microelectronic Packaging III	Lead-Free Solders and Materials Issues in Microelectronic Packaging IV	Lead-Free Solders and Materials Issues in Microelectronic Packaging V	
613	Fundamentals of Advanced Materials for Energy Conversion I	Fundamentals of Advanced Materials for Energy Conversion II	Fundamentals of Advanced Materials for Energy Conversion III	Fundamentals of Advanced Materials for Energy Conversion IV	Fundamentals of Advanced Materials for Energy Conversion V	Fundamentals of Advanced Materials for Energy Conversion VI	Fundamentals of Advanced Materials for Energy Conversion VII
614	Advances in Molten Salt Processing Technology I	Advances in Molten Salt Processing Technology II	Materials Processing Fundamentals I	Materials Processing Fundamentals II	Materials Processing Fundamentals III	Materials Processing Fundamentals IV	Fundamentals of Advanced Materials for Energy Conversion VIII
615-616	Fundamentals of Structural Intermetallics I	Fundamentals of Structural Intermetallics II	Fundamentals of Structural Intermetallics III	Fundamentals of Structural Intermetallics IV	Fundamentals of Structural Intermetallics V	Fundamentals of Structural Intermetallics VI	Fundamentals of Structural Intermetallics VII
617	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath I	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath II	InternationalSymposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath III	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath IV	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath V	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath VI	International Symposium on Science & Technology of Interfaces in Honor of Dr. Bhakta Rath VII
619-620	Computational Modeling of Matls, Mins and Metals Proc Plenary	Computational Modeling of Matls, Mins and Metals Proc Plenary	Computational Modeling of Matls, Mins and Metals Proc Plenary	Computational Modeling of Matls, Mins and Metals Proc Plenary	Computational Modeling of Matls, Mins and Metals Proc Plenary	Computational Modeling of Matls, Mins and Metals Proc Plenary	
619	Computational Modeling of Matls, Mins and Metals Proc Track A	Computational Modeling of Matls, Mins and Metals Proc Track A	Computational Modeling of Matls, Mins and Metals Proc Track A	Computational Modeling of Matls, Mins and Metals Proc Track A	Computational Modeling of Matls, Mins and Metals Proc Track A	Computational Modeling of Matls, Mins and Metals Proc	
620	Computational Modeling of Matls, Mins and Metals Proc Track B	Computational Modeling of Matls, Mins and Metals Proc Track B	Computational Modeling of Matls, Mins and Metals Proc Track B	Computational Modeling of Matls, Mins and Metals Proc Track B	Computational Modeling of Matls, Mins and Metals Proc Track B		

Table of Contents

Washington State Convention & Trade Center ● Seattle, Washington USA ● February 18-21, 2002

Advances in Metallic Glasses: Glass Formation

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

Monday AM Room: 212
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: A. L. Greer, Cambridge University, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; Matthew J. Kramer, Iowa State University, 37 Wilhelm Hall, Ames, IA 50011-3020 USA

8:30 AM Invited
Formation of (Fe,Ni)-Based and Al-Based Structural Amorphous Metals: *S. Joseph Poon*[1]; Gary J. Shiflet[2]; Faqiang Guo[1]; V. Ponnambalam[1]; [1]University of Virginia, Phys., Charlottesville, VA 22904 USA; [2]University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22903 USA

This talk will focus on the formability, stability, and some of the physical properties of metallic glasses in several ferrous-based and light-metal-based multinary alloy systems. Ferrous-based alloys will include those that contain Fe, Ni, Mo, and Cr; light-metal alloys will contain Al and Mg as the major components. The ferrous-based metallic glasses, which are non-ferromagnetic at ambient temperatures, can be compared with several types of austenitic steels. Magnetization and susceptibility results will be presented. The formability factors for forming bulk metallic glasses will be discussed in light of experiment and atomistic model.

9:00 AM
Multicomponent (Ti Zr Hf Nb)-(Ni Cu Ag)-Al Glasses: Ki-Buem Kim[1]; Paul Warren[1]; *Brian Cantor*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

A wide range of new metallic glasses has been developed in the (Ti Zr Hf Nb)-(Ni Cu Ag)-Al system by a novel equiatomic substitution technique for the early and late transition metal components, starting from the well known Zr-Cu-Al system. The thermal stability and crystallisation of these glasses has been studied by x-ray diffractometry, differential scanning calorimetry, transmission electron microscopy and atom probe analysis. This paper describes the resulting insight into the behaviour of these novel multicomponent metallic glasses.

9:20 AM
A Topological Model for Metallic Glass Formation: *Oleg N. Senkov*[1]; Daniel B. Miracle[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

An approach based on analysis of atomic size distributions has been developed and applied to multicomponent amorphous alloys with different glass forming ability. The atomic size distributions were obtained by plotting atomic concentrations versus atomic radii of constitutive elements. Amorphous alloys with high critical cooling rates were found to have single-peak distributions with a concave downward shape. These amorphous systems have at least one alloying element with a smaller radius and at least one alloying element with a larger radius relative to the base element. The concentration of an alloying element decreases rapidly as the difference in the atomic sizes of the base element and the alloying element increases. Atomic size distributions of Zr, Pd, or RE -based bulk amorphous alloys, which have low critical cooling rates, have a completely different, concave upward shape with a minimum concentration at an intermediate atomic size. A topological model that explains the concave upward shape of atomic size distributions for the bulk amorphous alloys has been suggested.

9:40 AM
Atomistic Simulations of the Phase Stability and Elastic Properties of the Nickel-Zirconium Alloy System: *Frank J. Cherne*[1]; Michael I. Baskes[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, MST-8, PO Box 1663, MS G755, Los Alamos, NM 87545 USA

Numerous simulations of binary amorphous Zr-Ni alloys have been performed utilizing the potential formalism of Massobrio, Pontikis, and Martin (MPM). Here we present a thorough study of the phase stability and the elastic properties of both the amorphous and the crystalline nickel-zirconium alloys utilizing the modified embedded-atom method (MEAM) approach. We will compare and contrast MEAM with MPM. The MEAM potentials developed predict the correct crystal structures for NiZr and $NiZr_2$. The nickel-zirconium system was chosen primarily due to the complexity of the phase diagram and its glass forming ability. Furthermore, we are interested in examining the compositional dependence of the shear modulus. We will also present the free energies as a function of composition and temperature for both amorphous and crystalline phases.

10:00 AM
Stability of Undercooled $Pd_{40}Ni_{40-x}Cu_xP_{20}$ Liquids: *T. D. Shen*[1]; Ulrich Harms[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MS G755, Los Alamos, NM 87545 USA

The question of what determines the stability of undercooled liquids remains unanswered. In the $Pd_{40}Ni_{40-x}Cu_xP_{20}$ system, the best bulk glasses are found for x=0 and x=30. Properties of the glass, such as shear modulus, hardness, and width of the first maximum in the X-ray diffraction pattern, show extreme values at approximately x=10, which we attribute to strong short-range ordering at that composition. We have found that the stability of the undercooled liquids correlates best with the difference ΔV=Vg-Vc between the molar volumes of the glassy and crystalline states. For 0<x<30, ΔV is positive, whereas for 30<x<40, ΔV is negative. Both positive and negative values of ΔV decrease Tx-Tg. The minimal in |ΔV| at x=0 and x=30 correspond to the largest values of Tx-Tg. A positive ΔV implies a negative pressure around growing crystalline embryos and this could explain the observed decrease in Tx-Tg for x<30.

10:20 AM Break

10:40 AM Invited
Atom Probe Studies of Metallic Glasses: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The distribution of the atoms in bulk metallic glasses in both the as-prepared condition and after heat treatments in the region of the glass transition temperature provides important information on the stability of the alloy. The spatial coordinates and the elemental identities of the atoms in the amorphous alloy may be determined with near atomic resolution by atom probe tomography. The three-dimensional data collected by this technique can be analyzed to determine the local atomic configuration of each atom type and thereby estimate the tendency for solute clustering and chemical short range order. These data can be also divided into small volume elements that may be statistically analyzed to detect and quantify concentration fluctuations due to phase separation, precipitation and crystallization. A review of the decomposition of a number of different types of bulk metallic glasses will be presented. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:10 AM
EFTEM Study of Compositional Variations in Mg-Ni-Nd Bulk Metallic Glasses: *S. V. Madge*[1]; D. T.L. Alexander[1]; A. L. Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A range of Mg-Ni-Nd bulk metallic glasses has been cast. Around the composition $Mg_{60.5}Ni_{24}Nd_{15.5}$ (at.%), but not at neighbouring compositions, energy-filtered TEM provides evidence for strong compositional inhomogeneities in the as-cast glass. In this fully glassy material, an apparent two-phase cellular microstructure is seen; one phase is rich in Ni, the other in Mg and Nd. Small-angle X-ray scattering offers additional evidence for phase separation in the glassy state. Annealing can eliminate the inhomogeneities, and possible explanations for the phenomena are considered, including the role of oxygen.

11:30 AM
Nanoscale Phase Separation and Local Icosahedral Order in Amorphous Alloys of Immiscible Elements: *Evan Ma*[1]; [1]Johns Hopkins University, Matls. Sci. & Eng., Baltimore, MD 21218 USA

We explore the nature of amorphous alloys formed by nonequilibrium processing in immiscible systems, to uncover their differences from those well-studied metallic glasses in systems with negative heat of mixing. We demonstrate spinodal compositional modulations with nanometer wavelengths, along with local icosahedral ordering, in amorphous structures formed in the immiscible Ag-Ni system. This unique atomic-level structure allows the amorphous alloy to lower its enthalpy such that the heat of crystallization is of a magnitude comparable to those typically seen for metallic glasses in easy glass forming systems. The development of local structure has been monitored using EXAFS[1], TEM, XRD, and magnetic measurements, as well as MD simulations[2,3], and correlated with the enthalpy states measured using DSC. [1]Phys. Rev. Lett. 83, 2826 (2001). [2]Appl. Phys. Lett. 78, 1343 (2001). [3]Phys. Rev. B Oct. 1 issue (2001).

Advances in Molten Salt Processing Technology: New Electrochemical Methods

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439-4837 USA

Monday AM	Room: 614
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div., Argonne, IL 60439 USA

8:30 AM Opening Remarks

8:35 AM
New Advances in Molten Salt Research: Data for Pyrochemical Reprocessing of Nuclear Fuels: *Marcelle Gaune-Escard*[1]; [1]Universite de Provence, CNRS UMR 6595, IUSTI, Technopole Chateau-Gombert, 5 rue Enrico Fermi, Marseille 13453 France

Lanthanide and actinides halides and their mixtures with alkali halides play a major role in the processing of nuclear waste and recycling of spent nuclear fuel. However, reference data are missing both for the pure salts and the mixtures, whatever the halides (chlorides or fluorides) under consideration. However the related data on molten lanthanide and actinide salts (and compounds), are not easily available. Because of characteristic physicochemical properties, which do not promote experimental investigations, data are scarce. When they exist, most of the data is generally buried in grey literature. Also, when available, they reveal to arise from estimations. Coordination and standardization of existing data is therefore essential, as research efforts should be intensified together with the development of numerical prediction tools. This paper will, firstly, discuss new experimental data on lanthanide- and actinide-based halide systems. These are obtained through an intensive international research cooperation and multiinstrumental methods. It will be shown that data can be obtained not only from these physicochemical measurements (thermodynamic, electrical, electrochemical, structural), but also from numerical simulations based on structural features and proper modeling. Secondly, ongoing efforts for the development of a molten salt database will be presented.

9:05 AM
Direct Electrochemical Reduction of Niobium Pentoxide to Niobium Metal in a Eutectic of $CaCl_2$-NaCl Melt: Xiao Yong Yan[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A novel electrochemical approach is being developed using the method of the electrodeoxidation to reduce solid Nb_2O_5 directly to niobium metal in a eutectic melt of $CaCl_2$-NaCl at temperatures around 900°C. Electrolysis was carried out at the applied constant voltage of 3.1 V, below the decomposition potentials of the chloride melt. After electrolysis, the oxide cathode, pellets of Nb_2O_5, transformed into porous pellets of metallic niobium that could be ground manually into niobium powders with a wide range of niobium particle sizes. In addition to the niobium powders, the niobium sponge has a similar structure to that of Kroll titanium sponge and was readily prepared by controlling the conditions for pellet-making and electrolysis. The niobium metal prepared under the present experimental conditions contained as low as 3000 mass ppm oxygen. The results obtained clearly demonstrated that this direct solid-state electrochemical reduction of Nb_2O_5 is an efficient, low cost, and environmentally friendly process compared with the existing conventional reduction processes, and it can be potentially an electrolytic reduction process for the production of niobium metal.

9:35 AM
Electrochemical Reduction of Metal Oxides in Molten Salts: *Karthick V. Gourishankar*[1]; Laszlo Redey[1]; Mark A. Williamson[1]; [1]Argonne National Laboratory, Cheml. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

Argonne National Laboratory (ANL) has demonstrated a direct electrochemical reduction process for converting uranium oxide to uranium metal. The reduction process is versatile and can be extended to the extraction of other actinide and rare-earth metals from their oxides. Although UO2 reduction has been demonstrated in molten LiCl-Li2O electrolyte at 650ºC, the choice of electrolyte and process temperature depends on the oxide charge. For example, the reduction of rare-earth oxides requires CaCl2-CaO melts at 800-900ºC. However, the fundamental mechanism underlying the technology is the same. The oxygen in the oxide charge is ionized into a soluble species at the cathode, leaving behind the reduced metal, and the oxide ion is transported to the anode, where it is converted to oxygen gas. The electrochemical cell consists of a non-consumable anode and a cathode that is configured to contain the oxide charge. Independent reference electrodes are used to monitor the anode and cathode potentials. The cell is operated under constant current or constant potential conditions. This paper will discuss the cell design and operation, and will present results from the UO2 reduction experiments.

10:05 AM Break

10:20 AM
Synthesis of Pb-Ca Alloys by Electrolysis of CaO Solution in Molten Salt: *Derek J. Fray*[1]; Jane Freidina[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

In the search for an environmentally friendly, efficient technology for calcium addition to lead alloys a study of calcium electrolysis from molten salts was performed. The research showed that calcium can be electrowon to a liquid lead cathode at temperatures below 923 K. Alloys containing up to 2 wt.% Ca were synthesised from CaCl2-NaCl-CaO mixtures with a current efficiency of 40-60%. Sodium impurity content was as low as one tenth of the calcium content. The effects if factors, such as temperature, current density, time of electrolysis on the performance of the cell and the quality of the alloys were investigated.

10:50 AM
Development of High Throughput Uranium Electrorefining: *James L. Willit*[1]; Eddie C. Gay[1]; [1]Argonne National Laboratory, Cheml. Tech., 9700 S. Cass Ave., Argonne, IL 60439-4837 USA

At the heart of Argonne's electrometallurgical treatment technology for spent metallic reactor fuel is an electrorefining step in which chopped spent fuel is loaded into a basket, and submerged in a pool of molten salt. A current passed between the basket (anode) and a cathode oxidizes the uranium and all active metal fission products forming soluble metal chlorides. At the cathode, uranium, the most noble of the dissolved metal chlorides, is electrodeposited. This basic concept has been scaled up from the laboratory scale to a device that holds ~100 kg of chopped fuel. The presentation will consist of a brief review of the basic electrochemistry of uranium electrorefining as well as the engineering challenges encountered during the scale-up of the technology.

11:20 AM
High-Temperature Electroforming in Molten Salts: Joseph Biondo[1]; *Anatoliy Shchetkovskiy*[1]; Alexander Smirnov[1]; Vitaly Sikin[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

Electroforming is a viable process for manufacturing near-net shapes of product. A number of metals can not be electrodeposited from water solution, but can successfully be obtained with electrolysis of the molten salts. Electroforming such metals as iridium, rhenium, niobium and platinum-iridium alloy is used by Engelhard-CLAL for commercial application under the name EL-Form™ process. High-temperature electroforming is based on electrodeposition of metals onto mandrel of required shape. After electrodeposition, the mandrel is removed.

The electroforming in molten salts provides seamless design of items, efficient metal use and metal purification during electrolysis. High uniformity and tight tolerances can be achieved especially for axisymmetrycal items such as tubes, crucibles and nozzles. Advanced production equipment allows the electroforming of items with a maximum diameter of 8.5" and maximum length of 15". The necessary process control provides stable and reproducible properties of the electroformed metals. Various examples of electroformed product will be discussed.

Alumina and Bauxite: Bauxite Digestion

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

Monday AM — Room: 609
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Jacques Mordini, Aluminium Pechiney, Direction Industrielle, Boite Postale 54, Gardanne Cedex 13541 France; Steven P. Rosenberg, Worsley Alumina Pty Ltd., Proc. Chem. Grp., PO Box 344, Collie, WA 6225 Australia

8:30 AM Introductory Remarks

8:40 AM
Improved Preparation of the Greek Diasporic Bauxite for Bayer-Process: *Dimitris Papanastassiou*[1]; Barnabás Csõke[2]; Károly Solymár[3]; [1]Silver&Baryte Ores Mining Co. S.A., Bauxite Div., 21 Amerikis str., Athens GR-10 672 Greece; [2]University of Miskolc, Dept. of Proc. Eng., Miskolc-Egyetemváros, Miskolc H-3515 Hungary; [3]ALUTERV, Ltd., Chief Techl. Adviser, Fehérvári út 144., Budapest H-1116 Hungary

The Greek diasporic bauxite plays an important role in the medium- and long-term bauxite supply of Europe. The chemical and mineralogical composition and the textural and physical properties of the high-grade, but hard and by calcite contaminated bauxite of S&B will be presented. The grindability lab tests and the industrial operation of AdG confirmed the important role of the lime dose as a grinding aid in hot caustic liquor, which can reduce the energy requirement of the mill by about 50% in comparison with the dry grinding. The calcite removal has been solved at S&B by "Dynamic Heavy-Media Separation". Plant-scale abrasion tests carried out in tube-in-shell preheaters of the tube digester at MOTIM Works Hungary did not indicate any measurable abrasion. So, the hard Greek diasporic bauxite can effectively and economically be prepared.

9:05 AM
Digestion Alternatives of the Greek Diasporic Bauxite: *Károly Solymár*[1]; Tibor Ferenczi[2]; Dimitris Papanastassiou[3]; [1]ALUTERV, Ltd., Chief Techl. Adviser, Fehérvári út 144., Budapest H-1116 Hungary; [2]ALU-LAB, Ltd., Managing Dir., Fehérvári út 144., Budapest H-1116 Hungary; [3]Silver&Baryte Ores Mining Co. S.A., Bauxite Div., 21 A, Amerikis str., Athens GR-10 672 Greece

Optimum digestion parameters (lime dosage, retention time and final A/C) of the S&B diasporic bauxite have been determined by lab simulation tests between 240 and 260°C. Co-grinding of bauxite and lime and lime addition to the slurry during predesilication resulted in similar advantageous effect on alumina yield and kinetics. Lime feeding at the final digestion temperature was found as the most effective. The required lime dose depends on the quantity and stability of the diaspore in the process bauxite and the digestion temperature. The results are in good agreement with the AdG practice and operational experiences of other European alumina refineries, achieved by processing S&B bauxite. The tests confirmed that high alumina yield and adequate cycle efficiency(high A/C ratio in blow-off liquor) can be achieved by well selected digestion parameters at 240°C already.

9:30 AM
Intensifying Method of Bayer Digestion Process of Diasporic Bauxite: *Yin Zhonglin*[1]; Gu Songqing[1]; [1]Zhengzhou Light Metal Research Institute, Shangjie, Zhengzhou, Henan 450041 China

The indirect preheating process of bauxite slurry is necessary for intensifying the Bayer digestion of diasporic bauxite. However, scale formation on indirect heating surfaces is unavoidable in single-stream digestion technology. In this paper a dual-stream and lime Bayer process is proposed, that combines the new dual-stream digestion process with the lime Bayer digestion process. The proposed process solves a series of key problems associated with indirect preheating technology and intensifies the digestion of diasporic bauxite. As a result, soda consumption can be reduced and the digestion process accelerated, allowing advanced technological and economical indexes to be obtained.

9:55 AM Break

10:15 AM
New Process Technologies of Bauxite Concentrate-Tube Digestion for Alumina Production: *Wangxing Li*[1]; *Shaolun Wang*[1]; Peikai Song[1]; Yongxing Hou[1]; [1]China Great Wall Aluminium, Tech. Dept., No 28, Changqian Rd., Shangjie Dist., Zhengzhou, Henan 450041 China

This paper provides a brief description of the bauxite reserves and status of alumina production in China. It then gives the results of studies carried out to solve the key technical problems involved in the processing of diasporic bauxite. A new process of diasporic bauxite concentrate preparation associated with tube digestion is presented that strides over the existing technology in terms of better bauxite usage, and lower energy, raw material and equipment requirements.

10:40 AM
Digestion of Diasporic Bauxite with Mass Ratio of Al_2O_3/SiO_2 No Greater than 7 by Bayer Process with an Excessive Addition of Lime: *Zhao Hengqin*[1]; Hu Hongjie[1]; Jin Mei[1]; Li Jie[2]; Li Qingyu[2]; Liu Yexiang[2]; [1]National Engineering Research Center for Multipurpose Utilization of Non-Metallic Mineral Resource, Metall. & Cheml. Eng., No. 328 Longhai W. Rd., Zhengzhou, Henan 450006 China; [2]Central South University, Dept. of Metlgcl. Sci. & Eng., Changsha, Hunan 410083 China

It is usually believed that digestion of the diasporic bauxite with mass ratio of Al_2O_3/SiO_2 no greater than 7, which constitutes over 70 percent of China proved bauxite reserves, by adopting ordinary Bayer Process, is uneconomically viable because of consumption of higher caustic soda. At higher digestive temperature simultaneously with a more addition of inexpensive lime than ordinary Bayer Process, the bauxite can be economically digested by Bayer Process. Leaching rate of alumina from the bauxites can reach over 85% after 60 minutes of digestion at 280°, with Na_2O_k 220g/l of a sodium aluminate liquor and with lime addition of over 16%wt. of bauxite. The content of Na_2O in the produced red mud is under 2.5%wt. The XRD analysis results of the red mud indicate that an excessive addition of lime causes big changes of constituents of red mud. The improved Bayer Process is of significance for exploitation of China bauxite.

11:05 AM
Thermal Behaviors of Kaolinite-Diasporic Bauxite and Desilication from it by Roasting-Alkali Leaching Processing: *Tao Jiang*[1]; Guanghui Li[2]; Zhucheng Huang[2]; Xiaohui Fan[2]; Guanzhou Qiu[2]; [1]University of Utah, Dept. of Metlgcl. Eng., Rm. 412, WBB, 135 S. 1460 East, Salt Lake City, UT 84112 USA; [2]Central South University, Dept. of Minl. Eng., Changsha 410083 China

By means of XRD, TG and DTA techniques, the thermal behaviors of kaolinite-diasporic bauxite are investigated in this paper. Results show that the kaolinite in the bauxite loses its constitutional water and turns into meta-kaolinite when temperature is elevated above 450°C. The meta-kaolinite is decomposed into amorphous silica and non-crystal alumina in the range of 990-1100°C, but latter two compose into mullite if the temperature exceeds 1100°C. It is found that diaspore is changed into a-alumina with the removal of its constitutional water at the temperature over 650°C. A desilication process of roasting-alkali leaching for the bauxite is developed on the basis of the results. The optimum roasting parameters are attained to be a grain size of 0~20mm, a roasting temperature of 1050~1100°C, and a roasting time of 15~20 minutes. The proper leaching conditions are found as 100 g/l Na2Ok and 140°C. By alkali leaching of the roasted bauxite, a concentrate of 9.92 A/S ratio is achieved for a raw bauxite ore with a 4.4 A/S ratio. It is found the silica removed by the roasting-alkali leaching processing is mainly the amorphous silica and the reaction of mullitization causes reduction in desilication. The a-alumina yielded by diaspore decomposition during roasting is not dissolved under the alkali leaching conditions. The Bayer leaching tests of the concentrate show that the desilication by roasting-alkali leaching processing improves the digestion properties of Al2O3 in the bauxite.

Aluminum Reduction Technology: Fundamentals

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

Monday AM	Room: 6B
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Geir Martin Haarberg, Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Sem Saelands vei 6, Trondheim NO-7491 Norway

8:30 AM
The Role of Dissolved Metal during Electrodeposition of Aluminium from Cryolite-Alumina Melts: *Geir Martin Haarberg*[1]; Jomar Thonstad[1]; Stanislaw Pietrzyk[2]; James J. Egan[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Sem Saelands vei 6, Trondheim NO-7491 Norway; [2]University of Mining and Metallurgy, Dept. of Metall. of Non-Ferrous Metals, Krakow 30-059 Poland; [3]Brookhaven National Laboratory, Upton, NY 11973 USA

In molten cryolite in contact with aluminium dissolved aluminium as well as dissolved sodium are formed. Dissolution of sodium gives rise to the formation of mobile electrons. Ac resistance measurements were performed to determine the electronic conductivity in cryolite-alumina melts as a function of temperature, electrolyte composition (AlF_3) and additives (CaF_2, LiF, MgF_2). Model calculations were performed to estimate the effect of electronic conductivity on the current efficiency for aluminium deposition. The behaviour of so-called metal fog generated at the cathode during deposition of aluminium was also investigated.

8:55 AM
Effect of the Bubble Growth Mechanism on the Spectrum of Voltage Fluctuations in the Reduction Cell: *Laszlo Istvan Kiss*[1]; Sandor Poncsak[1]; [1]UQAC Université du Québec à Chicoutimi, Dépt. des Sciences Appliquées, 555 Blvd. de l'Université', Chicoutimi, Quebec G7H 2B1 Canada

The spectrum of the voltage oscillations in the aluminum reduction cell depends on the size of the anode. Small laboratory cells and industrial size cells exhibit different fluctuation patterns. The growth of bubbles can be divided into two periods controlled by different physical mechanisms. The detachment frequency of the bubbles from the nucleation sites depends on the current density and detachment size, the latter being influenced by the material, microstructure and geometry of the anode. After detachment the main factor causing growth of the traveling bubbles is coalescence. A mathematical model that keeps track of each and every individual gas bubbles generated under the anode was developed and used to analyze the character of the fluctuations of the cell voltage. It is shown that in the case of industrial size anodes the voltage oscillations are dominated by the dynamics of the bubble interactions (coalescence) in the two-phase layer, while in small size laboratory systems the frequency of nucleation of the individual bubbles can be observed.

9:20 AM
Crystallization of Cryolite and Alumina at the Metal-Bath Interface in Aluminium Reduction Cells: *Asbjorn Solheim*[1]; [1]SINTEF, Matls. Tech., NO-7465 Trondheim, Norway

It is a well-known fact that the electrolyte (bath) facing the cathode in aluminium electrolysis cells contains less aluminium fluoride than the bulk, due to the diffusion processes taking place in the boundary layer close to the cathode. A lower AlF3 concentration gives a higher liquidus temperature. Under certain conditions, the local liquidus temperature at the cathode may become equal to the bath temperature, which provokes crystallization of solid cryolite and/or alumina. The present paper presents the relevant theory for such crystallization, as well as some calculations based on a numerical solution of the Stefan-Maxwell equation. The results show that formation of solid cryolite at the cathode may take place during normal cell operation. The rate increases with increasing AlF3 content in the bath, increasing cathodic current density, decreasing superheat, and decreasing mass transfer coefficient for AlF3. Some possible consequences of the precipitation are discussed.

9:45 AM
Electric Conductivity of Cryolite Electrolytes during the Liquid-Solid Phase Transformation: *Stanislaw Pietrzyk*[1]; Ryszard Oblakowski[1]; [1]University of Mining and Metallurgy, Dept. of Metall. of Non-Ferrous Metals, Fac. of Non-Ferrous Metals, Al. Mickiewicza 30, 30-059 Krakow, Poland

Is generally accepted that flow of current during aluminium electrolysis may take place through the layer of side ledge (frozen electrolyte) to the carbon side lining of cathode. It may lead to current losses and this way contribute to decrease of current efficiency. Interesting from this of point of view is recognition of changes of electrical conductivity of the sodium cryolite melts during the liquid-solid phase transition and in solid state. In this work electrical conductivity measurements were performed on natural cryolite and its binary solutions with AlF3, CaF2, MgF2, LiF and also on industrial electrolytes. In solidified mixtures of cryolite with additions of AlF3, CaF2, MgF2, the conductivity is higher than in pure cryolite in the same temperature range. This observation suggests that in solid state additives of AlF3, CaF2, MgF2 cause changes in structure of solid cryolite by introduces cation vacancies which facilitate the ion transport.

10:10 AM Break

10:20 AM
Electrochemical Studies of Aluminum Deposition in Ionic Liquids at Ambient Temperatures: *Venkat Kamavaram*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metlgcl. & Matls. Eng. & Ctr. for Green Mfg., Tuscaloosa, AL 35487 USA

Ionic liquids are low melting salts used as potential non-aqueous electrolytes for the electrodeposition of aluminum. In the present study, an ionic liquid based on AlCl3-1-methyl-3-butylimidazolium chloride was used as an electrolyte for the electrodeposition of aluminum at ambient temperatures. Electrochemical studies were carried out using cyclic voltammetric (CV) and chronoampereometric (CA)techniques. Electrodeposition of aluminum was carried out in the above mentioned ionic liquid on copper cathode. The process of electrodeposition was found to be quasi-reversible and the kinetic mechanism was identified as instantaneous nucleation followed by diffusion controlled three-dimensional growth. Kinetic parameters such as charge transfer coefficient (α) and diffusion coefficient(D0) of the species Al2-Cl7- were obtained from the CV and CA data, which compared well with those available in the literature for aluminum deposition.

10:45 AM
The Anode Effect Revisited: *Jim B. Metson*[1]; R. G. Haverkamp[2]; M. M. Hyland[3]; Jingxi Chen[1]; [1]University of Auckland, Dept. of Chem., PB 92019, Auckland, New Zealand; [2]Massey University, Inst. of Tech. & Eng., PB 11 222, Palmerston N., New Zealand; [3]University of Auckland, Dept. of Cheml. & Matls. Eng., New Zealand

Despite an extensive literature and long history of plant experience, there are still puzzling aspects of the anode effect as observed in aluminium reduction cells. The nature of the onset of the anode effect and its variable persistence, are still the subject of debate. In this study, contact angles at carbon/electrolyte interface have been measured, as a function of electrolyte composition. In line with previous studies, CaF2 is observed to effect anode wettability, and contact angle increases with alumina depletion. Voltage/time behaviour of the cell is thus interpreted in terms of chemical changes affecting the interfacial surface tension, particularly those arising from the influence of additives in the bath and anode. Modification of the anode surface, either by doping or by reaction with the electrolyte, and the non-wettability of the modified surface by certain bath compositions, explains why these effects can be difficult to quench, even with elevated alumina concentrations.

11:10 AM
A Predictive Thermodynamic Model for the Al-NaF-AlF3-CaF2-Al2O3 System: *Patrice Chartrand*[1]; Arthur D. Pelton[1]; [1]Ecole Polytechnique, CRCT, CP 6079, Sta. "downtown", Montreal, Quebec H3C 3A7 Canada

A large body of experimental thermodynamic and phase-equilibrium data exists for the Al-Na-Ca-F-O system. All available data are critically evaluated to obtain optimized parameters of thermodynamic models of all phases. The bath model is the Quasichemical model in the quadruplet approximation that evaluates 1st- and 2nd-nearest-neighbor short-range-order. Other solution models are used for non-stoichiometric solid cryolite, the gas phase and molten Al. The models are then used to predict the thermodynamic properties and metal-bath-gas equilibria in the multicomponent heterogeneous system. All experimental data were reproduced within experimental error limits, and in most cases ternary and multicomponent data were predicted by the model, not fitted. The model parameters form a database that is part of the FactSage Thermochemical Software. This software can be used to predict/simulate metal-bath-gas equilibrium (phase amounts, compositions, activities/partial pressures, enthalpies, Tliquidus, etc.) for 25°C < T < 1100°C, for P < 4 atm, and for BR(wt.%) from 3.0 to AlF3 saturation.

Cast Shop Technology: Modeling I

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

Monday AM — Room: 6A
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Alvaro Giron, Alcoa Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA; Asbjorn Mo, SINTEF, Box 124 Blindern, N-0312 Oslo, Norway

8:30 AM
Applications of Optimization in Metal Casting and Heat Treatment: *Ravi Vijayaraghavan*[1]; Mei Li[1]; Xuming Su[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Rsrch. Labs., MD 3182, Rm. 2122, 2101 Village Rd., Dearborn, MI 48124 USA

A major thrust of cast aluminum research at Ford is the development of computer-aided engineering methods to reduce cycle time and cost for producing optimized cast aluminum automotive components. The goal is to provide tools that simulate casting solidification and predict microstructure, mechanical properties and durability in a cast component. The vision is to create and test virtual aluminum castings, reducing the need for physical prototypes. Optimization has been identified as a critical tool to accomplish this. Many major foundries are using software such as ProCAST and MagmaSOFT, to simulate casting and heat treatment and to optimize process design based on the simulations. A number of input parameters to these casting simulation programs (such as material properties and boundary conditions) are not well known. This has motivated the need for an optimization tool, which uses an inverse modeling approach to determine these parameters given the experiment results. The talk presents applications of optimization methods in casting process development and heat treatment. Use of efficient and robust optimization based methodologies can play a major role in reducing costs and improving productivity and quality of cast products.

8:55 AM
Mathematical Modeling of Melt Flow in Die-Casting Shot Sleeve: Carroll E. Mobley[1]; Junmin Park[1]; *Yogeshwar Sahai*[1]; [1]The Ohio State University, MSE Dept., 2041 College Rd., Columbus, OH 43210 USA

In a typical cold chamber die-casting operation, molten aluminum alloy is poured into a shot sleeve and a plunger rapidly pushes the metal into the die cavity. During this process, air can be trapped in the liquid alloy, and some liquid alloy can solidify in the shot sleeve. Entrapped air and Externally Solidified Product (ESP) degrade the quality of the die-cast products. To reduce the amount of trapped air and ESP, metal flow was mathematically simulated using a commercial code, FLOW3D. The effects of plunger velocity and acceleration, density, dynamic viscosity, kinematic viscosity, and surface tension of molten alloy were studied. The simulated flow patterns were compared with the experimentally obtained flow patterns in a water model. The plunger velocity and acceleration were found to have strong influence on the wave pattern and hence on the amount of trapped air. The effect of other parameters that are stated above on the wave pattern was relatively small. As plunger velocity increases, the amount of trapped air first decreases, followed by an increase. Thus, there is an optimum plunger velocity at which the amount of entrapped air is minimum. The tendency of rolling-over of wave created by metal flow in shot sleeve increases as acceleration increases. The simulations in this study agree well with the available experimental data.

9:20 AM
Determination of Boundary Conditions for Solidification Simulation in an Aluminum Permanent Mold Test Casting: *Joy Adair Hines*[1]; Ravi Vijayaraghavan[1]; Emmanuel K. Glakpe[2]; [1]Ford Motor Company, Ford Rsrch. Lab., MD 3182/ SRL, PO Box 2053, Dearborn, MI 48121-2053 USA; [2]Howard University, Dept. of Mechl. Eng., 2300 Sixth St., N.W., Washington, DC 20059 USA

Computer simulation has played an increasingly important role in the manufacture of shape castings in recent years. One of the major challenges to accurate casting simulation has been the determination of accurate boundary conditions for a particular casting. In this work, a specially designed test casting was cast using a low pressure process under a variety of conditions. Cooling curves were obtained from regions in both the casting and the mold. Those results were then compared with cooling curves from the same locations in a finite element mesh of the entire cast system. A Ford Motor Company proprietary software program was used to optimize the interfacial heat transfer coefficients between the metal and the mold as well as to verify the thermal properties within the mold itself. These results were then used to predict the solidification behavior of a cylinder head casting.

9:45 AM Break

10:00 AM
Modeling of Twin-Roll Thin Strip Casting of Aluminum Alloys: *Yogeshwar Sahai*[1]; Amit Saxena[1]; [1]The Ohio State University, MSE Dept., 2041 College Rd., Columbus, OH 43065 USA

In twin-roll thin strip casting processes, thermo-mechanical stresses in solidifying alloy may result in formation of defects. The first part of this modeling study consisted of predicting melt flow, temperature profiles, and resulting solidification during the thin strip casting process. The predicted temperature gradients in the solidifying alloy and externally applied mechanical force values, such as roll force and strip exit tension, were used to predict the thermo-mechanical stresses caused in the cast strip. A visco-plastic constitutive relation was used to describe the behavior of solidifying aluminum alloy. The effects of inlet melt feed rate and contact strip/roll heat transfer coefficient on the resulting stress profile in the cast strip and rolls were investigated. The implications of these results in industrial practice are discussed.

10:25 AM
Mathematical Modelling of Wire Rod Casting: *Dag Lindholm*[1]; Einar Arne Sørheim[1]; John Rødseth[2]; Dag Mortensen[1]; Harald Heggland[2]; [1]Institute for Energy Technology, PO Box 40, 2027 Kjeller, Norway; [2]Hydro Aluminium, RD Matls. Tech., 4265 Håvik, Norway

Wire rod casting is a complex process where molten aluminium solidifies within a rotating wheel mould. Bar shrinkage and air gap formation influence on the cooling conditions and thus the quality of the bar leaving the wheel. This work focuses on a mathematical model that is tailor-made for continuous rod casting. The two-dimensional transient model is based on a finite volume technique, and it computes the development of temperature field in the bar, mould and steel belt during the casting, as well as in the bar alone when it leaves the rotating wheel. The work is supplemented with presentation of full-scale measurements of temperatures and a technique for inverse modelling to find both internal and external heat transfer coefficients.

10:50 AM
Multiphase Modeling of Gas Purging during Continuous Metal Casting: *Anirudh A. Mukhopadhyay*[1]; Andrey A. Troshko[1]; [1]Fluent, Inc., 10 Cavendish Ct., Centerra Resource Park, Lebanon, NH 03766-1442 USA

Flow field in the continuous casting molds is critically important for quality and consistency. Two-phase flow of liquid metal and gas bubbles has been simulated with two-fluid model. Ability of the two-fluid model to accurately simulate such flows strongly depends on the correct closure of interfacial forces governing momentum transfer across the interface. The interfacial forces include: drag force: drag law is available for viscous, distorted and spherical cap regimes; lift force: modified lift coefficient takes into account inviscid lift and aerodynamic lift due to the vortexes shed by the bubble wake; turbulence dispersion force: this force accounts for the bubble dispersion caused by turbulent eddies in the mold; Another important aspect is non-uniformity of bubble size. Bubble size is controlled by liquid turbulence and several sizes are investigated. This study focuses on bubble induced flow modification in the mold.

11:15 AM
A Cooperative Process for Conducting R&D in the Aluminium Casthouse Products Sector: *John A. Taylor*[1]; David H. StJohn[1]; [1]CRC for Cast Metals Manufacturing (CAST), Dept. Mining, Minls. & Matls. Eng., The University of Queensland, Brisbane, QLD 4072 Australia

CAST conducts research, commercialisation and education activities across four sectors within the light metals industry. The Aluminium

Casthouse Products sector spans activities ranging from improving production processes, to expanding product range and quality, to providing technical support for downstream users. Strategic and industry-focussed projects with medium to long term goals are conducted with Centre partners and are supplemented with shorter-term projects with fee-for-service customers. The strength of the Cooperative Research Centre approach for providing innovative solutions lies in the networking of appropriately-skilled teams and the creation of value chains across diverse organisations. Flexibility and creativity in funding, IP and commercialisation arrangements exist. Cross-fertilisation of ideas from adjacent sectors enhance the solution-generation process. This paper explores the CRC process as a model for conducting industrial R&D and uses examples to elucidate the basic principles. Comparisons are made with more traditional R&D approaches.

Computational Modeling of Materials, Minerals & Metals Processing: Monday AM Opening Plenary Session 9:00 AM – 10:30 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

Monday AM	Room: 619-620
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Mark Cross, The University of Greenwich, Sch. of Compg. & Mathl. Scis., Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK; James W. Evans, University of California, Dept. of Matls. Sci. & Minl. Eng., 585 Evans Hall, Berkeley, CA 94720 USA

Keynote
Multi-Physics Simulation of Metal Processing: *Mark Samonds*[1]; [1]UES Software, Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA

Manufacturing processes which involve liquid to solid and solid state transformations in metals encompass a wide variety of physical phenomena. The past twenty years have seen a steady evolution in the complexity and scope of numerical models of these processes, beginning with thermal analyses without phase change. Recently, a few software packages have become available which have true multi-physics capabilities. This permits the treatment of thermal, fluids, stress, electromagnetic, chemical reaction and microstructure development aspects altogether in a fully coupled simulation. This paper will consider various aspects of these types of models.

Keynote
Computing the Dynamic Interaction of Magnetic Fields and Turbulent Conducting Fluids in Metals Processing: *Koulis Pericleous*[1]; [1]University of Greenwich, Greenwich Maritime Campus, Queen Mary Ct., Rm. 361, Greenwich, London SE10 9LS UK

Magnetic fields have many actual applications in the metals processing industry. Externally applied magnetic fields give rise to electromagnetic (Lorentz) forces formed by the cross product JxB, between the induced current density J and the magnetic field density B. When the metal is in liquid form, the Lorentz force generates motion in the fluid which in applications of practical interest becomes turbulent. In modelling terms, the Lorentz force appears as a source in the momentum equations. In addition, the induced current generates heat (Joule heating) in the metal that is in proportion to J2, with a corresponding source of heat in the energy equation. Whether as heat or as a force, these effects represent action at a distance–a most useful attribute when dealing with hot metal. The Lorentz force is used to stir solidifying alloys, pump liquid metal in conduits, dampen the flow in the meniscus of a continuous caster, levitate metal drops, induce artificial gravity conditions in suspensions or contain liquid metal. Elsewhere, the Lorentz force may be a by-product of some other operation, so leading to wave excitation in aluminium electrolysis cells, or altering the shape of the weld pool in arc welding. Joule heating is most commonly used with applied AC fields, to melt metal in induction furnaces. The author and his colleagues have been involved in the modelling of most of these processes in the past decade. Modelling is not however straightforward, since most of the examples mentioned represent genuine multi-physics challenges. There is a strong coupling between the flow field and electromagnetic field. The addition of a dynamically varying metal free surface and the moving solidus front means the flow, heat and electromagnetic fields need to be computed simultaneously. In situations involving metal containment, the metal free surface position is governed by the interplay of gravity, Lorentz force, surface tension and fluid inertia. Since all the interesting effects often happen in thin boundary layers at the surface due to the skin effect, mesh generation and mesh control during the computation become non trivial problems that need to be addressed. This paper presents a review of numerical methods used to model droplet levitation, semi-levitation melting and cold crucible induction melting of metals. The first method is based on spectral collocation techniques and the second is the traditional FV approach. Steps taken to validate the computations and typical transient results are also given.

10:30 AM Break

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

Monday AM	Room: 619
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Mark Samonds, UES Software Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA; Mayur Patel, University of Greenwich, Ctr. of Numcl. Modlg. & Proc. Analy., London SE109LS UK

10:45 AM
Simulation of Turbulent Flow and Particle Transport in the Continuous Casting of Steel: Q. Yuan[1]; T. Shi[1]; S. P. Vanka[1]; *B. G. Thomas*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mechl. & Indl. Eng., 1206 W. Green St., Urbana, IL 61801 USA

The quality of continuous cast steel is greatly affected by fluid flow in the mold region, especially involving transient phenomena. Mathematical models are being applied to investigate many different aspects of these phenomena, but their accuracy must be validated before they can be applied with confidence. As part of a long-term effort to develop and apply comprehensive models of the continuous casting process, this work evaluates the relative accuracy of models of three different fluid flow phenomena in continuous casting through comparison with measurements. Firstly, transient flow simulations of velocities in the mold region are compared with digital particle image velocimetry (PIV) measurements in a single phase water model. Large-eddy simulations (LES) are found to reasonably match the flow measurements, including transient flow variations, except at long time scales, which could not be modeled owing to the excessive computation costs. The standard K-ε model produced very good agreement with time-averaged velocities for relatively little computation time, although it is inaccurate at predicting the transient variations. Secondly, particle trajectory calculations are compared with water model measurements to study the distribution and flotation removal of inclusion particles. The LES model was able to match the measurements both qualitatively and quantitatively. Thirdly, steady, multiphase flow computations are compared with flow patterns observed in both a water model and an operating steel caster with argon gas injection. For the same conditions, the water model and steel caster produced very different flow behavior. The computational model was able to match the measured flow patterns in both cases. This work suggests that computational flow modeling has the potential to match real processes as well or better than water models, especially when complex related phenomena such as particle motion and multiphase flow are involved. Much work is still needed to further improve the models and to apply them in parametric studies.

11:10 AM
CFD Modeling of the Hydrodynamics of Fluidization in the Sand Surrounding a Lost Foam Casting Pattern: *Nathanael Harrison Hudson*[1]; *Sushil Bhavnani*[1]; Ruel A. Overfelt[1]; [1]Auburn University, Dept. of Mechl. Eng., 213 Ross Hall, Auburn University, AL 36849 USA

In the aerospace and automotive industries, shapes arise which require casting. The problem with these cast parts is the empiricism and expense in developing an efficient casting process. There has been an interest in using the fluidized bed to allow sand to better encapsulate the complicated surface geometry of a lost foam pre-casting mold. Fluidization helps to eliminate the voids in the sand and improves the integrity of the casting. At issue is the hydrodynamics of the sand and air around the pre-casting mold. The software PHOENICS, employing a two-fluid approach, is used to simulate the flow of sand and air as interpenetrating continua. The kinetic theory of granular flow for the sand phase is incorporated into the re-compilable PHOENICS code. The results of this study consist of voidage patterns and the velocity components of the respective phases around the pre-casting submerged in a two-dimensional fluidized bed. The model is benchmarked against experimental voidage patterns without a foam pre-casting and to some trials with a pre-casting. The final test consists of a series of computer runs with an obstacle submerged in the bed at various aspect ratios of length to width.

11:35 AM
Continuum Modelling of Granular Flows using PHYSICA, a 3-D Unstructured, Finite-Volume Modelling Framework: *Nicholas Christakis*[1]; Mayur K. Patel[1]; Mark Cross[1]; John Baxter[2]; Hadi Abou-Chakra[2]; Ugua Tünün[2]; [1]University of Greenwich, Ctr. of Numcl. Modlg. & Proc. Analy., Sch. of Compg. & Mathl. Scis., 30 Park Row, London SE10 9LS UK; [2]University of Surrey, Dept. of Cheml. & Proc. Eng., Guildford, GU2 5XH Surrey, UK

In recent years significant effort has been put in using Continuum Mechanics for the description of granular flows. Although these models are partially successful in capturing some flow characteristics, they lack essential information on material properties, which are needed to account for the interactions between different particles. Thus, they are incomplete and can not be used to describe processes such as hopper filling/emptying and pneumatic conveying, where particle-particle interactions can lead to phenomena such as segregation, degradation and agglomeration. In this paper, a 3-D unstructured Finite-Volume framework is presented, which employs interface tracking techniques (VOF/SEA algorithms) to determine the material-air interface. Separate routines are employed to perform the tracking of the individual material components of the granular mixture in the bulk. Various transport processes, arising from the micro-mechanical properties of the different particle species in the granular mixture, can be obtained through kinetic theory. The transport coefficient(s) of each of the individual species can be determined and analysed in a micro-mechanical framework and the transport process parametrised in the form of constitutive model. These models provide the continuum theory with information on the micro-mechanics. This work describes in detail the numerical schemes employed in the Continuum Mechanics framework and the micro-mechanical parametrisations that are implemented in the transport equations. Model predictions for different flow conditions and comparisons with experimental data are presented and conclusions are drawn on the model capability to realistically predict and quantify the main characteristics of granular flows.

12:00 PM
Mathematical Model of Inclusion Removal during Steel Degassing: Michel Cournil[1]; Frédéric Gruy[1]; *Pascal Gardin*[2]; Hubert Saint-Raymond[3]; [1]Ecole des Mines de Saint-Etienne, SPIN Div., 158 Cours Fauriel, Saint-Etienne, Cedex 2 42023 France; [2]IRSID, Themef, Voie Romaine, BP 30320, Maizières-lès-Metz, Cedex 57283 France; [3]IRSID, PCMO, Voie Romaine, BP 30320, Maizières-lès-Metz, Cedex 57283 France

The control of inclusion elimination is getting more and more important to obtain clean steel. But having a predictive tool is still a challenge, because the number of phases is important in steelmaking industry: liquid steel, slag layer, bubbles and inclusions (with a large range of composition and rheology). The paper presents the methodology which is developed at IRSID to predict oxygen content evolution during RH degassing.The main mechanisms which have to be considered are:-inclusion growth by turbulent aggregation of elementary inclusions (keeping in mind that liquid steel is a non-wetting medium for inclusions); the difficulty is to express collision efficiency for alumina particles, -inclusion removal by flotation; the difficulty for alumina clusters stems from the complex morphology of particle; fortunately, the use of fractal concept makes it possible to cope with this problem.The proposed paper describes the general modelling of inclusion removal taking into account the previous mechanisms. Hydrodynamic parameters are obtained by means of Fluent CFD package and a specific coding is developed for cluster growth. Influence of different parameters (fractal dimension, argon flow rate) on time-dependant inclusion size distribution is given.

12:25 PM
Application of Coupled Continuum-Mesoscopic Computational Methods for the Simulation of Complex Fluids in Industrial Processes: Greg Glinski[1]; *Chris Bailey*[1]; Koulis Pericleous[1]; [1]University of Greenwich, 30 Park Row, Greenwich SE10 9LS UK

Recent advances in new computational modelling techniques such as Lattice-Boltzmann and Dissipative Particle methods offer the prospect of simulating complex fluids such as colloidal and dense suspensions in industrial processes. These methods provide a means to overcome challenges that arise in modelling such fluids due to the disparate temporal scales present. These methods are termed mesoscopic methods as they lie between computational intense molecular dynamics and traditional macroscopic CFD methods. This paper will discuss the use of the methods to simulate the movement and subsequent processing of solder paste material in electronic component manufacture. Results will be presented that show how these methods are coupled within a macroscopic CFD code to provide detailed predictions of solder paste deposition onto a printed circuit board. Comparisons between the model results and experimental data will also be presented.

Computational Modeling of Materials, Minerals & Metals Processing: Track B - Heat & Mass Transfer - 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

Monday AM — Room: 620
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Koulis Pericleous, University of Greenwich, Greenwich Maritime Campus, Greenwich, London SE10 9 LS UK; Stavros A. Argyropoulos, University of Toronto, Dept. of Metall. & Matls. Sci., Toronto, Ontario M5S 3E4 Canada

10:45 AM
Heat Load Control of Blast Furnace Wall using Statistical Optimization Techniques: *Tae-hwa Choi*[1]; Yong-hwan Chu[2]; Chonghun Han[2]; [1]Pohang Iron and Steel Company(POSCO), Techl. Rsrch. Labs./Iron & Steel Making Rsrch. Grp., 1 Goedong-dong, Nam-gu, Pohang-shi, Gyungbuk 790-785 Korea; [2]Pohang University of Science and Technology, Dept. of Chem. Eng., 31 san Houja-dong, Nam-gu, Pohang-shi, Gyungbuk 790-300 Korea

In the blast furnace, various complex phenomena take place including mass transfers, heat transfers, a lot of reactions and phase equilibriums, but systems of these phenomena are not found out clearly even until now. Consequently, it is very difficult to make fundamental models on these systems, which results in the operation based on the heuristics of industrial operators by changing operating condition little by little due to ignorance of the optimum point. However, since this method is not systematic, we propose statistical optimization technique based on analysis of historical data and empirical model building. In this approach, we first find the variables which must be optimized and the manipulated variables to adjust those response variables. Next, we collect the necessary data for these variables after preprocessing such as removal of noise and outlier. Finally, we construct empirical models describing the patterns of response variables in terms of manipulated variables by PLS regression method, and then these models are used as objective functions for entire optimization problem. By using appropriate optimization algorithm, this multi-objective optimization problem is solved, which gives us the compromising optimum operating condition considering all response variables from the past operating conditions.

11:10 AM
Mathmatical Modeling and Experimental Verification of Assimilation of Exothermic Additions in Liquid Metals: *Stavros A. Argyropoulos*[1]; Henry H. Hu[2]; [1]University of Toronto, Dept. of Metall. & Matls. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]University of Windsor, Dept. of Mech., Automot. & Matls. Eng., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada

The assimilation of exothermic additions in liquid metals exhibit an array of unique coupled heat, mass and momentum transport phenomena. The phenomena are further complicated with the presence of a moving boundary. In this paper a mathematical model will be described which solves these coupled complex phenomena. The SIMPLER algorithm was employed to solve numerically the pertinent partial differential equations. The computational results indicated that the exothermic heat of mixing leads to a rapid increase of temperature around the moving boundary, which produced an enhanced convective flow in the liquid phase. The intensification of fluid flow around the moving boundary resulted in an acceleration of the melting process. An extensive verification of the mathematical model was carried out and will be described in the paper. First, in a low temperature physical model consisting of ice immersion in different sulfuric acid solutions. In this physical model, both temperature and velocity measurements were carried out. The model results were compared with experimental measurements and they were found to be in good agreement. Second, in a high temperature work involving assimilation of silicon in high carbon liquid iron. The model was also applied to predict fluid flow, heat and mass transfer for this high temperature experiments and a reasonable agreement was obtained. In addition new dimensionless heat transfer correlations that quantify these complex phenomena will be presented.

11:35 AM

A Model of the Cathode Dynamics in Electric Field-Enhanced Smelting and Refining of Steel: *David Michael Dussault*[1]; Adam Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139 USA

A mathematical model of coupled diffusion, electrochemical reactions and fluid dynamics at the cathode in electric field-enhanced smelting and refining of steel is developed using the Navier-Stokes equations and the phase field method. Experimental evidence indicates that the reaction rate is limited by ferrous ion transport to the cathode, resulting in a Mullins-Sekerka instability at the slag-metal interface and the growth of liquid iron fingers into the liquid slag. The differential equations are discretized using finite differencing with a uniform mesh, and the resulting system of nonlinear equations is solved using a multidimensional Newton-Krylov method. Presented are the formulation and two dimensional results, and issues expected to arise on extension to three dimensions are discussed.

12:00 PM

A Mathematical Model for the Control of Metallurgical Properties of the Product Sinter: *Ndabezinhle Manengi Dube*[1]; E. F. Vegman[2]; [1]University of Zimbabwe, Fac. of Eng., Dept. of Metall. Eng., PO Box MP167, Mount Pleasant, Harare Zimbabwe; [2]Institute of Steel and Alloys, Moscow Russia

In the conventional practice of sintering, the quality of the product sinter down the cross-section of the bed is quite variable,with the top layer consisting of weak sinter and the bottom layer, strong over-fused sinter of poor reducibility. This points to a variable distribution of the heat balance in the sintering process down the sinter bed. This causes losses in the productivity of the sinter machine and the blast furnace. The experienced phenomenon caused by varying input of regenerated heat requires a zonal approach to the study of the sintering process with the aim of producing a uniform quality of the sinter cake. The current work includes a mathematical model that would make possible optimization of multiple-layer sintering and segregation technologies. It is based on a zonal heat balance. This calculation method presents the following prospects: 1. The minimum theoretically possible coke fines consumption during the sintering of any type of iron ore fines can be established. The optimum difference between the coke fines rates in the top and bottom layers during multiple-layer sintering should be easily determined with the aid of this model. 2. On the basis of this model an automatic control system for the coke rates in the top and bottom layers during two-layer sintering is possible. 3. The model can be used to control the quality (chemical, physical and metallurgical) of the sinter in any point down the sinter bed since the data input includes basicity, combustion zone temperature, and FeO content in the product sinter.

12:25 PM

Modelling the Magnetostriction of Textured Ferromagnetic Materials with a Cubic Structure: *Ruben Decocker*[1]; Leo Kestens[1]; Yvan Houbaert[1]; [1]University Ghent, Metall. & Matl. Sci., Technologiepark 9, Ghent, East-Flanders 9052 Belgium

A magneto-elastic model is presented to calculate the orientation dependence of the magnetostrictive strain, observed at saturated magnetization in ferromagnetic materials with a cubic crystal structure and an arbitrary crystallographic texture. The formula of Becker and Döring is used to express the anisotropy of magnetostriction for a single crystal. In order to simulate the macroscopic average magnetostriction of a polycrystalline aggregate (with an arbitrary texture) the Reuss assumption of the elasticity theory was applied. According to this assumption the various orientations of the polycrystal can deform without constraints, producing local strain incompatibilities in the microstructure but observing a total stress equilibrium. The macroscopic strain is calculated as the weighted average of the individual strains of all orientations composing the polycrystal. The weight factors are determined by the volume fractions of the corresponding orientations of the given texture, which can be measured by standard X-ray diffraction techniques. The model is applied to simulate the variation of magnetostriction (at saturation) with respect to the rolling direction for a standard grade of non-oriented electrical steel. A brief comparison with experimental data allows validating the basic model assumptions.

Computational Phase Transformations: Stress-Dominated Processes

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

Monday AM	Room: 201
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Long-Qing Chen, Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM Opening Remarks

8:35 AM

3D Phase Field Microelasticity in 3D Mesoscopic Modeling of Complex Structures: Yongmei M. Jin[1]; Yu U. Wang[1]; *Armen G. Khachaturyan*[1]; [1]Rutgers University, Dept. of Cer. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

Recent developments in the Phase Field Microelasticity theory and its applications to phase transformations and plastic deformation (dislocation dynamics) of structurally inhomogeneous single and polycrystals as well as the development of the computer simulation techniques based on this theory open the way to a realistic 3D simulation of technologically important materials. The strength of the Phase Field Microelasticity is its ability to describe the long-ranged strain-induced interaction between structural inhomogeneities of arbitrary topological complexity in the spontaneous self-organization processes without any a priory assumption. The incorporation of dislocations into the phase transformation theory and taking into account the grain structure allow one to attack the entire spectrum of important problems of the computer-aided materials design. Examples of 3D simulations of a real martensitic transformation in Au-Cd polycrystalline shape memory alloy and a simulation of the evolution of multi-dislocation systems in single and polycrystals during plastic deformation are presented.

9:20 AM

Phase Field Modeling of Fracture and Deformation of Non-Crystalline Materials: *Mo Li*[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Deformation and fracture are non-equilibrium dynamic problem. Continuum and, most recently, atomistic dynamic modeling are used for fracture in crystalline and other homogeneous materials. These methods, however, are not quite suitable for fracture and deformation in non-crystalline materials. The primary reason is that there is no clear understanding of the constitutive behaviors as well as detailed microscopic mechanisms in these materials. In addition, the minimum dimension of the fracture or deformation regions in non-crystalline materials is of the order of submicrons, which is too large for atomistic modeling and too small for continuum modeling. To overcome this difficulty, we developed a new approach using the phase field model. In this talk, I will describe the formulation of this new method for deformation and fracture in non-crystalline materials; I will also give examples from the modeling using this method in crack initiation, propagation, and branching or shear localization in amorphous metals.

9:50 AM
Computer Simulation of Interactions of Migrating Grain Boundaries and Dislocations with Solutes and Precipitates: *Yunzhi Wang*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The interactions between migrating defects such as grain boundaries and dislocations with impurities and precipitates determine many important properties of materials. In this presentation, we discuss recent advances in phase field modeling of solute segregation and phase transition at migrating grain boundaries and dislocations and migration of dislocations through g channels in single crystal cuboidal gamma/gamma microstructures and gamma/gamma multilayers of superalloys. In particular, the transition from low solute segregation to high solute segregation will be characterized as function of temperature, solute concentration and defect migration velocity. The motion of confined dislocation propagation within g channels and layers will be characterized as function of channel width and layer thickness and misfit dislocation content. The simulation results will be discussed against the ones obtained from other methods that update defect and microstructural states according to a path of steepest decent in energy or according to a Monte Carlo process.

10:20 AM Break

10:40 AM
Study of Strain-Mediated Transitions via Discrete Atom Method: *Jong K. Lee*[1]; [1]Michigan Technological University, Matls. Sci. & Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

Materials processing presents various strain-mediated phenomena. For example, heteroepitaxially-grown thinfilms split into islands, or certain gamma prime precipitates during the processing of nickel-based superalloys split into smaller particulates. Such elastic instabilities occurring in complex boundary conditions were studied through the discrete atom method (DAM), which is predicated upon both statistical mechanics and linear elasticity. Precipitate splitting was found to depend on the commensurateness in elastic anisotropy between a precipitate and its matrix phase. A driving force for ordering was long known to be strain relaxation due to difference in atomic size. Such an ordering in a substitutional alloy was also examined, and the transition was found to be, depending on the degree of mismatch, of either a second-order or a first-order kinetics. The DAM work was recently extended to simulate the Jahn-Teller distortion which is partially responsible for the colossal magnetoresistance, i.e., a large decrease in resistance upon application of a magnetic field for certain perovskite-type manganese oxides such as La0.7Ca0.3MnO3. The distortion due to the spin-orbit coupling of Mn ion was approximated to be harmonic and its related ordering behavior such as ferromagnetic phase transformations was studied.

11:10 AM
Ostwald Ripening in Elastically Stressed Solids: K. Thornton[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Phase separation in crystalline solids is usually accompanied by long-ranged elastic stress. We have investigated the dynamics of Ostwald ripening in elastically stressed crystalline solids through large-scale numerical simulations. Using the insight provided by the calculations, a theory for the kinetics of ripening in elastically anisotropic homogeneous solids is developed. Both the theory and simulations show that elastic stress does not alter the exponent of the temporal power law for the average particle size, but does affect the amplitude of the power law in a manner that is only a function of the symmetry of the particle morphology. At sufficiently large misfit or volume fraction of precipitate interparticle coalescence may play a significant role in the coarsening process. The results of calculations that follow this coalescence process using a hybrid boundary-integral level-set method will be presented. Work supported by the National Science Foundation.

11:40 AM
A Mesoscopic Electromechanical Theory of Polycrystalline Ferroelectrics: *Jiangyu Li*[1]; [1]University of Nebraska, Dept. of Eng. Mech., Lincoln, NE 68588 USA

We present an effective mesoscopic theory on the electromechanical behavior of polycrystalline ferroelectrics which takes into account both the effect of domains and grains, and gives remarkably good predictions on the polarizability, saturation polarization and strain, the effective texture, and the effective piezoelectric constants of ferroelectric polycrystals. The superior electromechanical property of PZT at morphotropic phase boundary is explained, and optimal texture of polycrystals for high-strain actuation is identified.

Creep Deformation: Fundamentals and Applications: Fundamentals Behavior - I

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

Monday AM	Room: 214
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: K. L. Murty, North Carolina State University, Box 7909, Raleigh, NC 27695-7909 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Elevated Temperature Crystalline Plasticity at Diminished Length Scales: *Amiya K. Mukherjee*[1]; [1]University of California, Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Creep and superplasticity have been studied in metallic and ceramic nanocrystalline materials. The nanocrystalline structures have been produced using one of the following techniques: high pressure torsion, pyrolysis of polymer precursor, crystallization from bulk metallic glass and electrodeposition. The processed nanocrystalline microstructure consisted of neat matrix, dispersion strengthened alloy, nano-nano and micro-nano composite and nanocrystalline thin film multilayers. The ceramic nano-nano composites exhibited extremely low creep rates. The metallic nanomaterials demonstrated both high strain-rate as well as low-temperature superplasticity. They also exhibited very high flow stresses and strain-hardening rates that cannot be explained by either grain growth or dislocation storage. The thin film multilayers revealed interesting scale-dependent behavior at elevated temperatures. The results will be discussed in terms of creep and superplastic deformation mechanisms in the domain of significantly dimin ished length scales. This investigation is supported by grants from NSF (#DMR-9903321) and ONR (#N00014-00-1-0186).

9:05 AM Invited
From Power Laws to Constitutive Relations–Creep as Special Case of Plastic Deformation: *Wolfgang Blum*[1]; Philip Eisenlohr[1]; [1]University of Erlangen-Nuernberg, Inst. f. Werkstoffwissenschaften LS1, Martensstr. 5, 91058 Erlangen, Germany

Research in creep is usually focusing on the power law relation between minimum creep rate and stress at constant temperature. Regions with a particular constant value of the stress exponent n are generally interpreted in terms of a particular deformation mechanism. The aim of the present contribution is to interpret creep as a special case of plastic deformation, starting with overelastic loading of the material up to a certain stress σ and then continuing at constant σ with work hardening or softening towards the steady state. The state of development of constitutive relations for the evolution of the dislocation structure with particular emphasis on dynamic recovery of dislocations and the kinetics of dislocation motion is presented. It is shown that contemporary constitutive relations developed for describing work hardening are also suited to describe creep. The constitutive modeling is helpful in checking the validity of the traditional power law interpretations.

9:30 AM Invited
Transitional Creep Mechanisms in Zr-Alloys: Application to Dry Storage of Spent Nuclear Fuel: *K. L. Murty*[1]; Y. Zhou[1]; B. Devarajan[1]; [1]DMR, National Science Foundation, Metals Rsrch., Prog. Dir., 4201 Wilson Blvd., Arlington, VA 22230 USA

Transitions in creep mechanisms are investigated in thin walled tubing of Zircaloy (Zr alloyed with Sn and Fe) and Nb-added Zircaloy under biaxial loading using internal pressurization superimposed with axial load. The diametral strains are monitored in-situ using a Laser telemetric extensometer. Both the alloys followed an exponential stress variation of the creep-rate at high (>10-3E) stresses. At very low stresses, viscous creep (n=1) was noted identifiable with Coble creep corresponding to small grain sized materials. At intermediate stress levels, Zircaloy behaved like class-M alloy with the climb of edge dislocations as the rate-controlling mechanism whereas addition of Nb seems to indicate class-A type with stress exponents of 3 and 5 at lower and higher stress levels respectively. These findings are consistent with the earlier creep and TEM studies. In addition, short-term burst rupture properties of Zircaloy and Nb-added Zircaloy are investigated at temperatures ranging from 638K to 843K by internal pressurization of closed-end tubing samples. The data enabled an evaluation of Larson-Miller parameter and the present experimental results on Zircaloy-4 were in excellent agreement with those reported in the literature. Negligible differences were noted in the rupture characteristics between the standard Zircaloy and Nb-added Zircaloy. These results have significance on the feasibility of surface storage of spent fuel where the creep deformation of the cladding could be a failure mode because of the residual heat and fission products following exposure to neutron irradiation in commercial nuclear plants. Although blind extrapolation of short-term creep results to low stresses encountered during dry storage could lead to nonconservative estimates of the creep-rates and creep-strains, our estimations indicate extremely small contributions of viscous creep to the total strain.

9:55 AM Invited

Creep Processes at Low Stress Levels: *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matl. Sci., Los Angeles, CA 90089-1453 USA

Creep experiments on pure metals and metallic alloys often show evidence for the occurrence of different creep mechanisms at very low stresses. For example, the stress exponent typically shows a transition to ~1-2 at low stresses. Possible mechanisms of flow under these conditions are grain boundary sliding, diffusion creep and Harper-Dorn creep. This paper examines the characteristics and viability of these various creep mechanisms.

10:20 AM Break

10:30 AM Invited

Thermal Activation in Plasticity: *U. Fred Kocks*[1]; [1]PO Box 89, Placerville, CO 81430 USA

In a recent paper, Cahn and Nabarro[1] and, earlier, Nabarro[2] have raised some old issues in this area–without, however, providing the answers that have been generally accepted (or at least widely used) since the publication, in 1975, of "Thermodynamics and Kinetics of Slip" by Kocks, Argon and Ashby[3], which has been recently summarized by Kocks[4]. The present short contribution aims at emphasizing some salient points, using the assertions in[1] as an organizing principle. Particular points to be discussed are: the meaning of activation parameters (including the 'pre-exponential factor') and their tensor character; the role of the 'mechanical threshold'; the influence of multiple slip and 'multi-axial tests'; a distinction of the directional from the scalar relation between stress and strain-rate; the relevance of 'reverse jumps' and of the hyperbolic-sine relation. [1]Cahn JW, Nabarro FRN, Thermal Activation under Shear: Phil. Mag. A81 [5],4409-4426; [2]Nabarro FRN, Thermal Activation and Andr ade Creep: Phil. Mag. Letters 75, 227 (1996) [3]Kocks UF, Argon AS, and Ashby MF, Thermodynamics and Kinetics of Slip: Prog. Mater. Sci. 19, 1-288 (Pergamon 1975) [4]Kocks UF, Plasticity: Thermal Activation Approach: Encyclopedia of Materials Science and Technology (Elsevier 2001).

10:55 AM

Deformation Processes during Creep of Pure Aluminium: *B. Wilshire*[1]; C. J. Palmer[1]; [1]University of Wales Swansea, Dept. of Matls. Eng., Singleton Park, Swansea SA2 8PP UK

When power-law relationships are used to quantify the dependences of the secondary or steady-state creep rates on stress (σ), the gradients (n) of the resulting log σlog (strain rate) plots vary in different stress/temperature regimes. Thus, pure metals are commonly considered to show regimes with $n \cong 1$ at low stresses and $n \cong 4$ at higher stresses, with n increasing rapidly as the stress increases further into the 'power law breakdown' range. Traditionally, the stress dependences of the stress exponent (n) are rationalised on the assumption that different creep mechanisms become dominant over different stress ranges. Yet, while creep is known to occur by diffusion-controlled generation and movement of dislocations when $n \cong 4$, no general agreement has been reached on the precise mechanisms involved. Moreover, an unresolved debate continues over whether creep in the $n \cong 1$ regime takes place by dislocation processes or by diffusional creep process which do not require dislocation movement. Given the controversies still surrounding the mechanisms controlling the creep properties of pure metals after more than 50 years of study, it is necessary to consider whether the continued adoption of power-law approaches has any prospect of proving successful in the future. For this reason, the creep behaviour patterns observed for pure aluminium have been re-evaluated. This analysis suggests that the concept of 'steady-state' creep should be abandoned on the grounds that a steady-state creep rate is rarely achieved. Moreover, standard stress/creep rate plots are shown to be well represented by continuous curves, with the dislocation processes governing the rates of creep strain accumulation being essentially the same at all stress levels, irrespective of the n value exhibited.

11:20 AM

Activation Volume for Shear under Low Stresses: *F. R.N. Nabarro*[1]; [1]Materials Research Institute, Univ. of the Witwatersrand, PB 3, Johannesburg WITS 2050 S. Africa

A system shows a strain rate ε under a shear stress σ. The customary definition of the experimental activation volume $V'^{e}(\sigma) = kT(\partial \ln\varepsilon/\partial\sigma)$ at constant temperature is valid only when σ is of the order of the flow stress at low temperatures. For Newtonian flow at low stresses $V'^{e}(\sigma) = kT/\sigma$, independently of the nature of the system. A similar anomaly occurs for power-law creep. We show that, for a potential that is symmetrical with respect to forward and backward jumps, if the true activation volume V'(σ) tends to a constant value V'(O) as σ tends to zero, then V'(O) cannot be determined by strain-rate changes in the Newtonian regime.

11:40 AM

A Microstructurally Based Model of Steady-State and Constant Structure Creep: *Jeffery C. Gibeling*[1]; Martin Heilmaier[2]; [1]University of California, Dept. Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Plansee AG, Tech. Ctr., A-6600, Reutte, Tirol, Austria

Current models have proven unsuccessful in fully rationalizing the unusual creep characteristics of dispersion strengthened metals. The most notable characteristics are the high value of the activation energy as compared to particle-free materials and the specific quantity and temperature dependence of the particle-induced threshold stress. While existing models generally provide satisfactory descriptions of creep under steady-state conditions, second order effects such as the response to stress changes have not been investigated to discriminate between models. To overcome these shortcomings, we propose a microstructurally based model that rests on the idea that internal back stresses develop during creep due to dislocation/dislocation and dislocation/particle interactions. Thus, the effective stress rather than the applied creep stress is the key factor for dislocation motion. This approach enables a unified description of the steady-state creep behavior of copper with and without dispersoids with a single s et of microstructurally founded parameters. In addition, the temperature dependencies of the creep resistance (incorporating the activation energies for self-diffusion and core-diffusion) and of the threshold stress (via the shear modulus) are correctly reflected. Finally, the difference in constant structure creep after stress reductions is predicted: while in pure copper the constant structure creep rate is found to be lower than the eventual steady-state rate, the opposite behavior is observed in dispersion strengthened copper.

David L. Davidson Symposium on Fatigue: Fatigue Mechanisms

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

Monday AM — Room: 208
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Kwai S. Chan, Southwest Research Institute, 6220 Calebra Rd., San Antonio, TX 78238 USA; Wole Soboyejo, Princeton University, Mechl. & Aeros. Eng., Princeton, NJ 08544 USA

8:30 AM Opening Remarks

8:35 AM Introduction

8:45 AM Keynote
Fatigue Crack Initiation Mechanisms and Fatigue Life in High-Cycle and in Ultrahigh-Cycle Fatigue: *Hael Mughrabi*[1]; [1]University Erlangen-Nuernberg, Institut fuer Werkstoffwissenschaften, Martensstr. 5, Erlangen D-91058 Germany

First, the different fatigue crack initiation mechanisms will be reviewed. Emphasis will be placed on cyclic strain localization phenomena, trans- and intergranular crack initiation mechanisms and crack initiation at second-phase particles and at near-surface pores and cavities. Then, attention will be turned to fatigue life diagrams both in the form of S-N curves and Manson-Coffin plots. Points of particular interest will be the consideration of fatigue life-controlling mechanisms in the ultrahigh-cycle fatigue (UHCF) or gigacycle range (fatigue lives in excess of ca. 10^8 cycles). Microstructural interpretations of so-called "two-stage" or "multi-stage" fatigue life diagrams exhibiting a second lower fatigue limit in the UHCF range will be proposed. In particular, it will be argued that such fatigue life diagrams are not confined to materials containing inclusions, such as steels, and in which internal fatigue cracking, originating from the inclusions, has been observed in the UHCF range. Rather, similar fatigue life diagrams are also to be expected for pure single-phase metals and alloys in which cracks always initiate at the surface.

9:15 AM Invited
Fatigue: SEM Based Studies of Crack Initiation and Early Growth: *Angus J. Wilkinson*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxon OX1 3PH UK

This talk will illustrate our use of SEM based diffraction methods in the study of some fatigue problems. The electron channelling contrast imaging (ECCI) method has allowed us to image dislocation substructures in bulk samples in the SEM. Some advantages afforded by ECCI are (i) large areas can be observed, (ii) repeated intermittent observations of the same area can be made, and (iii) sample containing cracks are readily studied. Examples of direct imaging of dislocation configurations at the tips of stage I and stage II cracks will be given. The use of electron back scatter diffraction in studying the effects of grain boundary geometry on the strength of short fatigue crack retardation will also be discussed. Studies in Al-Li 8090 alloy have shown that cracks propagate onto slip planes in the next grain so as to minimise the twist deflection, and for those boundaries at which the minimal twist deflection is large the crack retardation tends to be strong.

9:45 AM Cancelled
Mechanisms of High-Cycle Fatigue Crack Growth and Investigations on the Influence of Loading Frequency: *Stefanie Stanzl-Tschegg*

9:45 AM Invited
A New Concept of Sn Curve and Fatigue Strength in the Giga-cycle Fatigue: Claude Bathias[1]; [1]CNAM/ITMA, 2 rue Conte, Paris 75003

Safe-life design based on the infinite-life criterion was initially developed through the 1800s and early 1900s, one of which is the stress-life or S-N approach related to the asymptotic behavior of steels. A lot of materials display a fatigue limit or "endurance" limit at a high number of cycles (typical $>10^6$). Most other materials do not exhibit this response, instead displaying a continuously decreasing stress-life response, even at a great number of cycles (10^6 to 10^9), which is more correctly described by a fatigue strength at a given number of cycles. Time and cost constraints usually rule out the use of conventional fatigue tests of more than 10^7 cycles to check the structural materials. In contrast to conventional fatigue tests, which require a long duration of time due to the high numbers of load cycles at low frequencies (typical < 50 Hz), the proposed piezoelectric fatigue technique operates at much high frequency (about 20 KHz). Thus the required time for measurements in the high cycles fatigue range is considerably reduced. In the area of application and design, high-cycle fatigue (HCF) failures are very common. Unfortunately, they are not well understood nor there is a methodology available. The current standardized design methodologies are unable to accurately predict HCF damage. The goal of the research is to develop experimental and analytical techniques to understand the onset of HCF behavior of structural materials. The mechanisms of fatigue cracks have been determined thanks to microfractographic analyses on the scanning electron microscope. The fatigue strength is also estimated by the Murakami model, which has been applied to many fatigue problem of high strength steels including nonmetallic inclusions and small defects, but the model does not fit the gigacyclic fatigue well. A modified empirical equation was proposed to predict high cycle fatigue life of high strength steels. Investigations concerning long fatigue lives ($>10^7$ cycles) are relatively rare. The reason is obvious, the testing time and costs are too much to perform the fatigue tests of more than 10^8 cycles using a conventional testing machine. A possibility of accelerated tests of the structural materials (metals, alloys, composites) is considered by using high frequency cyclic loading. The ultrasonic fatigue is one of the most economical and practical ways to tests the structural materials. The S-N curves for the high strength aloys were obtained between 10^5 and 10^9 cycles. The most noticeable was that the continuous failure could occur over 10^7 cycles and the fatigue limit could not be obtained until 10^9 cycles. It should be described by a fatigue strength at a given number of cycles, and realized the risk of obtaining the fatigue limit at 10^7 cycles.

10:15 AM Break

10:25 AM Keynote
Assessment of the Origin of a Gas Turbine Disk Crack: *David L. Davidson*[1]; [1]Southwest Research Institute, 117 Elm Spring Ln., San Antonio, TX 78231 USA

An analysis has been made to determine the cause of cracking found in the disk of a fighter aircraft gas turbine engine. The investigation integrates (1) fractography of the attachment surface and metallography of the cracked disk, (2) stress analysis of the blade/disk attachment, (3) recent theoretical analyses of fretting fatigue, and (4) fatigue crack growth and stress-life characterization for the disk material. Information about the cracking gained from each of these techniques has uncertainty associated with it, for reasons specific to each. Thus, the cause of the crack remains uncertain (at least at the time of this abstract), but the analysis helps to clarify additional research that is needed to prevent this type of disk cracking.

10:55 AM Invited
Analysis of Fretting Fatigue with Spherical Contact in 7075-T6 Aluminum Alloy: *Jaime Dominguez*[1]; [1]ESI, University of Seville, Dept. of Mechl. Eng., Camino de los Descubriminentos s/n, Sevilla 41092 Spain

This paper analyses some aspects of fretting fatigue in 7075-T6 aluminum alloy. The case of uniaxial elements under cyclic loading and the action of a spherical fretting pad subjected to constant normal loading is considered. Friction forces and stresses involved in partial slip and small amplitude global sliding between the surfaces in contact are examined. The analysis includes an approach to calculate analytically the displacement of the stick zone induced by the bulk stress applied to the specimen. A series of tests controlling normal and tangential contact loads, and cyclic displacements in addition to the fatigue loading parameters cyclic stress amplitude, frequency, and load ratio has been carried out. Metallographic and fractographic examinations have been done, and the experimental results have been critically examined in the light of a variety of known multiaxial fatigue criteria. A method for estimating the total fatigue life in fretting fatigue is proposed.

11:25 AM
Orientation Dependence of the High Cycle Fatigue Properties in an Al-Li 8090 Alloy: *Tongguang Zhai*[1]; Angus J. Wilkinson[2]; John W. Martin[2]; [1]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA; [2]University of Oxford, Matls., Parks Rd., Oxford OX1 3PH UK

High cycle fatigue properties of a hot-cross rolled Al-Li alloy plate (45 mm thick) were studied in the rolling, transverse and short transverse directions of the plate. The fatigue tests were conducted at a stress amplitude between 40%-100%sy, R=0.1, room temperature in air using a self-aligning four-point bend rig which was able to minimize all the possible misalignment commonly associated with a four-point bend test and allowed measurement of the fatigue property in the short transverse direction of the Al-Li alloy plate. It was found that the high cycle fatigue property in the short transverse direction was significantly inferior to those in the rolling and transverse directions. The crack in the short transverse samples were predominantly intergranular, whereas they were mainly crystallographic (along a 111 plane in each grain) in the rolling and transverse samples. The number of secondary cracks observed on the surface between the two loading bars varied markedly with the stress amplitude as well as the loading direction.

11:45 AM
Temperature Evolution during Fatigue: *Bing Yang*[1]; Peter K. Liaw[1]; Hsin Wang[2]; J. Y. Huang[3]; R. C. Kuo[3]; J. G. Huang[4]; [1]University of Tennessee-Knoxville, Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research (INER), PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, Nucl. Oper. Dept., Taipei 100 Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 0.5 Hz, 20 Hz and 1,000 Hz fatigue testing. Five stages of temperature evolutions were observed: an initial increase of the average specimen temperature, a followed temperature decrease, an equilibrium (steady-state) temperature region, an abrupt rise of the temperature, and a drop of temperature following specimen failure. The relationship among the temperature, stress-strain state, and fatigue behavior is discussed. Both thermodynamics and heat transfer theories are applied to model the observed temperature variation during fatigue. The predicted and measured temperature evolutions during fatigue were found to be in good agreement. Temperature evolution along the specimen gage length is predicted. Furthermore, the back calculation from the observed temperature to obtain inelastic deformation indicates the material damage during fatigue. Life prediction using measured temperature was performed.

Energy Issues in the Aluminum Industry – A Keynote Symposium

Sponsored by: Aluminum Association
Program Organizer: TMS, Warrendale, PA 15086 USA

Monday AM — Room: 602-603
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: John Allison, Ford Motor Company, Scientific Rsrch. Lab., MD 3182, Dearborn, MI 48124-2053 USA

8:30 AM Keynote
The Perspective and Affects of Energy and Industry: *Brett Wilcox*[1]; [1]Golden Northwest Aluminum, Inc.

For years, the aluminum industry and the Pacific Northwest have maintained a mutually beneficial relationship based on the region's supply of sufficient and affordable electricity until power shortages and subsequent increased pricing almost completely curtailed primary aluminum production.

9:00 AM Keynote
Dealing with the Energy Crises by Reducing Demand: *Paul E. Norman*[1]; [1]Power Business Line

In April of 2001, BPA was faced with the possibility of raising wholesale rates to Northwest utilities and large industries by 250 to 300 percent. High market prices were the reason. BPA knew that it had to limit its exposure to the market if it was going to get the rate increase down to double digits. In less than two months, using an aggressive load reduction strategy, BPA–with the tremendous cooperation from the region and a major contribution from the aluminum industry–reduced the rate increase to 46 percent.

9:30 AM Keynote
Energy Policy Position of the Aluminum Association: *Robin King*[1]; [1]The Aluminum Association

Energy represents about one third of the total production cost of primary aluminum. Electricity is an essential ingredient in primary aluminum production. These factors together make energy efficiency and energy management prime objectives for the industry. The Aluminum Association supports efforts to create an effective national policy that resolves the power-shortage crisis in the Northwest while sustaining and protecting the regional aluminum industry–accounting for almost 40 perfect of domestic primary production and 5 percent of world supply.

10:00 AM Break

10:20 AM Keynote
Aluminium and Energy–An International Perspective: *Richard Evans*[1]; [1]Alan Aluminium Limited and Alcan Fabrication Group

A review of the effects of regional energy and trade issues on the global aluminium industry and its markets and possible future international trends and scenarios.

10:50 AM Keynote
New and Emerging Technologies: *Jud W. Virden*[1]; [1]Energy and Technology Division, Pacific Northwest National Laboratory

How quickly does science fiction become science fact? You may be surprised, especially with the upcoming technologies to help increase your energy efficiency and reduce your energy usage. Jud Virden will speak on new and emerging technologies that will soon change where you could get your energy, how you build your buildings and what will power your equipment. Hear about fuel cells for distributed power systems and automobiles, miniature technologies, smart appliances and more–the stuff of science fiction coming to you soon!

11:20 AM Panel Discussion

Flyash: Generation, Treatment, Metal Recovery and Disposal - I

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; I. Gaballah, Laboratoire Environmental et Mineralurgie, Associated to CNRS, ENSG-LEM, Vandoeuvre les Nancy 54501 France; David G. Robertson, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-1460 USA

Monday AM — Room: 604
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Junji Shibata, Kansai University, Dept. Cheml. Eng., Osaka 564-8680 Japan; Ji-Whan Ahn, Korea Institute of Geoscience and Mineral Resources, Teajon, Korea

8:30 AM Invited
R&D for Recycle and Decontamination of Fly Ashes from Municipal Waste Incineration: *Nobuyuki Masuda*[1]; Nobuyuki Okamoto[1]; Eiichi Arai[1]; Kazuyuki Kikuta[1]; [1]Metal Mining Agency of Japan, 1-24-14, Toranomon, Minato-ku, Tokyo, 105-0001 Japan

In Japan, the municipal solid waste, which amounts to 50 million tons, is generated every year and most of it is incinerated. The bottom and fly ashes are disposed to the registered disposal areas under the provisions of The Waste Disposal and Public Cleaning Law. Especially, as the fly ash from the municipal waste incineration (the primary fly ash) contains heavy metals (lead, zinc, etc) and dioxins, it cannot be disposed directly without decontamination, such as melting, cementation, chelating and dissolving processes provided in the law. However, these procedures for decontamination, except melting, are not enough for the elimination of dioxins. Even in case of melting, the fly ash from the process (the secondary fly ash) contains high concentration of heavy metals (e.g., Zn; 1-20%, Pb; 1-10%). Because of these reasons, Metal Mining Agency of Japan (MMAJ) that is a semi-governmental organization started a four-year project to develop the treatment technologies of these fly ashes in 1999. The purpose of the project is to establish the integrated technologies to recover the valuable metals from, and to decontaminate, the primary and secondary fly-ashes in the practical scale by utilizing the existing metallurgical processes and facilities, along with the energy saving and the reduction of the environmental impact.

9:00 AM Invited
Fly Ash: A Resource Material for Zeolite Production: *R. N. Singh*[1]; S. S. Rayalu[1]; S. U. Meshram[1]; Pawan Kuwar[1]; M. Z. Hasan[1]; [1]National Environment Engineering Research Institute, Nehru Marg, Nagpur 440 020 India

India is bestowed with substantial coal reserves, with generally high ash contents (40-50%). Utilization of this coal, as a source of energy, especially in thermal power plants generates extremely high quality of fly ash. Realizing adverse effects of fly ash, stringent regulations for its disposal have been formulated. According to ASTM (D-5239-92) classification, Indian fly ashes generally fall in the class F type; wherein the sum of silica and alumina and iron oxide corresponds to a minimum of 75%. These fly ashes have been employed for a promising application in vogue viz. production of fly ash based zeolites (FAZs). Studies conducted on zeolite synthesis from fly ash of class F-type indicated that this particular fly ash is the most amenable material for synthesis of FAZs. Due to lack of sufficient information on this virtuous value added product from fly ash, the fly ash producers and users did not exploit this technology; which definitely has edge over other fly ash utilization technologies in vogue due to attractive profit margins. Fly ash based zeolite (FAZs) was synthesized and investigated for surface area, crystallinity, sorption capacity, calcium binding capacity, particle size, etc. Calcium binding capacities of FAZ-A and FAZ-Y are about 540meq/100 g and 420meq/100 g respectively. The crystallinity of FAZ-A and FAZ-Y is found to be 99% and 97% respectively in comparison with commercial standards. Surface areas of FAZ-A, FAZ-Y are 580 m2/g, and 480 m2/g, respectively. The presentation highlights innovative usage of fly ash, and hydrothermal chemistry of novel FAZ synthesis.

9:30 AM
Treatment and Recycling of Incinerated Ash using Thermal Plasma Technology: T. W. Cheng[2]; *Jinn P. Chu*[1]; C. C. Tzeng[3]; Y. S. Chen[1]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]National Taipei University of Technology, Dept. of Matls. & Minl. Resources Eng., No. 1, Sec. 3, Jung-Hsiao E. Rd., Taipei 106 Taiwan; [3]Institute of Nuclear Energy Research, Phys. Div., 1000, Wenhua Rd., Chia-An Village, Lung-Tan 235 Taiwan

Thermal plasma vitrification is a robust technology to treat and recycle the ash residues. Under the high temperature plasma environment, incinerated ashes are vitrified into benign slag with large volume reduction and extreme detoxification. Several one-step heat treatment processes are carried out in the present study at four temperatures (i.e. 850°C, 950°C, 1050°C and 1150°C) to obtain various "microstructure materials." The major phase to form these materials is a solid solution of gehlenite (Ca2Al2SiO7) and åkermanite (Ca2MgSi2O7) belonging to the melilite group. The physical and mechanical properties of the microstructure materials are improved by using one-step post-heat treatment process after plasma vitrification. These microstructure materials with good quality have great potential to serve as a viable alternative for construction applications.

9:50 AM Break

10:05 AM
Environmentally Friendly Usage Method of Zeolite Derived from Coal Fly Ash: *Junji Shibata*[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

Zeolite synthesized from coal fly ash has many intelligent properties such as cation exchange ability, adsorption ability and so on. The usage method of the zeolite, however, is not established in actual commercial scale yet. In this study, from the viewpoints of an effective usage for coal fly ash and a source recycling, we propose the new concept for environmentally friendly usage of zeolite, in which coal fly ash safely returns to the soil with growing plants and purifying wastewater. This method can make great contribution to recycle of coal fly ash, to purification of high nutritious water and to tree planting.

10:25 AM
Industrial Fly Ash as a Soil Amendment Agent for Raising Forestry Plantations: *Dinesh Goyal*[1]; K. Kaur[2]; R. Garg[2]; V. Vijayan[3]; S. K. Nanda[4]; A. Nioding[4]; Sunil Khanna[1]; V. Ramamurthy[2]; [1]School of Biotechnology, Thapar Inst. of Eng. & Tech., Patiala, 147 004 India; [2]Thapar Center for Industrial Research & Development India; [3]Institute of Physics India; [4]Ballarpur Industries, Ltd.

Industrial fly ash from (electro static precipitator) was mixed with soil at different levels on v/v basis for raising of pulpwood tree species. In field trials conducted on degraded soils increase in the plant growth of Eucalyptus tereticornis, Acacia auriculiformis and Casuarina equisetifolia by 10%, was observed during the initial six months after plantation at 18-24% (v/v) fly ash concentration which was at par with chemical fertilizer treatment. Level of As, Se and Mo in fly ash was 6.2, 3.6 and 4.0 ppm respectively. The radioactivity analyses of soil and fly ash showed that the activity levels of gamma emitting radionuclides 40K, 226Ra, 228Ac were within the permissible limits and mixing of fly ash with soil at tested concentration would have no deleterious effects. Available Pb, Co, Cr, Ni and Cd in rhizospheric soils were below detection limit. Fly ash at 10-20% level (v/v) was also found to be a good potting mix material in forestry nurseries in terms of improving biomass production by 20%, when tested in nursery seedlings of Eucalyptus tereticornis. Nursery addition of fly ash in soil decreased soil dehydrogenase activity and the soil bacterial population was negatively affected. These results indicate that use of fly ash in forestry sector involving nursery and plantations as a soil amendment agent up to 10-20% v/v is a gainful utilization of fly ash.

10:45 AM
Hydrothermal Synthesis and its Reaction Mechanism of K Type Zeolite from Coal Fly Ash using KOH Solution: *Norihiro Murayama*[1]; Hideki Yamamoto[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

Hydrothermal synthesis for K type zeolite is carried out from coal fly ash by using KOH as an alkali source. Through the overall reaction of zeolitization, the changes in various properties of obtained zeolite, such as the kind of zeolite crystal, cation exchange capacity, surface structure of zeolite, particle size distribution, concentration of various ion species in alkali solution and so on, are investigated in this study. From these results, the reaction mechanism of K type zeolite from coal fly ash can be clarified.

11:15 AM
Making Use of Fly Ash to Protect Environment–An Experience at Hindalco Industries, Ltd.: *A. K. Karmakarsr*[1]; [1]Hindalco Industries, Ltd., Renukoot, UP 231217 India

Hindalco Industries, Ltd., Renukoot is situated at the bank of Rihand Dam. This leads to stringent control of Fly ash disposal in the open area- by the side of the Dam. The co-generation plant at Renukoot is responsible to feed steam required to produce 4,50,00 MTPY alumina from "Low alumina-Bauxite" ore. This leads to the problems of disposal of 'Red-Mud' generated out of the alumin are finery and the Fly ash disposal from the Co-generation Plant. Primarily these disposable items are being used in the land filing of the Hill-area surrounding Renukoot. Looking into the disposal problems, Hindalco decided to change over the 'Wet Ash Disposal System' to Dry ash Disposal System and to utilize 'Dry Ash' -To protect environment from washing off the red-mud and fly ash, during rainy season, -To abide by the Regulation of fly ash disposal of Govt. of India. The fly ash from Electrostatic Precipitators is collected in silo under vacuum conditions. The fly ash, thus, collected is mainly used in Cement Plants located in near vicinity and also to develop land for vegetation. This article presents the chronological changes brought by the Co-generation Plant to use Fly ash and to protect environment by growing vegetation in the ash disposal area. This has yielded good results in protecting environment. This paper shares the experience of utilization of fly ash at Hindalco Co-generation Plant.

11:35 AM
Simultaneous Removal Technology of N and P Components in Aqueous Solution by Ca Type Zeolite Synthesized from Coal Fly Ash: *Hideki Yamamoto*[1]; Norihiro Murayama[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka, 564-8680 Japan

The simultaneous removal for NH4+ and PO43- in aqueous solution is carried out by using Ca type zeolite synthesized from coal fly ash. The effect of pH on cation exchange capacity and adsorption properties of obtained zeolite, the simultaneous removal ability for NH4+ and PO43- in various solutions and the mechanism of simultaneous removal were investigated, respectively. The Ca type zeolite used in this study can simultaneously remove NH4+ and PO43 in aqueous solution without releasing co-ion, which gives great advantages over the other ordinary removal methods.

Fundamentals of Advanced Materials for Energy Conversion: Superconductors

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

Monday AM	Room: 613
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Amit Goyal, Oak Ridge National Laboratory, PO Box 2008, MS 6116, Oak Ridge, TN 37831-6116 USA; Renato G. Bautista, University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

8:30 AM Plenary
An Overview of Advanced Materials for Energy Conversion: *Dhanesh Chandra*[1]; Renato G. Bautista[1]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

An overview of advanced materials for energy conversion and storage will be presented. In this symposium, emphasis will be given to superconducting, magnetic, fuel cells/membrane, hydrogen and tritium storage, battery, and thermal energy storage materials. The advances in second-generation superconductors for large-scale applications, magnetic materials for heating and cooling used in home air conditioners and refrigerator/freezers, thermoelectric power developments for electronic applications, and battery materials will be presented. Advances in core materials for transformers and motors using amorphous (glassy) ferromagnetic glasses rather than crystalline Fe-Si alloys, which show very low coercivity and hysteresis losses have recently received considerable attention. Several advances have been made in the development of fuel cells, membrane materials, interconnects for high temperature planar SOFC's. Hydrogen and tritium storage in intermetallic and other complex hydrides look very promising; recent advances will be discussed. In addition, lightweight alanate hydrides have shown significant improvements. The fundamental understanding of emerging Clathrate hydrates materials for solid-state methane storage, and organic thermal energy storage materials will also be discussed.

8:50 AM Plenary

Second Generation High Temperature Superconducting (HTS) Wires for Large-Scale HTS Devices: *Amit Goyal*[1]; [1]Oak Ridge National Laboratory, PO Box 2008, MS 6116, Oak Ridge, TN 37831-6116 USA

A review of advances made towards the development of second generation superconducting wires which are necessary for most large-scale, bulk applications of high temperature superconducting (HTS) materials will be given. Particular emphasis will be given on wires made using the rolling-assisted-biaxially-textured-substrates (RABiTS), a technique to fabricate long lengths of flexible, single-crystal-like, HTS wires. The technique involves creating a single-crystal-like, biaxially textured metal substrate which can be done using simple rolling and annealing procedures. This is followed by epitaxial deposition of oxide buffer layers and superconductors. Superconducting properties of wires made using the second generation wire technology approach those of single-crystal superconductors. Such wires appear to be very promising for many large-scale applications involving superconducting devices.

9:20 AM Invited

Superconducting Electric Power Applications: HTS Conductor Requirements: *Richard Blaugher*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

Recent worldwide successes in demonstrating high-temperature superconductors (HTS) in electric power apparatus have buoyed the prospects for this technology. These achievements were only possible due to the rapid worldwide progress in developing HTS wire and tape. This talk will discuss the overall progress in developing superconducting electric power components. The early LTS work on energy storage (SMES) and electric power generators will first be reviewed followed by a discussion on current programs using HTS conductor. The major problems facing this technology will also be discussed, as well as the prospects for commercialization and integration into the utility sector. The eventual widespread utility acceptance for superconducting power equipment is dependent on demonstrated improvements in system performance and efficiency. The reliability and maintenance must also be comparable to conventional power equipment and offer reduced life-cycle costs and a "competitive" installed cost. The latter is impacted by the current high cost of HTS conductors that must be lowered to costs comparable to conventional copper wire. This cost factor, coupled with the requirement for improved HTS transport performance in magnetic fields greater than one tesla at 77K, are the critical factors that will limit or seriously compromise the consideration of HTS power components for the power sector. (Supported by the Department of Energy Superconductivity Program for Electric Power Systems.)

9:45 AM Invited

Flywheel Electricity Storage System using High Temperature Superconductors: *Michael Strasik*[1]; [1]Boeing, PO Box 3707, Seattle, WA 98124-2207 USA

Boeing Phantom Works, Seattle, Washington, and six partners are developing a 10-kWh flywheel energy storage system using superconducting bearings. The system will help utility companies and other large power users eliminate power surges that often disrupt reliable transmission of power. Flywheels based on frictionless superconducting bearings improve power quality and reliability. Boeing has also had a leading role in the push to use flywheels in satellites and to replace batteries in the International Space Station. The reason is because flywheels wont wear out like batteries. Batteries fail after a limited number of charge/discharge cycles. By simply replacing batteries with flywheels, you can extend the life of some satellite systems. A proof-of-concept system has been designed, built and tested.

10:10 AM Break

10:25 AM Invited

Operation of the Southwire 30-m High Temperature Superconducting Cable Installation: *David T. Lindsay*[1]; R. L. Hughey[1]; Mark Roden[1]; Uday Sinha[1]; Mike Gouge[2]; John Stovall[2]; [1]Southwire Company, Wire & Cable Tech., One Southwire Dr., Carrollton, GA 30119 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Southwire Company of Carrollton, Georgia in cooperation with Oak Ridge National Laboratory has designed, built, installed and is operating the world's first field installation of a High Temperature Superconducting (HTS) cable system. The cables supply power to three Southwire manufacturing facilities at the company's main campus in Carrollton, GA. The system consists of three 30-m single phase cables rated at 12.4 kV, 1250 Amps, liquid nitrogen cooling system, and the computer-based control system. The cables are built using BSCCO-2223 powder-in-tube HTS tapes and a proprietary cryogenic dielectric material called CryoflexÔ. The cables are fully shielded with a second layer of HTS tapes to eliminate any external electric fields. The Southwire HTS cables were first energized on January 6, 2000. To date, they have logged over 8,200 hours of operation while supplying 100% of the required customer load. The cables have worked without failure and operations are continuing.

10:50 AM Invited

High Temperature Superconductor-Based HTS Electric Power Devices: *Venkat Selvamanickam*[1]; [1]IGC-SuperPower, 450 Duane Ave., Schenectady, NY 12304 USA

IGC-SuperPower is developing High-Temperature Superconductor (HTS)-based products for the electric power industry, specifically cables, transformers, and fault-current controllers. These HTS devices are expected to result in higher efficiency, lower losses, increased capacity, and generate environmental benefits. The HTS conductor that will be used in all these devices is a high-performance coated conductor fabricated by thin film processes. GC-SuperPower has established pilot-scale facilities for scale up of Coated Conductor processes to manufacturing. These facilities have been designed for continuous processing of substrates, buffer layers, and superconducting layers over time periods of a week. These facilities are being used to scale up coated conductor technology to manufacturing. The overall activities on scale up of Coated Conductor processes at IGC-SuperPower and the progress with HTS devices for electric power will be summarized in this presentation. Part of the work was performed under a CRADA with Los Alamos and Argonne National Labs.

11:15 AM Invited

Integrated Momentum and Energy Storage for Mini-Satellites: *Ki Bui Ma*[1]; Yong Zhang[1]; Yevgeniy Postrekhin[1]; Wei-Kan Chu[1]; [1]University of Houston, TX Ctr. for Superconductivity, 3201 Cullen Blvd., Houston, TX 77204-5002 USA

Momentum wheels with superconducting bearings can integrate both attitude control and energy storage functions, with attendant savings in the total volume and weight of hardware, and power consumption, all of which are in short supply on mini-satellites. We have built and tested a prototype with a wheel of mass 1.9 kg, diameter 3.25 in, and height 3 in, which can store 3.5 J-sec of angular momentum and 5 kJ of energy when it rotates at an angular speed of 15000 RPM. The energy density of 2.5 kJ/kg achieved is comparable with the lower range for lead-acid batteries. To show that the wheel is energy efficient, we have performed spin down tests and measured the input power required to sustain rotational speed. Results indicate that power consumption, even accounting for the needs of cooling system, is significantly smaller than that for state of the art commercial reaction wheels.

11:40 AM Invited

Crystal Lattice Defects, Nanoscale Structure, and the Properties of Advanced Superconductors: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Matls. & Cheml. Scis. Div., Bldg. 480, PO Box 5000, Upton, NY 11973-5000 USA

Optimization of the properties, especially the current carrying capacity, of advanced superconductors requires a very high level of control of crystal lattice defects and their organization into nanoscale structure in order to minimize the effects of weak links and maximize the pinning of magnetic flux. This degree of control gives rise to the need for a deep understanding of the effects of defects on charge distributions, elastic strain fields, as well as local atomic and electronic structure. A combination of modern methods of electron microscopy, microscale transport property measurements, and theoretical analysis facilitates the achievement of such understanding. I will use results from such an approach to contrast the character and effects of defects in high-Tc cuprates and the latest addition to the family of advanced superconductors, the intermetallic compound MgB2. This research was supported by the US Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

Fundamentals of Structural Intermetallics: Environmental Effects and Fe Aluminides

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

Monday AM	Room: 615-616
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Ronald Gibala, University of Michigan, Dept. of Matls. Sci. & Eng., 2026 H.H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; Seetharaman C. Deevi, Chrysalis Technologies Inc., Rsrch. Ctr., 7901 Whitepine Rd., Richmond, VA 23237 USA

8:30 AM Opening Remarks

8:40 AM

Moisture-Induce Hydrogen Embrittlement of Ordered Intermetallics: *Chain T. Liu*[1]; Easo P. George[1]; Lee M. Pike[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., #1 Bethel Valley Rd., Oak Ridge, TN 37831 USA; [2]Haynes International, Eng. & Tech., Kokoma, IN 46904-9013 USA

This paper summarizes recent advances in understanding moisture-induced environmental embrittlement in intermetallics. The embrittlement occurring at ambient temperatures is quite different in fcc- and bcc-ordered intermetallics. It causes brittle intergranular fracture without affecting the yield strength of intermetallics with the L12 structure. On the other hand, bcc-ordered alloys exhibit cleavage fracture and reduced yield strength when tested in moist air. The change in yield strength may not be detected in some cases because of excess point defects induced in bcc-ordered alloys. Hydrogen diffusion along the grain boundary and bulk is responsible for the different embrittlement behaviors observed in fcc- and bcc-lattices. This research was sponsored by US DOE Division of Materials Sciences and Engineering under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:10 AM

Surface Chemistry of Adsorbates on Intermetallics: *Masahiro Inoue*[1]; Katsuaki Suganuma[1]; [1]Osaka University, The Inst. of Scientific & Indl. Rsrch., 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

The surface modification technology is expected as one of the useful techniques for extending material's lifetime because the environmental degradation of intermetallics and related materials is initiated by surface reaction of adsorbates. In order to establish the techniques for extending lifetime, the surface chemistry is necessary to understand in detail. This paper discusses the adsorption behavior of environmental species on intermetallic alloys. The adsorption usually occurs through the steps; back donation from the intermetallics, followed by relaxation and decomposition of adsorbates. The adsorption mechanism of these species on aluminides is studied using the molecular mechanics and first principle molecular orbital simulation.

9:30 AM

Water Dissociation on Single Crystal Ni3(Al,Ti) and (Ni,Fe)Ti Surfaces: Monica de Mesquita Lacerda[1]; Jinliu Wang[1]; *Yip-Wah Chung*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

While the mechanism of moisture-induced embrittlement in polycrystalline Ni3Al is well understood, the role of boron to suppress this embrittlement is less clear. In this work, temperature-programmed desorption and x-ray photoelectron spectroscopy were carried out after D2O dosing on clean Ni3(Al,Ti) (110) surface at < 130K, with and without surface boron. Water dissociates on clean surfaces at ~190 K, resulting in D2 evolution at ~400K. This D2 evolution is completely suppressed by ~0.30 monolayer boron. We found that atomic hydrogen is strongly bound to surface boron. Surface diffusion measurements showed that the surface diffusivity of atomic hydrogen is reduced by 10 times by 0.05 monolayer boron. This should lead to lower concentration of atomic hydrogen at the crack tip, thus explaining the improvement of ductility. We have extended the same surface science approach to understand the embrittlement of NiTi alloys due to Fe addition beyond 9 a/o.

9:50 AM

High Temperature Corrosion of Fe3Al in Chlorine-Containing Environments: *Gilsoo Han*[1]; W. D. Cho[1]; [1]University of Utah, Dept. of Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The high-temperature corrosion of iron aluminide (Fe3Al) in environments containing chlorine and oxygen has been studied using thermogravimetric method. The corrosion experiments were carried out in various oxygen to chlorine volume ratios at the temperature range of 750-900°C. The corrosion behavior in the mixed gas was observed to be significantly different from that in pure oxygen in terms of kinetics and corrosion products. The cross-section of the specimens and the corrosion products were examined using X-ray diffraction, scanning electron microscopy, and energy-dispersive X-ray spectroscopy. Based on the analysis of kinetic data and corrosion products, the corrosion mechanism has been discussed.

10:10 AM Invited

Yield Strength Anomaly in FeAl Single Crystals: *E. P. George*[1]; D. M. Wu[2]; I. Baker[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6093 USA; [2]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA

FeAl alloys exhibit the so-called yield strength anomaly. We report here the effects of orientation in B-free and B-doped FeAl single crystals. The single crystals were grown by the Bridgeman technique and oriented for single slip to facilitate determination of the critical resolved shear stress (CRSS). Yield strength was measured in tension as a function of temperature from 77-1173K. Optical and scanning electron microscopy were used to confirm that only a single slip system was active during plastic flow. The CRSS was plotted as a function of temperature for different single-slip orientations to determine if there is, in fact, an orientation dependence of the yield strength or if all orientations fall on one curve. The implications of these results for our vacancy-hardening theory [E. P. George and I. Baker, Phil. Mag. A, 77, 737-750 (1998)] are discussed. Research sponsored at ORNL by the Division of Materials Science and Engineering, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and at Dartmouth College by NSF grant DMR-9973977, by DOE contract DE-FG02-87ER45311, and by the Oak Ridge Associated Universities SHaRE program contract DE-AC05-76OR00033.

10:40 AM

Strain Rate Effect on the Anomalous Temperature Dependence of Flow Stress in a Fe-24at.%Al Intermetallic Alloy: *Tae Kwon Ha*[1]; Jin-Hwa Song[2]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Ctr. for Adv. Aeros. Matls., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea; [2]Research Institute of Industrial Science and Technology, Reliability Assessment Ctr., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-600 S. Korea

In this study, the effect of the strain rate on the anomalous temperature dependence of flow stress was mainly investigated in a Fe-base intermetallic alloy with the composition of Fe-24 at.% Al. Phase composition and degree of order in Fe-24 at.% Al alloy were varied by aging at temperatures corresponding to the various phase fields of $D0_3$, α+D03, α+B2, and B2. The room temperature deformation behavior of the Fe_3Al alloys with the various phase compositions and degree-of-orders has also been examined. Load relaxation tests and tensile tests were conducted at temperatures ranging from room temperature to 800°C and the results were analyzed based on the internal variable theory of inelastic deformation. Room temperature strength of the Fe_3Al alloy appeared to increase with decreasing degree of order and the hardening effect caused by the precipitation of disordered α phase was found to depend on the shape of the precipitate. While the α phases in the α+$D0_3$ region precipitated in the form of a network were found to have a strengthening effect, those in the α+B2 region precipitated in the form of a coarse island had no effect of strengthening. The room temperature strength of the Fe_3Al alloy increased with decrease in the degree of order. The peak strength was observed at higher temperatures with the strain rate increased. The maximum strength was obtained at 530°C, which is right below the $D0_3$-B2 transition temperature Tc of 550°C, under the strain rate of $5X10^{-2}$ s^{-1}. By employing the internal variable theory of inelastic deformation, flow stress of the Fe_3Al alloy could be described as the sum of contributions from internal stress and frictional stress. The anomalous temperature dependence of strength in the Fe_3Al alloy was mainly attributed to the internal stress.

11:00 AM

Mechanical Behavior of $L2_1$-Structured Compounds Fe_2AlMn and Fe_2AlTi: *I. Baker*[1]; M. Wittmann[1]; S. L. Johns[1]; V. N. Durand[1]; P.

R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA; [2]University of New South Wales, Matls. Sci. & Eng., Sydney, New South Wales NSW 2052 Australia

Single crystals of Fe_2AlMn were grown and confirmed, by X-ray diffraction, to have the $L2_1$-structure. Mechanical tests showed that the compound exhibits a yield strength anomaly and tensile ductility at room temperature, which depend on strain rate. Transmission electron microscope analyses of strained specimens showed that deformation occurs by glide of <111> dislocations at all temperatures. Complex anti-phase domain structures, some with a/4<111> and other with a/2<100> fault vectors, were also observed, consistent with the compound ordering from a b.c.c. and then a B2 structure. Tests have also been performed on polycrystals of the related compound Fe_2AlTi as a function of temperature. The results will be compared to FeAl and both Co- and Ni-based Heusler alloys, and the origin of the yield anomaly will be discussed. This work was funded by NSF grant DMR-9812211.

11:20 AM

Effect of Alloying Additions on the Strength and Creep Resistance of FeAl Alloys: *R. S. Sundar*[1]; D. H. Sastry[2]; S. C. Deevi[1]; [1]Chrysalis Technologies, Inc., 7801 Whitepine Rd., Richmond, VA 23237 USA; [2]Indian Institute of Science, Dept. of Metall., Bangalore India

Iron aluminides based on FeAl are well known for their excellent oxidation and sulfidation resistances. They are being developed to replace conventional steels and stainless steels for intermediate temperature applications. Due to their open B2 structure, binary FeAl exhibits significant creep deformation at temperatures greater than 700C. Alloying elements are necessary to improve the high temperature deformation resistance of FeAl. The present study aims to bring out the effect of various substitution and interstitial alloying elements on the strength and creep resistance of FeAl alloys. Carefully planned experiments reveal that the importance of processing and heat treatment conditions on the properties of FeAl alloys.

11:40 AM

Superplasticity of Coarse-Grained Iron Aluminides: Thermal Cycling Effects: *Jinn P. Chu*[1]; W. Kai[1]; H. Y. Yasuda[2]; Y. Umakoshi[2]; K. Inoue[3]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Osaka University, Dept. of Matls. Sci. & Eng., 2-1, Yamada-oka, Suita, Osaka 565-0871 Japan; [3]University of Washington, Dept. of Matls. Sci. & Eng., Seattle, WA 98195 USA

As a general rule, superplastic metals and ceramics normally have fine-grained structures (typically <10 µm). Nevertheless, Fe-Al based alloys have been shown superplasticity even with much coarser grain structures, >500 µm. Fe-Al based alloys have been demonstrated to exhibit all the deformation characteristics that conventional fine-grained superplastic materials possess. The objective of the present study is directed toward the microstructural examination of thermal cycle effects on superplastic properties of Fe-27Al alloys. To visualize the grain-structure evolution, the electron backscattered diffraction technique has been employed because it is capable of identifying the type of grain structures. The superplastic behavior will be presented and discussed in light of the microstructure/crystal structure results.

General Abstracts: Joining, Testing and Characterization

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

Monday AM — Room: 310
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: John J. Stephens, Sandia National Laboratory, Albuquerque, NM 87185-0889 USA

8:30 AM

A Study of Active Metal Braze Alloys Based on the Ag-Hf Binary System: *John J. Stephens*[1]; F. Michael Hosking[1]; Fred G. Yost[2]; [1]Sandia National Laboratories, 1833, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA; [2]Trapezium Technology, 1901 Cleopatra Ct. N.E., Albuquerque, NM 87112 USA

This talk will present the results of an experimental project which has examined the potential of two binary Ag-Hf alloys for use as active metal braze alloys. The composition (wt.%) of these alloys are 97.5Ag-2.5Hf and 96.6Ag-3.4Hf. Due to the absence of a complete binary phase diagram for Ag-Hf in the literature, the compositions were chosen by analogy with other, related binary phase diagrams. Due to the favorable thermodynamic tendency for Hf to react with a number of ceramic substrates, both alloys show considerable promise for metal/ceramic brazing based on initial wetting and ASTM F19 tensile button results. The microstructure and products of the reactions observed at the braze/ceramic interface will be compared those observed in other active metal braze/ceramic interfaces. This work was supported by the US Dept. of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Department of Energy.

8:55 AM

Fracture Surface and Failure Mode Analysis of Polycrystalline Silicon: *Ryan C. Brodie*[1]; Marian S. Kennedy[1]; David F. Bahr[1]; [1]Washington State University, Mechl. & Matls. Eng., 201 Sloan Hall, Pullman, WA 99164-2920 USA

Disk-shaped compact tension (DCT) specimens made from CVD grown polycrystalline silicon were fabricated using water jet methods. Compliance and fracture behavior were tested as a function of temperature, from ambient up to 1100 K. The fracture surfaces of the DCT specimens were then analyzed using SEM to identify any possible variations in fracture mode associated with increasing temperature. Such variations include a change from transgranular to intergranular failure, and changes in crack initiation mechanisms. Using the load and displacement data along with the SEM fractography, the brittle-to-ductile transition (BDT) temperature for polycrystalline silicon can be estimated. The effects of elevated temperature annealing, altering the grain size from between 0.5 and 5 microns, are also discussed. Comparisons between the DCT specimens and indentation induced fracture on annealed specimens tested at room temperature will be shown.

9:20 AM

Use of Advanced Scattering and Microtomography Techniques to Study the Complex Anisotropic Microstructures of Ceramic and Metallic Deposits: *Jan Ilavsky*[1]; Andrew J. Allen[2]; Gabrielle G. Long[2]; Anand Kulkarni[3]; Francesco De Carlo[4]; Herbert Herman[3]; [1]University of Maryland, UNICAT, Bldg. 438E, 9700 S. Cass Ave., ANL, Argonne, IL 60439 USA; [2]National Institute of Standards and Technology, Matls. Microstructure Characterization Grp., 100 Bureau Dr., MS 8523, Gaithersburg, MD 20899 USA; [3]State University of New York, Dept. Matls. Sci. & Eng., Old Engineering, Z=2275, Stony Brook, NY 11794-2275 USA; [4]Argonne National Laboratory, Experimental Facilities Div., Bldg. 431, B003, 9700 S. Cass Ave., ANL, Argonne, IL 60439 USA

Thermal-spray (TS) and electron-beam plasma-vapor deposited (EB-PVD) coatings possess unique multicomponent void microstructures which are responsible for their complex engineering properties, but which also pose major challenges for their characterization. Multiple void systems with different anisotropies, wide range of void shapes and sizes, and general deposit fragility all present major obstacles to many characterization methods. Often used intrusion porosimetry and imaging of cross-sections, provide either only basic volumetric data or information that is limited in reproducibility and reliability. In contrast, a combination of small-angle scattering of X-rays and neutrons (SAXS and SANS) and X-ray microtomography (XMT) provides a robust, nondestructive, statistically-valid, and comprehensive, microstructure characterization. SAXS and SANS can be used to characterize quantitatively the void systems through an analysis of the apparent Porod surface area distributions and yield the calibrated total specific surface area of the voids. The techniques of anisotropic multiple SANS and SAXS can yield more volumetric information about the void systems, via the introduction of model assumptions. XMT provides a complementary 3D view of the undisturbed structure of these materials. The improved resolution of this technique makes it a viable tool for providing density map data which, when combined with SANS and SAXS, can provide microstructure characterization of complex materials from the nanometer up to the millimeter-scale. Examples of recent application of this combination of techniques to TS and EB-PVD coatings will be presented, and it will be shown how the results can be related to the coating properties.

9:45 AM Break

10:20 AM
Framework for Designing Interlayers for Ceramic-to-Metal Joints: *Jin-Woo Park*[1]; Patricio F. Mendez[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-047, Cambridge, MA 02139 USA

This work introduces a framework for evaluating the strength characteristics of ceramic-to-metal joints with multiple interlayers. Strain energy in the ceramic is used as a strength metric instead of maximum tensile stress. Based on the FEM analysis and order of magnitude scaling (OMS), simple analytical formulations between the strain energy and material properties are developed, which provide a guideline in designing multiple interlayers. Our analysis reveals the important role of multiple interlayers, which reduce the strain energy in the ceramic, increasing the strength of the joint. Based on the proposed design rule, Si3N4 to Inconel 718 joints have been brazed with single, double and triple interlayers and the joint strength was evaluated using a shear test. The experimental results support the design rules and confirm that strain energy is a good strength metric.

10:45 AM
Characterization of Photovoltaic Cell Solder Joints: *F. Michael Hosking*[1]; Michael A. Quintana[2]; [1]Sandia National Laboratories, PO Box 5800, MS0889, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, PO Box 5800, MS0752, Albuquerque, NM 87185-0752 USA

Photovoltaic silicon cells are electrically connected by soldering copper strips to a deposited metal grid network. System performance depends on the reliability of the resulting solder joints. Typical soldering materials and processing conditions are reviewed. Nondestructive and metallographic test results are presented for a variety of soldered modules, including field-tested units. The coring technique for obtaining test samples is described. Factors that impact module durability and efficiency are discussed. Of particular concern to photovoltaic solder joints are extended exposure to hot, humid field conditions and temperature extremes. The effects of moisture, elevated operating temperatures, and thermal cycling on corrosion, intermetallic growth, and mechanical fatigue failures are examined. The formation of shorts or opens in the photovoltaic circuit can rapidly degrade module electrical efficiency and field life expectancy. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy under Contract DE-AC04-94AL85000.

11:10 AM
Non-Destructive Evaluation of Ceramic Candle Filters using Artificial Neural Networks: *Kuan-Chen Fu*[1]; Yanqin Zhou[1]; Lijun Li[1]; [1]The University of Toledo, Civil Eng., 2801 W. Bancroft St., Toledo, OH 43606 USA

Ceramic candle filters play an important role in coal-base turbine system for a modern power plant. However, after exposure to the high pressure and high temperature in the gas turbine chamber, the physical property of the ceramic deteriorates over time and the effectiveness of the filters reduces. Their failure to perform may create catastrophic consequences for the multi-million dollar equipment downstream. A non-destructive evaluation procedure using an emerging technique known as artificial neural networks is proposed to examine the filters. In lieu of experimental data, the vibration signatures of filters damaged to various degrees are created by means of analytical simulation. Then, a feed-forward artificial neural network and a radial basis function neural network are built and trained to evaluate the signatures for the purposes of determining the filters' degree of deterioration. Excellent results have been obtained. The technique may be applied to evaluate structural components made of aluminum, alloy or other materials.

11:35 AM
Temperature Discontinuity Assumption at Solid-Liquid Interface in Rapid Cellular Solidification: *Eisaku Tokuchi*[1]; Kimioku Asai[1]; [1]Musashi Institute of Technology, Mechl. Eng., 1-28-1 Tamazutumi, Setagaya-ku, Tokyo 158 Japan

On the basis of experiments, a calculation method to predict all the solidification conditions and dendrite arm spacings of rapidly solidifying cells is proposed. The assumptions are no solute pile-up at cell tip and temperature discontinuity at solid-liquid interface. The calculation results theoretically evidenced temperature none-uniformity in the cross-sectional area of a cell and the adjoining liquid, and thus validated the no-pile-up assumption by way of thermodynamic restrictions of rapid cellular thickening that is a dynamic behavior at solid-liquid interface along with the temperature discontinuity. The calculation results are also in favor of limited selection of solidification conditions in a given rapid cellular solidification regime. The method was validated in principle for use in the rapid cellular solidification range less than 7ìm of dendrite arm spacing.

General Abstracts: Microstructural Phase Stability

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

Monday AM	Room: 211
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Dan J. Thoma, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM
Grain Refinement of Cast p-Type Bi2Te3 Alloy by Severe Plastic Deformation: *Jae-taek Im*[1]; *Karl Ted Hartwig*[1]; Jeff Sharp[2]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77843-3123 USA; [2]Marlow Industries, Inc., 10451 Vista Rd., Dallas, TX 75238-1645 USA

The objective of this work is to refine the microstructure of cast p-type Bi2Te3 alloy by severe plastic deformation using multipass equal channel angular extrusion (ECAE). Successful extrusions are accomplished at a temperature of 500C and a rate of 4.23 mm/sec. The microstructure is characterized by polarized optical microscopy. The level of grain refinement is found to be dependent on total strain. The results indicate that ECAE processing is a viable method for grain refinement of cast Bi2Te3 alloy.

8:55 AM
Microstructure and Annealing Behavior of Cold Deformed Copper: *Mohammed Haouaoui*[1]; Karl T. Hartwig[1]; [1]Texas A&M University, Mechl. Eng., 319 Engineering Physics Bldg., Spence St., College Station, TX 77843-3123 USA

CDA 101 copper was deformed at room temperature to a strain of 4.6. 25.4 by 25.4 mm square copper bars were deformed by equal channel angular extrusion through four passes in a tool containing a 90° angle at a strain rate of approximately 2 per second. The nucleation of new grains from heavily deformed material was found to occur along sites with heavy distortions. Nucleation sites for recrystallized grain start in shear bands with subsequent growth in the direction of slip lines. The annealing behavior is found to be a function of the multipass extrusion route. Processing that causes intersection of shear planes creates more sites for nucleation and leads to a shift to lower recrystallization temperatures. All annealing curves exhibit a slight increase in hardness prior to the sharp drop that accompanies recrystallization. Optical microscopy reveals uniform recrystallized microstructures for the different routes.

9:20 AM
In-Situ Annealing Observations during Epsilon-to-Tau Phase Transformation in MnAl-Based Ferromagnetic Alloys: Cagatay Yanar[1]; Eric A. Stach[1]; *William A. Soffa*[1]; Jörg M.K. Wiezorek[1]; [1]LBNL, NCEM, Berkeley, CA USA; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Mn-Al alloys of nearly equiatomic composition show a metastable ferromagnetic tau-phase (L1o) after suitable heat treatment, the magnetic properties of which are extremely sensitive to microstructure and defect structure. The formation of these complex defect structures has been attributed to atomic processes occurring at the migrating interphase interfaces during the "massive transformation" of tau-phase from the high-temperature epsilon-phase (A3). Models for the defect formation have been proposed. In this study, in-situ TEM annealing experiments have been performed to elucidate the details of the epsilon-to-tau transformation and defect genesis. Dynamic observations of the formation of twins, pseudo-twin and other defects are related to the proposed models. An additional mode of epsilon-to-tau transformation, namely a displacive transformation, was observed during in-situ TEM. This alternate displacive transformation and the massive mode compete during the in-situ experiments. Local stresses are believed to trigger the displacive mode. The role of these observations for microstructural control in tau-phase alloys is discussed.

9:45 AM
Critical Phenemona at the Martensitic Transition in the Shape-Memory Alloy Gold-Zinc: *Jason C. Lashley*[1]; Timothy Darling[1]; D. J. Thoma[1]; Albert Migliori[1]; F. Chu[1]; M. Lopez[1]; B. Lang[2]; Juliana Boerio-Goates[2]; Brian F. Woodfield[2]; [1]Los Alamos National Laboratory, Matls. Sci. Div., PO Box 1663, Los Alamos, NM 87545 USA; [2]Brigham Young University, Dept. of Chem. & Biochem., Benson Bldg., Provo, UT 84602 USA

Since the discovery of the shape-memory effect, the martensitic transition has been described within the framework of classical equilibrium thermodynamics as a first-order displacive transition. However, as we investigate the physical properties (elastic moduli, specific heat, and stress/strain measurements) through the martensitic transition in AuZn at cryogenic temperatures, we find clear signatures of recoverable plastic strain and a continuous (second-order) transition at 64.7 K. It is argued that the combination of equiatomic composition (removing internal strains) and a low transition temperature (reducing both diffusion and entropy effects) constrain the chemical potential and its derivatives to exhibit behavior that lies at the borderline between that of a first-order (discontinuous) and a continuous phase transition. For these reasons, we propose a critical point in composition-temperature space located at mole fraction, x = 0.5 Zn and T ~ 65 K, connecting two coexistence lines of first-order martensitic phase transitions. Further support of the critical point is based on resistivity data of (Pops and Ridley, 1970), cold-stage optical results of (Pops and Massalski, 1965), de Haas-van Alphen measurements of (Beck et. al., 1963).

10:10 AM
Nucleation of Stress-Assisted Martensite: *R. E. Hackenberg*[1]; J. C. Cooley[2]; K. C. Chen[3]; D. J. Thoma[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G770, Los Alamos, NM 87545 USA; [3]California Polytechnic State University, Dept. of Matls. Eng., San Luis Obispo, CA 93407 USA

Many unresolved questions exist regarding the nucleation of martensite, due to the small time and length scales over which this process occurs. Although shock-loading experiments done at T>Ms have clarified some aspects of martensite growth, the gas-gun techniques traditionally used have lacked the temporal resolution to bracket the initial nucleation event apart from autocatalytic nucleation of additional units from fully-grown martensite units. This study reports the use of much shorter duration (nanosecond scale) stress pulses generated by an ultrafast laser, which allow only partial growth of martensite units to be realized, thus avoiding autocatalytic effects. Progressive reduction in the pulse duration allows the nucleation site to be more clearly identified; TEM is employed to this end. Polycrystal and single crystal specimens are examined to assess the nucleation potency of grain and twin boundaries. Preliminary results will be presented for near-equiatomic Ni-Ti and ferrous alloys.

10:35 AM
Phase Relations in the Ti-Pt System in the 30 to 60 at.% Pt Region: *T. Biggs*[1]; L. A. Cornish[2]; M. J. Witcomb[3]; M. B. Cortie[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 6350 Stores Rd., Vancouver V6T 1Z4 Canada; [2]Physical Metallurgy Division, Mintek, PB X3015, Randburg, 2125 S. Africa; [3]University of Witwatersrand, Electron Microscope Unit, PB 3, Wits, 2050 S. Africa

In the titanium-platinum system, around compositions of 50 at.% platinum, a phase transformation can be observed from a B2 parent phase to an orthorhombic product. This TiPt phase transformation occurs around 1000C and is displacive in nature. The transformation temperature and hardness of the alloy varied with composition, reaching a maxima and minima respectively, at 50 at.%. The morphology and hardness of the orthorhombic phase varied with cooling rate. The morphology could be lath-like or finely twinned depending on the cooling rate. At lower platinum contents a novel phase, which has not been reported before, was observed. Investigations suggested that the phase formed peritectically around 1200C. The TiPt phase transformation was observed to continue in the neighbouring two-phase regions, at high (TiPt and TiPt3) and low platinum contents (TiPt and a novel phase). At even lower platinum contents, the Ti3Pt phase and the novel phase were observed to form eutectically.

11:00 AM
Microstructure and Phase Chemistry of IN 100: Characterization and Phase Calculations: *Agnieszka Wusatowska-Sarnek*[1]; Gautam Ghosh[2]; Mark Aindow[1]; Martin Blackburn[1]; Gregory B. Olson[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Eng., Inst. of Matls. Sci., 97 N. Eagleville Rd., Storrs, CT 06269-3136 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA

The fidelity of thermodynamic calculations of phase stability and constitution continues to improve rapidly and it is now possible to simulate the behavior of complex multi-component systems. In the current work the results of a comprehensive analysis of the structure of a powder metallurgical superalloy IN100 are compared with the results of such computations. A variety of analytical methods was used to establish quantitatively the volume fractions and chemical compositions of phases after complex heat treatments resulting in a tri-modal distribution of gamma' particles. Thermodynamic computations were made using the Ni-database developed by Saunders and Thermo-Calc software systems. The predictions of primary phase constitution were found to be in excellent agreement with the measurements. Carbide and boride phases were also predicted accurately. Partition coefficients for most elements were close to calculated values, but Mo and Ti show more deviation. Some kinetic features of the achievement of equilibrium will be examined.

General Pyrometallurgy - I

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee
Program Organizer: Adrian C. Deneys, Praxair, Inc., Tarrytown, NY 10591 USA

Monday AM — Room: 601
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Adrian C. Deneys, Praxair, Inc., PMT R&D, 777 Old Saw Mill River Rd., Tarrytown, NY 10591 USA; Thomas P. Battle, Dupont, White Pigments & Minl. Prod., 104 Hay Rd., Edge Moor, DE 19809 USA

8:30 AM
Mechanism and Technology of Segregation Roasting of Oxide Nickel Ores with Subsequent Calcine Flotation and Concentrate Leaching: *I. D. Reznik*[1]; A. V. Tarasov[1]; T. A. Kharlakova[1]; T. A. Mayorov[1]; V. I. Goryachkin[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

1. Oxide nickel ores occur in Russia in the Urals Mountains. As to their composition they are divided into iron-bearing and magnesium-bearing ores. Both types of ore contain about 1% Ni and 0.04-0.09% Co. 2. Three nickel smelters were constructed in the Urals region in the 1930s and they have been in operation until now using the matte smelting technology. Matte is processed to produce converter matte and fire refined nickel, as well as cobalt and nickel salts as by-products. Due to the changes in the prices these smelters are currently operated with low profitability. 3. The authors believe that the most efficient way to improve the economic performance of the smelters is to apply segregation roasting of ores, flotation or magnetic separation of calcine and sulfuric acid pressure leaching of concentrate. As compared with the shaft-furnace smelting, the proposed flow-sheet will reduce the production cost by 22% and even with the lowest nickel price recorded during the recent years, i.e., US $4,180 per 1 tonne, the operation of the smelters will be highly profitable and the payback period for the construction of a new shop will be 2.3 years. 4. Thermodynamic computations and the experimental data indicate that there is no need for moisture in the chain of reactions of calcium chloride decomposition and chlorination of nickel oxide, while the reduction of nickel chlorides requires the presence of hydrogen, which can be produced by hydrolysis of water on solid carbon particles or primary metal grains. 5. The reaction mechanism has been investigated providing additional substantiation for selection of a double-stage segregation roasting flow-sheet: removal of moisture and heating of ore to 950-1000°C during the first stage and chlorination and chloride reduction in the presence of small amounts of water at 800-950°C during the second stage performed in another unit. 6. The table below presents the results of laboratory tests carried out in an intermittent mode with close-circuit flotation and magnetic separation steps. The data presented confirms the high performance values of the process. 7. In the process of oxidizing pressure leaching of concentrates with sulfuric acid with an acid requirement of 55%, at 150°C and an oxygen pressure of 4 atm, about 96-98% Ni and 88-95% Co reported to the solution virtually with complete absence of iron. 8. The overall world resources of nickel in deposits of ores with less than 1% Ni are estimated at 80 million tonnes. For this reason upgrading of low-grade nickel ores using the segregation roasting technology may be of interest for a number of countries. Comparative performance values of flotation

and magnetic separation of calcine obtained from iron- and magnesium-bearing ores (close circuits).

8:55 AM

Optimisation of Pellet Reduction in a Phosphorus Furnace: C. Dresen[1]; *J. H.L. Voncken*[1]; W. Schipper[2]; R. de Ruiter[2]; M. A. Reuter[1]; [1]Delft University of Technology, Appl. Earth Scis., Raw Matls. Tech., Mijnbouwstraat 120, Delft 2628 RX The Netherlands; [2]Thermphos International BV, PO Box 406, Vlissingen 4380 AK The Netherlands

Research is presented with respect to investigations on the reduction process of phosphate pellets. In the reduction process of phosphate ores, the following parameters were investigated: 1. Diffusion ability of the phosphorus oxide and gaseous products from the carbon. 2. Influence of reaction flux on reaction kinetics. 3. Temperature influence. 4. Influence of mineralogy and morphology (different ores). 5. Grain size influence of silica and coke. Experiments were carried out in a vertical tube furnace with 2 ores, 2 types of silica and one type of coke. Experimental conditions simulated furnace conditions. The following conclusions could be made: Reaction rate increases with the addition of SiO_2 to the pellet; Reaction rate increases above 1350°C; Mixing different ore types in the pellets leads to differences in reduction kinetics; Grain sizes of the coke particles influence reduction kinetics. The amount of slag formation is influenced by silica addition, coke sizes, temperature.

9:20 AM

Submerged Lance Injection Dynamics: *Sacha Neven*[1]; Maurits Van Camp[2]; Bart Blanpain[1]; Patrick Wollants[1]; [1]Katholieke Universiteit Leuven, Metaalkunde en Toegepaste Materiaalkunde (MTM), Kasteelpark 44, Heverlee, Brabant 3001 Belgium; [2]Union Minière Research, Kasteelstraat 7, Olen, Antwerpen 2250 Belgium

Submerged lance technologies combine high intensity processing characterized by rapid mixing and reaction rates using a simple reactor design. The Isasmelt technology uses a central submerged lance to inject oxygen-enriched air and fuel into a bath thus creating large gas bubbles and intensive turbulence and splash. The lance is equipped with swirlers to increase gas velocity and heat transfer. This enables the formation of a frozen slag layer on the lance which protects it from the aggressive environment. Water models as well as high temperature reactors were used to validate the Davidson-Schüler model on bubbling frequencies [1]. Resonance effects related to the bubbling frequency have proven to be very relevant to reactor construction and operation. Frequencies and pressure amplitudes were measured in a wide range of gas flow rates: from 10-4 m^3/sec using a water model to more than 30 m^3/sec in an industrial process. Agreement with the model was satisfactory. The experiments however also clearly showed that crossflow and bubble wake induce slightly higher frequencies. No influence of lance depth and use of swirlers on the bubbling frequency was observed in our experiments with water models. [1] J. F. Davidson and B. O.G. Schüler, "Bubble Formation at an Orifice in an Inviscid Liquid", TRANS. INSTN. CHEM. ENGRS., vol. 38, 1960, pp. 335-342.

9:45 AM

Electric Arc Furnace Dusts Recycling Process in a Cupola to Produce Commercial Zinc Dusts and High Quality Cast Iron: *Grégory Miette*[1]; Xavier Mévisse[1]; André Van Lierde[1]; Herman Dehon[2]; [1]Université Catholique de Louvain, Unité PCIM, Place Sainte-Barbe 2, Louvain-la-Neuve 1348 Belgium; [2]Fonderie Piret S.A., Chaussée de Châtelet 273, Gilly 6060 Belgium

The paper deals with a process that allows the recycling in a cupola of EAF dusts with 18% Zn. It produces dusts with zinc grade higher than 50-55% and commercial cast iron with less than 200 ppm Zn, 50 ppm Pb, 0.15% S. The paper focuses mainly on lab tests made with the aim to develop an effective formulation of self reducing-melting pellets. With such a formulation, agglomerates develop a breaking load higher than 125-150 kg without any thermal treatment. Zinc and lead are volatilized in the cupola with yields higher than 95% at 1200°C, before any melting. Sulphur is transferred into gas and slag. All these conditions allow to lower zinc, lead and sulphur grades below the cast iron specifications. Results of first bench scale tests performed at Fonderie Piret to validate the process will be presented.

10:10 AM Break

10:20 AM

Measuring Total Gas-Liquid Interfacial Area from Submerged Gas Injection into Pyrometallurgical Reactors: *Sarah Jane Buckler*[1]; David E. Langberg[1]; Douglas R. Swinbourne[2]; [1]CSIRO Minerals, Box 312, Clayton, Victoria 3169 Australia; [2]RMIT, Cheml. & Metlgcl. Eng., PO Box 2476V, Melbourne, Victoria 3001 Australia

Many chemical and metallurgical processes use submerged gas injection to increase contact area between gas and liquid phases in order to promote faster overall reaction or refining rates. Estimating total interfacial area from submerged injection into industrial pyrometallurgical vessels from existing knowledge is a problem with two aspects, laboratory measurement and scale-up. The work presented in this paper is part of an ongoing experimental investigation, using chemical measurement methods, to quantify the total gas-liquid interfacial area available in pyrometallurgical vessels. Experiments were performed for different operating conditions in a medium-scale, high temperature (1300-1400°C) metal system, and at both medium and large-scale (1/3-scale industrial vessel) in an aqueous system. The effects of the three operating variables, injected gas flow rate, nozzle diameter and nozzle submergence, are quantified in both systems and implications for scale-up of the high temperature results are discussed in light of the scale-up characteristics in the aqueous system.

10:45 AM

Aluminum Deoxidation Equilibrium in Liquid Fe-36%Ni Alloy at 1773K: *Sang-Beom Lee*[1]; Hae-Geon Lee[1]; Chang-Hee Rhee[1]; Dong-Sik Kim[2]; [1]POSTECH, MSE, San31, Hyoja-Dong, Namgu, Pohang, Kyungbuk 790-784 S. Korea; [2]POSCO, #5 Dongchon-Dong, Nam-Gu, Pohang, Kyungbuk 790-784 S. Korea

The deoxidation equilibrium for aluminum in liquid Fe-36%Ni alloy was carried out by using Cold Crucible under Ar gas atmosphere at 1773K. The interaction parameter between aluminum and oxygen in liquid Fe-36%Ni alloy was determined as -1.42 within the composition range of [%O] < 0.015 and [%Al] < 1. The equilibrium constant for the deoxidation reaction: Al2O3(s) = 2Al + 3O for Fe-36%Ni alloy was obtained to be -13.01 in logarithmic scale.

11:10 AM

Pyrometallurgical Processing of Low-Grade Lead-Zinc-Copper Raw Materials as a Source for an Increase in Production of Non-Ferrous and Precious Metals: *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 significant part of natural resources of heavy non-ferrous metals is represented by refractory polymetallic ores containing precious metals. Production of standard-grade monometallic concentrates from such ores results in substantial losses of valuable constituents, especially precious metals, which report to intermediate products, when efforts are taken to improve the grade of main concentrates. A number of concentrators deliberately produce low-grade polymetallic concentrates in order to maintain an adequate recovery of precious metals from ores. The lack of suitable technology for treating sub-grade concentrates and middlings with satisfactory recoveries of zinc, lead, copper and precious metals during the metallurgical stage of processing poses a limitation to the production of these metals despite a significant shortage of supply. The technology concept proposed by the Gintsvetmet Institute is based on an improved process for electrothermal smelting of calcined charge substantially reducing thereby the total expenses (and first of all energy cost) and creates favorable conditions for solution of environmental problems associated with treatment of small volumes of metallurgical off-gas. Elimination of the need for soda in the smelting process permits production of lead bullion and good segregation of matte and slag into which valuable constituents are concentrated (copper in matte and zinc in slag). Since crude lead is the best collector for precious metals, the latter are virtually completely recovered into lead in the process of smelting, and partially into the matte, and further transferred into commercial-grade products. Lower losses of precious metals in slag in case of the new technology are attributed to the fact that slag contacts with matte with low contents of gold, silver and lead, rather than with lead bullion. The new electrothermal technology produces only small amounts of off-gas resulting in a more cost-effective operation of emission control equipment and lower emissions of deleterious pollutants into the environment.

11:35 AM

Dezincing of Iron-Containing Secondary Raw Materials in Frothed Slag Layer in an Electric DC Arc Furnace: *A. V. Tarasov*[1]; *V. A. Bryukvin*[1]; V. M. Paretsky[1]; V. G. Leontyev[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

The proposed process is based on creating a controlled frothed layer of molten slag, in which fine particles of carbonaceous reductant and raw material to be processed are mixed with emulsified iron. Frothing of the slag layer without external blowing occurs as a result of intensive evolution of gaseous reduction products. The froth reaction process is maintained due to the heat of an electric arc in the slag layer. When the metal bath is heated by the arc to 1400-1500°C, the low temperature of the slag surface of about 1200-1300°C is maintained due to low thermal conductivity of slag froth preventing heat loss. In order to maintain the required slag froth level and control the process temperature, air tuyeres are installed above the slag bath; blowing air serves also for oxidation of zinc in fumes and for its transportation. High adsorption and thermal insulating properties of slag froth ensure virtually complete elimination of dust and mist entrainment along with low heat losses. The main macrokinetic regularities of the carbon thermal reduction of zinc-containing slag melts have been studied under frothing conditions. The mechanism of formation of gas-slag emulsion has been identified under the conditions of reaction [C]+[O]=CO occurring locally at the slag-metal interface. It has been demonstrated that under the froth reduction conditions the zinc removal rate from the melt increases on the average by 3 to 5 times. Based on the physical and chemical data obtained, basic principles of the dezincing technology have been developed for different intermediate oxide iron-containing products produced in the process of ferrous and non-ferrous metals production. Separation of zinc and iron under reducing conditions is achieved by transfer of reduced zinc into fumes and transfer of reduced iron into metal melt. Zinc fumes are carried over, without external blowing, with reduction products and undergo subsequent oxidation above the slag by air blowing. The residual zinc and iron contents of slag are 0.01-0.05% and up to 3%, respectively. The high intensity of carbothermal reduction in slag froth with good shielding with electric arc makes it possible to ensure a productivity higher by 3 to 5 times than in case of conventional electric-arc smelting with power requirement by 1.5 to 1.8 times lower. The main process parameters: zinc recovery with its initial content of over 1% is 95% with residual zinc concentration in slag of 0.01-0.05%; the degree of iron reduction depending on the technological and economic reasonability as high as 90% or higher with its residual content in slag of up to 3%; the temperature of molten metal is 1300-1650°C depending on the required level of saturation of iron with carbon within a range of 0.05 to 2.5%; the slag layer temperature of 1200-1350°C depending on viscosity for a particular slag composition; the molten slag composition varies depending on the purpose of refining and alloying of molten metal; the requirement of pulverized carbonaceous reductant (coke, coal, etc.) is about 12% of the feed material depending on the zinc content, required degree of reduction and carbon saturation of iron, carbon content of the feed material; it is close to the theoretical reaction requirement for direct reduction with formation of CO and for iron reduction up to 50%; the unit productivity of the process is within 100 to 300 kg/m2 hour; the estimated electric power requirement is 300-400 kWh per 1 t of feed material, which is lower than the common level due to lower heat losses under the slag foam and post-combustion of carbon monoxide above the slag by air blowing.

High Performance Metallic Materials for Cost Sensitive Applications: Overview and Titanium Alloys I

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

Monday AM — Room: 213
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Edward Y. Chen, TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91769 USA; F. H. (Sam) Froes, University of Idaho, Inst. of Matls. & Adv. Proc., Mines Bldg., Rm. 321, Moscow, ID 83844 USA

8:30 AM Invited
Metals for Cost-Sensitive Applications–An Overview: *E. Y. Chen*[1]; E. Taleff[2]; R. Boyer[3]; D. Zhang[4]; Lu Li[5]; M. Ward-Close[6]; D. Eliezer[7]; F. H. (Sam) Froes[8]; [1]TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91768 USA; [2]University of Texas, TX Matl. Inst., Col. of Eng., Austin, TX 78712 USA; [3]The Boeing Company, PO Box 3707, MS 6H CJ, Seattle, WA 98124 USA; [4]The University of Waikato, Dept. of Matls. & Proc. Eng., PB 3105, Hamilton New Zealand; [5]National University of Singapore, Dept. of Mechl. Eng., 10 Kent Ridge Crescent, Singapore 119260; [6]DERA, Titanium Alloys Metallics Div., Griffith Bldg. (A7), Rm. 2008, Hampshire GU14 OLX, Farnborough UK; [7]Ben-Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel; [8]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

This paper will present an overview of the various factors which contribute to the cost of metals in final assembled configurations. This will include costs of extraction, melting and fabrication to mill products, machining, assembly and quality assurance. Examples will be given for various metals and for use in differing industries such as automobiles and aerospace.

9:10 AM Invited
Prospects for Cost Reduction of Titanium via Electrolysis: *Harry Rosenberg*[1]; [1]Amargosa Group, 9371 Almar Pl., Pittsburgh, PA 15237 USA

Fused salt electrolysis is one of the more versatile techniques available for producing titanium. It is quite versatile, not only in control of metal purity, particle size, and distribution, but also in cell configuration and operating mode. Three basic operating techniques are available: electrowinning, electrorefining, and electron mediated reaction. At present, only electrorefining is commercial; although electrowinning titanium was demonstrated on the industrial scale for a time by TIMET some 40 years ago. Current technologies and prospects for further progress are reviewed.

9:40 AM Invited
Titanium: Why is it Expensive and What Can be Done?: *Stephen J. Gerdemann*[1]; [1]Albany, Thermal Treatment Technologies, 1450 Queen Ave. S.W., Albany, OR 97321 USA

Titanium is considerably more expensive than the light metals aluminum and magnesium. This high cost has limited the use of titanium to specialty markets. This paper will examine the history of commercial titanium production and how that history continues to affect the cost today. A brief description modern titanium extractive metallurgy will be given. The costs associated with each step starting with the ore and ending with a titanium part will be identified. The most promising of the proposed new processes will be discussed as well as their chance of success and potential for cost savings. Finally, an estimate for the cost for titanium made by the "perfect process" will be made.

10:10 AM Break

10:30 AM Invited
Implementation of Advanced Metal Technologies to Reduce the Cost of Aerospace Systems: *Ricky L. Martin*[1]; [1]Boeing, Phantom Works, PO Box 516, MC S276-1240, St. Louis, MO 63166-0516 USA

Leveraging the inherent affordability of metallic materials and processes provides excellent opportunities for researchers to minimize costs in existing and advanced aerospace systems. Boeing is participating in a wide range of cost reduction initiatives that are demonstrating and implementing advanced metal product technologies that provide documented affordability benefits over traditional approaches to manufacturing aerospace structures. Although aerospace industry investment in structural metals research and development has been declining recently, participation in company partnerships, industry consortiums, and government-sponsored development initiatives have allowed accelerated transition of metal product technologies into production. Application of new technology to improve existing manufacturing methods, and the introduction of new revolutionary metal processes are both highlighted in this overview.

11:00 AM
Recent Developments in the Manufacturing of Low Cost Titanium Alloys: *Yoji Kosaka*[1]; Stephen P. Fox[1]; [1]TIMET, Henderson Techl. Lab., PO Box 2128, Henderson, NV 89009 USA

About a half dozen of so-called "Low Cost Titanium Alloys" were developed almost a decade ago being targeted to emerging markets such as automotive and armor applications. Initial efforts were focused on the formulation cost of alloys by increasing revert content or designing new alloys that require less expensive raw materials. None of these alloys could achieve a solid presence in these markets until very recently. The environment for low cost titanium has been changing favorably to low cost titanium alloys. EB cold hearth melting is recognized as a versatile tool to provide low cost alloys as well as premium grade titanium alloys. The reduction of conversion cost is critical to an overall cost reduction. A combined effort to introduce new titanium alloys by vertical integrated teams with users and suppliers has started to open up the opportunity for low cost titanium alloys in cost sensitive applications. This paper will review and discuss the recent developments in the manufacturing of low cost titanium alloys.

11:20 AM
Direct Production of Ti-6-4 Alloy Plate from Single Melt, Electron Beam Cold Hearth Melted Slab Ingot: *J. R. Wood*[1]; Steven H. Reichman[2]; [1]Allegheny Technologies, Inc., Allvac, 2020 Ashcraft Ave., Monroe, NC 28111-5030 USA; [2]Allegheny Technologies, Inc., Allegheny Ludlum, 500 Green St., Washington, PA 15301 USA

As part of the Metals Affordability Initiative Consortium Program, this study was initiated to produce aerospace quality, standard grade Ti-6Al-4V alloy plate directly from single melt, electron beam cold hearth melted (EBCHM) rectangular slab ingots. It is to be demonstrated that the EBCHM process is capable of producing chemical homogeneity, macro and microstructure and mechanical properties comparable to plate made from conventional double melt vacuum arc refined (VAR) round ingots. It is also to be demonstrated that large EBCHM slab ingots can be rolled directly to plate on conventional equipment with significant cost savings compared to the processing route for conventional round VAR ingots. The lower cost is derived from the use of less costly raw materials, lower melt costs, streamlined processing and improved yields. The progress to date in this multi-year program is reported and discussed.

11:40 AM
Electron-Beam Cold-Hearth Single-Melt Ti-6Al-4V Plate for Armor Applications: *John C. Fanning*[1]; [1]TIMET, Techl. Lab., PO Box 2128, Henderson, NV 89015 USA

Although titanium provides high ballistic mass efficiency and other advantages, widespread use of titanium alloys in ground combat systems has been limited by the relatively high raw material cost. Ti-6Al-4V (and variations thereof) is the preferred alloy for both structural and applique armor applications. Up to the present virtually all of the production of this product has been per aerospace specifications, which typically require multiple vacuum arc re-melting (VAR). During the past several years cold hearth melting has been used in place of one of the vacuum arc melts because of the ability of the process to remove inclusions. Significant cost savings may be achieved by the use of a single melt process. Although single melt heats of commercially pure titanium are now being routinely produced, there is limited production experience for single melt of alloy grades such as Ti-6Al-4V. A recent study by the US Army provided an initial assessment of the product of an electron beam, cold hearth, single melt of Ti-6Al-4V plate for application to Army ground vehicles. This paper provides the results of ballistic and mechanical property testing of additional samples of this type of product.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Phase Equilibrium Modeling

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

Monday AM	Room: 204
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, C.&M.S. Direct. (L-353), PO Box 808, Livermore, CA 94551 USA; Suzana Gomez Fries, ACCESS e.V., RWTH-Aachen, Intzestrasse 5, Aachen 52072 Germany

8:30 AM Introductory Remarks

8:35 AM Keynote
Hume-Rothery and CALPHAD Thermodynamics: *Larry Kaufman*[1]; [1]140 Clark Rd., Brookline, MA 02445-5848 USA

H-R's "Atomic Theory for Students of Metallurgy" was a text for undergrads in the '50s. In '66 I was in Geneva for the Battelle's Conference on "Phase Stability in Metals and Alloys" with H-R, Brewer and Kubachewski. H-R invited me to review the subject for vol.14 (1969) of PMS that he edited. We corresponded 30 months till his death in '68. Kuba's Brunel and Sheffield conferences in '71 and Munster '72 spawned the CALPHAD method, Journal and conferences which have grown till today! The Symposium on Computational thermodynamics and Materials Design at the 2001 TMS Annual Meeting illustrated many cases where CALPHAD Thermdynamics affording the broadest description of stable, unstable and metastable phases over wide ranges of composition, pressure and temperature were used to predict useful materials and processes. In this presentation the connection will be made between H-R's work, the CALPHAD method and "Alloy Thermodynamics".

9:30 AM Invited
Phase Diagram Calculations: Contributions of Ab Initio and Cluster Variation Methods: *Catherine Colinet*[1]; [1]LTPCM, ENSEEG, BP 75, Saint Martin d'Heres, Cedex F-38402 France

The treatment of the short-range order and long-range order in solid solutions, and of order-disorder transformations need the use of the cluster variation method (CVM) or of Monte Carlo simulations. These methods need as input the interaction parameters between the atoms, called the cluster interactions. These parameters may be fitted using thermodynamic and phase diagram data, such as enthalpies of formation or order-disorder temperatures. However the procedure is ambiguous since the nature of the most important cluster interactions (pairs, triplets, quadruplets, etc), their range, and their composition dependence are a prior unknown. Several attempts have been made to deduce the cluster interactions from first principle calculations. Two approaches are available for such calculations. In the first one, the energy of the completely disordered solid solution is calculated by the coherent-potential approximation, the effective cluster interactions are calculated by the embedded-cluster method or by the generalized perturbation method. The second approach that is now extensively used consists in performing ab initio total energy calculations of perfectly ordered compounds in order to estimate the values of the cluster interactions. A review of the phase diagram calculations which have been performed using the methods described above will be presented.

10:00 AM Invited
Effect of the Interaction between the Chemical and the Magnetic Ordering on the Phase Equilibria of Iron Base Alloys: *Ikuo Ohnuma*[1]; Ryosuke Kainuma[1]; Kiyohito Ishida[1]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Senday 980-8579 Japan

It is well known that the magnetic properties such as the Curie temperature T_c and the mean magnetic moment β of ordered compounds have different values from those of the disordered solutions. For instance, both T_c and β of the Ni_3Pt ($L1_2$) and NiPt ($L1_0$) and T_c of the CoPt ($L1_0$) and $CoPt_3$ ($L1_2$) ordered compounds are strongly depressed due to the ordering compared with those of the metastable disordered Ni-Pt and Co-Pt alloys. On the other hand, the γ'-$FeNi_3$ ($L1_2$) and the α'-FeCo (B2) ordered compounds have higher T_c and β values comparing with the disordered solution phases, γ (A1) and α (A2), respectively. In consequence, the stability of the ordered phase is depressed or enhanced due to the interaction between the chemical and magnetic ordering caused by the decrease or increase of T_c and β values. The purpose of this study is to investigate the effect of the interaction between the chemical and the magnetic ordering on the phase equilibria in the Fe-X (X=Al, Co, Ni, Rh, Si) binary systems.

10:30 AM Break

10:45 AM Invited
Phase Diagram Calculation: Present State and Applications: *Y. Austin Chang*[1]; W. A. Oates[2]; Shuanglin Chen[3]; F. Zhang[3]; S. Daniel[3]; Xinyan Yan[3]; F.-Y. Xie[1]; R. Schmid-Fetzer[4]; [1]University of Wisconsin, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA; [2]University of Salford, Sci. Rsrch. Inst. M5 4WT UK; [3]CompuTherm, LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [4]University of Clausthal, Inst. of Metall., D-38678, Clausthal-Zellerfeld Germany

In this presentation, we will (1) assess the current state of the phenomenological-model approach in calculating phase diagrams stressing the great stride made in recent decades and the need for continuing improvement in the future and (2) present experimental and computational results obtained on the paths of solidification and microsegregation in multicomponent commercial aluminum alloys, i.e. more than 10 components. The calculated results were obtained by integrating phase diagram calculation (PanEngine) with a modified Scheil model incorporating solid state diffusion, dendritic coarsening and undercooling effects. The experimental results were obtained by directional solidification.

11:15 AM Invited
Chemical Criteria in the Assessment of Alloy Constitutional Properties: *Riccardo Ferro*[1]; Gabriele Cacciamani[1]; [1]Universita' di Genova, Dip. di Chimica e Chimica Industriale, via Dodecaneso 31, Genova I-16146 Italy

It is well known that an accurate prediction of phase equilibria in multi-component systems requires a sound thermodynamic characterization of the involved binary and ternary sub-systems. In turn this needs good sets of data precisely determined and critically assessed. Comments and examples about a number of semi-empirical chemical criteria which may be useful in the selection and application of the assessment procedures are reported. Regularities which may be found in considering the general alloying behavior of the metals pertaining to certain groups or blocks of the periodic table (such as, for instance, the alkali metals, the lanthanides, the actinides, etc.) are underlined. The results obtained in the author's laboratory in the study and evaluation of properties of lanthanide alloys (phase equilibria, thermodynamics, crystallochemistry, etc.) are presented and discussed.

11:45 AM Invited

Developments in Thermodynamic and Phase Equilibrium Modelling at the National Physical Laboratory: *John A. Gisby*[1]; Alan Dinsdale[1]; Hugh Davies[1]; Susan Martin[1]; Jim Robinson[1]; [1]National Physical Laboratory, NPL Matls. Ctr., Queens Rd., Teddington, Middlesex TW11 0LW UK

Almost since its foundation in 1900, the National Physical Laboratory in the UK has been involved in the study of the thermodynamic data and phase diagrams. Initially this tended to focus on the accurate measurement of the constitution of binary and ternary alloy systems. From the 1950's onwards due to the pioneering work of Kubaschewski this led to the study of thermodynamic properties through the development and use of key experimental equipment, the critical assessment of thermodynamic properties and their use in the calculation of the change in thermodynamic properties associated with chemical reactions. The natural direction of Kubaschewski's work led to the development at NPL of MTDATA, comprising software and databases utilising the growing availability and power of computers. MTDATA has long been one of the world's foremost thermochemical modelling packages and it has proved itself over many years of use in solving practical problems. It is a powerful and user-friendly software and data package for the prediction of phase equilibria in systems containing large numbers of chemical elements. It has been designed around the high reliability of the NPL Numerical Optimization Software Library. MTDATA is licensed to industrial, government and academic organisations worldwide and training courses are held regularly at NPL and in the US. A range of databases covering a very wide variety of system types is available for use with MTDATA reflecting numerous potential applications in the fields of metallurgy, chemistry, materials science and geochemistry. In this paper NPL's contribution to the critical assessment of thermodynamic data and the calculation of phase equilibria will be reviewed. Particular emphasis will be given to the SGTE database for elements, the development of MTDATA and some of the areas in which MTDATA has been used successfully for the benefit of industry.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Nanostructures and Materials

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

Monday AM	Room: 617
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Hubert I. Aaronson, Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; Chandra Shekhar Pande, Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6325, Washington, DC 20375 USA

8:30 AM Opening Remarks

8:40 AM Invited

Grain Rotation and Nanostructure Evolution: New Insights from Atomistic Simulations: *S. G. Srinivasan*[1]; J. Cahn[2]; [1]Los Alamos National Laboratory, T-11 Grp., Theoretl. Div., MS B262, Los Alamos, NM 87545 USA; [2]NIST, Matls. Sci. & Eng., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

Bhakta Rath has made seminal experimental contributions that has led to the theoretical understanding of grain boundary (GB) migration [Rath and Hu, Trans. AIME, Vol.245, pp.1577 (1968)]. We revisit this fundamental problem of GB motion and the concomitant grain growth (GG) on the nanometer scale using molecular dynamics simulations in 3-dimensions. We found frequent grain rotation with some surprising and counterintuitive results. For example, bicrystal systems of a circular cylindrical grain, embedded in another with a variety of initial misorientations, can rotate continuously from small angles to large angles, even where their GB specific surface energy increases, as they shrink. Suppression of the grain rotation by using rounded lath shaped cylindrical grains, with straight faces and curved ends, enable the grains to shrink considerably faster than the embedded circular-cylinders with the same initial misorientation. When such grains rotate, the velocity of their grain boundaries is not proportional to curvature. In an array of congruent hexagonal columnar grains, which should have been stable, grain rotation triggers unexpected GG. For small misorientations, many of these surprises can be understood in terms of motion of GB dislocations, but the behavior continues smoothly to higher angles. The implication of these findings for nanostructure stability and GG will be discussed.

9:05 AM Invited

Interfaces and Surfaces in Nanostructured Materials: *Miguel Jose Yacaman*[1]; [1]University of Texas-Austin, Dept. of Cheml. Eng., Austin, TX 78712-1062 USA

The study of metal nanoparticles and their assembling is becoming very important in many fields such as: nanoelectronics, photonics, sensing devices, drug delivery and so on. In these systems the surface atoms are a large proportion of the total number of atoms that produce unique properties. In the present work we discussed the structural changes on nanoparticles as a function of the size, two main cases are discussed: bare particles and particles capped with an organic molecule. It is shown that significant changes on the structure are observed. The coalescence behavior and their self-assembly is also discussed.

9:30 AM Invited

Solid State Alloying of Nanostructured Fe-Zn Binary System: Fei Zhou[1]; *Ye Tsang Chou*[1]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Chem. & Biochem. Eng. & Matl. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA

Iron-Zinc binary alloys in solid solution were processed by mechanical alloying (MA) of high-purity metal powders. The MA-induced microstructural evolution was characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC). The nanostructured alloys are maintained in single-phase, body-centered cubic lattice with the zinc content up to 65 at.%. Further addition of zinc, e.g. at 95 at.%, would shift the cubic lattice to hexagonal close packing. An important feature of this binary system is that the solid solutions formed by the MA obey the Vegard's law over the entire range of mixing. The effect of the addition of zinc on ferromagnetic properties was examined and compared with the data obtained from the same alloy system prepared by vapor quenching. Mechanisms underlining the solid state alloying in the Fe-Zn system are also discussed.

9:55 AM Invited

Chemical Reactions at the Gas-Solid Interfaces: Effects on the Electrical Conductivity of Semiconducting Nanomaterials: *Marie-Isabelle Baraton*[1]; Lhadi Merhari[2]; [1]University Limoges, SPCTS UMR 6638 CNRS, 123 Ave. Albert Thomas, Limoges 87060 France; [2]Ceramec, 64 Ave. de la Libération, Limoges France

Improvement of nanosized powder-based sensors requires a careful study of the surface reactions at the origin of the gas detection mechanism. This is a necessary step toward a rigorous industrial fabrication protocol. In this work, chemical reactions occurring at the interface between gases and semiconducting nanopowders are investigated by Fourier transform infrared (FTIR) spectrometry. These surface reactions are compared for several semiconducting materials (tin, indium and tungsten oxides) at different temperatures. Simultaneously, the effects of these surface reactions on the electrical conductivity of the metal oxides are analyzed and discussed in relation with their sensing properties toward oxygen and carbon monoxide. This work is funded by the European Commission under the "Information Society Technologies" programme (contract number IST-12615).

10:20 AM Invited
Interfaces in Zr Based Nanocrystals and Combustion Synthesized Ti Aluminide: *Gautam Kumar Dey*[1]; [1]Bhabha Atomic Research Center, Matls. Sci. Div., Trombay, Mumbai, Maharashtra 400 085 India

The nature of the interfaces associated with intermetallic phases produced in a very fine morphology by crystallization of Zr based bulk metallic glasses and combustion synthesis of TiAl has been examined by high resolution electron microscopy. The nanocrystals produced by the crystallization of $Zr_{52}Ti_6Al_{10}Cu_{18}Ni_{14}$ bulk glass have been found to lie in the size range of 15 to 50 nm and comprised phases isostructural with Zr_2Ni and Zr_2Cu. Various types of interfaces in these phases such as twin boundaries, stacking faults and antiphase domain boundaries have been studied. It has been possible to generate a very fine lamellar microstructure by combustion synthesis of TiAl with widths of the α_2 and γ lamellae in the range of 5 to 20 nm. The interface between the α_2 and γ phases has been investigated in detail in this microstructure. The interfaces between contiguous lamellae of the γ phase have also been examined.

10:45 AM Invited
Effects of Interfaces on the Properties of Nanostructured Magnetic and Optical Films: *Gan-Moog Chow*[1]; [1]National University of Singapore, Dept. of Matls. Sci., Kent Ridge 119260 Singapore

Nanostructured polycrystalline films with grain size less than 100 nm have a significant amount of grain boundaries and interfaces. Control of the grain size,chemistry and structure of the interfaces is essential to control material properties. We have fabricated nanostructured films (NixCo100-x, CoCrPt/Ti and Au/BaTiO3) for magnetic and optical applications. These films were studied using x-ray scattering, anomalous x-ray scattering, extended x-ray absorption fine structure, scanning electron microscopy, transmission electron microscopy, atomic force microscopy, x-ray photoelectron spectroscopy, vibrating sample magnetometry, alternating gradient magnetometry and UV-VIS-IR spectroscopy. In this talk, examples will be given to demonstrate the effects of interphase interface on the structural, magnetic and optical properties of these films. The experimental results of the long-range order and local atomic environment (in plane and out-of plane) and the interphase interfaces of these films will be discussed and correlated with that of thermodynamics modeling.

Magnesium Technology 2002: Magnesium Primary Production

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

Monday AM — Room: 606
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Howard I. Kaplan, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA; Neale Neelameggham, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA

8:30 AM
History Primary Magnesium since WWII: *Byron B. Clow*[1]; [1]International Magnesium Association, 6731 Whittier Ave., McLean, VA 22101 USA

Primary magnesium production just prior to WWII was around 32,000 tonnes with most of it coming from Germany and England with smaller amounts from France and the USA. Peak production during the war was 232,000 tonnes with most of it coming from the 15 plants that had been built in the United States. By 1946 worldwide demand had fallen back to pre-war levels and did not return to war levels until 1980. The paper will identify the various plants built around the world since WWII and the impact of exports from Russia and China beginning in the early 90's.

9:00 AM
Magnesium Market Model Simulation: The Impact of Increased Automotive Interest in Magneisum: *Randall Joseph Urbance*[1]; Richard Roth[1]; Joel Clark[1]; [1]Massachusetts Institute of Technology, Matl. Sys. Lab., 238 Main St., Ste. 319, Cambridge, MA 02142 USA

Increasing energy and environmental concerns are forcing automakers to consider light-weight alternatives to traditional component designs. Magnesium, the lightest engineering metal, has been cited as a potential weight-saving material for automobiles, but has been slowly adopted due to high prices and limited engineering experience. Despite the technical and financial challenges of magnesium design, recent trends in the auto industry show renewed interest in the material. The small size of the magnesium supply base could limit the use of magnesium in the automotive arena if increasing demand leads to material shortages and price volatility. A system dynamics model of the magnesium market was created to investigate the potential problems caused by a small, rapidly expanding supply base coupled with increasing, but very price-sensitive material demand. The model, which simulates supply, demand and price interactions, was used to investigate market stability strategies that will benefit all players.

9:25 AM
An Improved Process for Production of Magnesium: Hans R. Eklund[2]; Per B. Engseth[3]; *Thorvald Mellerud*[1]; Birger Langseth[4]; Oddmund Wallevik[4]; [1]Hydro Magnesium, Ave. Marcel Thiry 83, B-1200 Brussels Belgium; [2]Hydro Magnesium, N-0246 Oslo Norway; [3]Hydro Magnesium, N-3901 Porsgrunn Norway; [4]Norsk Hydro ASA, Rsrch. Ctr., N-3901 Porsgrunn Norway

Norsk Hydro started production of primary Magnesium in Porsgrunn, Norway 1951,using the IG Farben chlorination process for production of anhydrous MgCl2 as feedstock to the electrolysis. Norsk Hydro has continued to develop the IG Farben chlorination process, and an improved version is still in operation at the Porsgrunn plant. In the late 1960's Norsk Hydro started to develop a process for production of anhydrous MgCl2 based on dehydration of MgCl2 brines. The first generation of the process was started in Porsgrunn 1978, when a 15 kt Mg/yr demonstration unit was put in operation. A modified and developed version of the technology was installed in the Norsk Hydro green-field facility at Becancour (Quebec) Canada. The plant produce MgCl2 brine by dissolving of magnesite in hydrochloric acid and started operation in 1989. The new dehydration technology first of all offers an environmentally sound solution with minimal emissions of chlorine and chlorinated hydrocarbons, improved productivity and improved quality of the anhydrous MgCl2 feed, giving significantly improved performance of the electrolysis. The development of the Norsk Hydro dehydration process has continued, and today an improved second generation process is ready for implementation in brown-field expansions or green-field projects. Several elements of the improved process are already installed in the Becancour plant. Compared to previous generation, the improved dehydration process has further improved productivity and reduced energy consumption. A presentation is made of the Norsk Hydro improved dehydration process. Its basic features and performance are described, supporting that large scale operation based on this technology is very competitive compared to other existing and recently presented front end technologies.

9:50 AM
Process Optimization of Magnesium Thermal Reduction: *Henry Hu*[1]; Alfred Yu[1]; *Naiyi Li*[2]; [1]University of Windsor, Mech., Auto. & Matls. Eng., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys. Dept., Scientific Rsrch. Lab., Rm. 3011, MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA

A numerical model of heat and mass transfer in the vertical retort has been developed to simulate and optimize the process of magnesium thermal reduction. The simulations were run to determine the effect of varying parameters, such as the diameter of retort, thickness of the compound, and slot angle, on the magnesium reduction time. The model predicted the temperature distributions, the heating curves, the recovery ratio of magnesium, and the total process time. The predictions were used to optimize the magnesium reduction process, the dimensions of retort, the shapes of materials, and reaction cycle. The result shows that the optimizations will significantly improve the Pidgeon magnesium reduction process in vertical retort, increase production capacity and recovery, decrease reduction period, and save energy consumption.

10:15 AM Break

10:35 AM
Solid-Oxide-Oxygen-Ion-Conducting Membrane (SOM) Technology for Primary Production of Magnesium from its Oxides: *Uday B. Pal*[1]; Timothy Keenan[1]; Ajay Krishnan[1]; Christopher Manning[1]; [1]Boston University, Mfg. Eng., 15 St. Mary's St., Boston, MA 02446 USA

The work is aimed at producing Mg metal from oxide ores with oxygen gas as a by-product of the process. Oxygen-ion-conducting yttria-stabilized-zirconia (YSZ) crucibles are used to hold the melt (flux) containing dissolved magnesium oxide. A suitable anode is placed on the outside of the YSZ tube. An inert metal rod is placed in the melt, contained in the YSZ crucible, to serve as the cathode. An electric potential is applied between the cathode and the anode to dissociate the magnesium oxide and produce Mg metal at the cathode, while removing oxygen ions through the zirconia membrane and oxidizing them at the anode; a reducing gas is passed over the anode to getter the oxygen. To minimize the ohmic resistance across the YSZ membrane and obtain high current density, these experiments are conducted at temperatures between 1300 to 1400C. The results obtained are used to model the process.

11:00 AM
Physical and Chemical Investigations of Salt Melt Compositions of KCl-NaCl-MgCl-NaBr and KCl-NaCl-MgCl-CaCl Systems: *Ivan Andreevich Barannyk*[1]; [1]State Titanium Research and Design Institute, Magnesium Production, 180 Lenin Av., Zaporizhzhya 69035 Ukraine

In magnesium production by electrolysis and in magnesium and Mg alloys refining multicomponent salt melts, consisting of four and more alkaline halogens, alkali earth metals and magnesium are used. Integrated study of physical and chemical properties of complex halogen systems is of great interest for a justified choice of optimum salt melt compositions for magnesium production. The most important salt melt physical and chemical properties are as follows: fusibility, density in operating temperature limits(650-750°C), viscosity and conductivity. The results of density and fusibility measurements of some salt compositions of KCl-NaCl-MgCl2-NaBr and KCl-NaCl-MgCl2CaCl2 systems are given in the report. Influence of NaBr, CaCl2 and their mixture compositions on multicomponent halogen systems of salts fusibility and density was determined by experimental data processing. Investigated melts density dependence on the temperature in the limits of (700-800°C) was fixed. Using another physical and chemical salt properties data, taken from the literature, an attempt to substantiate the choice of optimum salt mixtures composition for Mg and Mg alloys melting and refining was made.

11:25 AM
Investigation of Granulation of Potassium Clorides and Cent-Electrolyte: *Andrey Vladimirovich Tarasov*[1]; [1]Gintsvetmet Institute, Metall., 13, Ac. Koroleva str., Moscow 129515 Russia

Potassium chloride is one of the most common mineral fertilizers transported to all continents. Its unstable crystals are attrited to dust and cake together during transportation. Granules of potassium chloride must have a diameter of at least 2mm and withstand forces of up to 2kg. Such properties make it possible to spray them rather uniformly within a band up to 10-12m wide. The research conducted with respect to granulation of fused potassium chloride has indicated that conventional methods of granulation of molten potassium chloride are ineffective and do not provide the desired results, primarily due to the physical properties of potassium chloride. Granules produced by filtration of melt through a sieve do not have the required strength and collapse under a force of 0.7kg. The most common method for potassium chloride granulation is pressing in a roll press with subsequent grinding of the pressed layer and strengthening of the granules produced. The yield of final granules is below 30%. Sorting of final granules of carnallite, potassium chloride by flotation permits separation of a fraction of not more than 1.5mm and the pressed granules produced have sharp-angled irregular form. Granulation tests were carried out on a bench-scale laboratory unit using flat water-cooled rolls. One of the rolls had a matrix of granules. Special material was selected for the matrix roll. The granules produced had a regular semispherical form. Rolled granules had a strength of 2.2-2.5kg. At the same time, tests were carried out to study the effect of addition of alkali metals on the strength of granules. Addition of 2-3% of such additives improved the strength of granules up to 4kg. An engineering design has been developed for a semi-commercial plant for potassium chloride granulation in a roll-type unit with a productivity of 2t/hr. Technical parameters of the plant: Fuel requirement, equivalent fuel per 1t of granules 11kg/t; Thermal efficiency of the melting unit 51-54%; Amount of recycled material 5-7%; Dust content of gas at the outlet 10mg/m3. This unit has not been built due to the lack of financing. Granulation of Cent-Electrolyte: Cent-electrolyte formed in the process of electrolytic magnesium production contains 61-63% of potassium chloride and is sent to further processing. Most commonly, cent-electrolyte is cast into blocks of 250-300kg and after cooling during 24 hours it is ground. This method results in formation of large amounts of fines and is associated with substantial emissions of dust, gas and heat into the ambient environment at workplaces. In order to correct those drawbacks, a method of pneumatic granulation of cent-electrolyte was tested and proven on a laboratory-scale unit with a productivity of 20kg of melt per hour. As a result, the appropriate aerodynamic conditions and the composition of spraying gas were determined. An engineering design has been developed for a shop for cent-electrolyte granulation at a plant with a productivity of 25,000 tpy of magnesium. This shop consists of an electrically heated holding furnace, a granulation chamber, a system for metered feeding of molten cent-electrolyte and a dry dust collection system. The main part of the spraying device is a nozzle of a special design. Granules produced are of irregular sharp-angled form, with an average size of 0.9-1.0mm, with a content of about 5% of size fractions of less than 0.9mm and over 1.0mm. Such a shop is currently in operation at the magnesium plant of the Dead Sea Works in Israel.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Structural and Multifunctional Applications - Session I

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

Monday AM	Room: 205
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Amit Ghosh, University of Michigan, Dept. of Matls. Sci. & Eng., 2066 H.H. Dow Bldg., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; William Clyne, Cambridge University, Matls. Sci. Dept., Pembroke St., Cambridge CB2 3QZ UK

8:30 AM Opening Remarks

8:35 AM
Multifunctionality of Closed-Cell Aluminum Foams: *T. Dennis Claar*[1]; Virgil Irick[1]; Jim Adkins[1]; Kenneth Kremer[1]; [1]Fraunhofer USA, Ctr. for Mfg. & Adv. Matls., 501 Wyoming Rd., Newark, DE 19716 USA

Closed-cell aluminum foams are an emerging class of ultra-lightweight materials that can be tailored to exhibit a broad range of properties. The powder metallurgy-based foaming process is capable of fabricating foams with porosity levels of 40 to 90 vol%. Various geometries are possible, including 3-D molded shapes, foam-filled tubes, and aluminum foam sandwich (AFS) panels. A significant characteristic of aluminum foams is their ability to absorb energy during compressive deformation. Energy is absorbed during crushing at a nearly constant "plateau" stress to approximately 60% strain, before the stress increases significantly. Aluminum foam inserts inside metal tubes increased the specific energy absorption by 35-40% compared to the hollow tubes. Foam inserts also increased the bending stiffness of hollow tubes by 40-45%. Foamed aluminum has demonstrated superior fire resistance compared to solid aluminum plates. During exposure to a gas flame at 850-870°C, a 6061 Al plate experienced melt-through, while an Al foam plate was unaffected. Aluminum foams also improve the performance of lightweight armor, providing improved projectile defeat, lower dynamic deflection, and enhanced ballistic shock attenuation. AFS panels have very high specific stiffness, allowing significant weight reduction. Applications that take advantage of these multifunctional features of Al foams will be presented.

9:00 AM
Perspectives on High-Volume Automotive Applications of Lightweight Cellular Metals and Structures: *Ray Jahn*[1]; Andrew M. Sherman[1]; [1]Ford Motor Company, FRL Mfg. Sys., MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA

Laminated and cellular materials have been proposed as a way of achieving higher weight reductions in automotive structures than available with high strength steels, aluminum, magnesium and polymer composites. However, it remains to be seen whether the applications in production vehicles predicted for these materials will develop. One reason is the high cost of making the materials and fabricating them into components. Also, although it is easy to show that such materials offer superior properties, e.g. specific stiffness, it is much more difficult to translate this into lighter weight in fully engineered structures. Among the difficulties are that in applications having complex loading the directional properties of these materials may be a disadvantage, the load carrying characteristics of the available joining methods do not permit full utilization of the material's capabilities, or other aspects of the specific application compromise the materials in some way. In this paper we present a cost/benefit analysis of the use of lightweight materials for automotive weight reduction, which helps establish cost and weight target ranges for the use of cellular and laminated metals.

9:25 AM Moved to Tuesday PM
Tailored Component Fabrication using Stabilized Aluminum Foam: *Greg Mills*

9:25 AM
The Stiffness and Weldability of an Ultra-Light Steel Sandwich Sheet Material with a Fibrous Metal Core: *A. E. Markaki*[1]; S. A. Westgate[2]; T. W. Clyne[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [2]TWI, Granta Park, Great Abington, Cambridge CB1 6AL UK

A sandwich material, based on a pair of thin stainless steel faces plates separated by a core incorporating stainless steel fibres, has recently been developed. This material has the potential to exhibit an attractive combination of properties, while retaining formability and general handling characteristics similar to those of conventional steel sheet. Three different core structures have been investigated: (a) transversely-aligned fibres bonded to the face plates by adhesive, (b) a sintered fibre mat bonded to the face plates by adhesive and (c) a sintered fibre mat brazed to the face plates. The beam stiffnesses of these three structures have been measured and compared with theoretical predictions. Through-thickness electrical conductances were measured and compared with predictions from simple analytical models. These data were correlated with the results of spot welding tests. It is shown that structure (c) is readily weldable, whereas there are certain difficulties with the adhesively-bonded sheet.

9:50 AM Break

10:10 AM
Constructed Cellular Metals: *David J. Sypeck*[1]; [1]University of Virginia, Matls. Sci. Eng., 116 Engineer's Way, Charlottesville, VA 22911 USA

Cellular solids are made up from interconnected networks of solid struts or plates that form the edges and faces of cells. To date, the main obstacle to obtaining superior properties with materials of this type has involved gaining good control over the distribution of material at the cell level. For many years, it has been known that cellular designs having the highest weight specific stiffness and strength are truss-like when the solid volume fraction is small. Imitating decades of work by structural engineers, cellular solids researchers have now begun to investigate open periodic cell lattices (trusses) with controlled distributions of material at the cell level. These are miniaturized versions of the large engineering type structures (e.g. skyscrapers, bridges, transmission towers, etc.) that have already been made and tested for many years. In this work, efficient truss architectures are identified, methods for affordably making them from metals are presented and properties are assessed.

10:35 AM
Cellular Structure by Direct Metal Deposition: *Jyoti Mazumder*[1]; [1]University of Michigan, Ctr. for Laser-Aided Intelligent Mfg., 2350 Hayward St., 2041 G.G. Brown Bldg., Ann Arbor, MI 48109-2125 USA

The Direct Metal Deposition (DMD) process is creating considerable contemporary interest due to its capability to deliver "Art to Part". DMD can reduce the lead time for a concept to product by eliminating several intermediate steps. The most attractive feature of the process is that not only can it produce functional parts but it can also be interfaced with the Homogenization Design Method (HMD), Heterogeneous Solid Model (HSM) and Computer Aided Design (HCAD) software to produce "Designed Material" with desired properties generally not observed in nature. Closed Loop DMD is a synthesis of multiple technologies including lasers, sensors, Computer Numerical Controlled (CNC) work handling stage, CAD/CAM software and cladding metallurgy. This paper describes the methodology used for designed cellular structures for closed loop DMD.

11:00 AM
Reinforced Alulight for Structural Use: *Frantisek Simanik*[1]; Walter Rajner[2]; Rainhard Laag[3]; [1]Institute of Materials and Machine Mechanics SAS, Ra ianska 75, Bratislava SK-831 02 Slovakia; [2]Alulight Int. GmbH, Ranshofen Austria; [3]Alulight Deutschland GmbH, Wasseralfingen Germany

PM foams can be prepared with pore size gradient and with preferred orientation of pores. There are almost no constraints in the complexity of outer shape and geometry. PM foams are always covered by a dense skin which improves the mechanical properties (e.g. bending stiffness, etc.). However the natural skin of foams has variable thickness and sometimes contains small holes or cracks. Reinforcing of tensile loaded surface skin with metallic or ceramic wires or fibers woven into grids with various mesh size can improve mechanical properties. The possibility to reinforce the foamed part selectively and anisotropically according to the expected load can produce significant improvement in the stiffness, strength, plasticity, energy absorption capacity and damage tolerance of foamed part with only slight weight increase (ca. 20-30%). The reinforcements prevent liquid foam from collapsing on cooling and thus they have also an additional stabilizing effect. Moreover the reinforcements increase the thickness of surface skin, simplify joining of foamed parts (welding is possible), and enable limited shaping after the foaming process.

Processing of Refractory Metals and Alloys - I

Sponsored by: Structural Materials Division, Refractory Metals Committee
Program Organizers: Clyde Briant, Brown University, Division of Engineering, Providence, RI 02912 USA; Pete Lipetzky, Pittsburgh Materials Technology, Inc., Large, PA 15025 USA

Monday AM — Room: 209
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Clyde Briant, Brown University, 182 Hope St., Providence, RI 02912 USA; Pete Lipetzky, Pittsburgh Materials Technology, 811 Rte 51 S., Bldg. #9, Large, PA 15025 USA

8:30 AM Opening Comments

8:35 AM
Refractory Alloy Development: *Pete Lipetzky*[1]; [1]Pittsburgh Materials Technology, Inc., 1801 Rte. 51, Bldg. 9, Jefferson Hills, PA 15025 USA

Over the last 40 years numerous refractory metals and other advanced alloy systems have been investigated for applications ranging from power generation to rocket nozzles to chemical processing equipment. The materials were primarily in the families of Mo-W, Ta-Nb, V-, Zr-, and Re-based. Alloy additions such as silicon were used to perform seminal work in intermetallics and in-situ composites. The fabrication of these alloys were by powder metallurgical methods and AC vacuum arc remelting, often followed by extrusion, forging, swaging and rolling. Microstructural effects on mechanical, electrical, corrosive and oxidation properties were investigated. Furthermore, ultra-high vacuum (UHV) heat treating and mechanical testing were utilized. These UHV facilities were used to demonstrate that creep properties are sensitivity to vacuum level in some alloys. Some successful results will be discussed.

8:55 AM

Processing of Refractory Metals using Radiant Arc-Lamp Heating: *E. K. Ohriner*[1]; C. A. Blue[1]; T. J. Huxford[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6083 USA

Radiant arc-lamp heating offers the potential for direct production of melted sheet products and foil. A focussed 300 kW arc lamp provides incident radiant heat at a flux of up to 3 kW/cm2. Among the refractory metals the melting of tungsten, rhenium, and tantalum have been demonstrated. The process offers the advantages of: 1) high efficiency of conversion of electrical energy to absorbed incident energy, 2) the ability to transmit radiant energy through a quartz window enabling environmental isolation of processed material, and 3) processing of large areas of material at rates of the order of 10 cm2/s. Current research efforts include the direct production of rhenium metal sheet from sintered powder compacts and include computer modeling of melting and solidification sheet materials.

9:15 AM

The Effects of Initial Grain Size on Pure Tantalum Processed by ECAE: *S. N. Mathaudhu*[1]; K. T. Hartwig[1]; [1]Texas A&M University, Mech. Eng., 319 Eng./Physics Bldg., Spence St., College Station, TX 77843-3123 USA

Bulk VAR tantalum with three different initial grain sizes (as-cast: >5mm, large: 200-800 microns, medium: 20-80 microns)was deformed to plastic strains of 1.16, 2.32, and 4.64 by equal channel angular extrusion (ECAE). Results of hardness measurements and metallography are reported. Recrystallized grain size and morphology were measured for each case. Results indicate that the recrystallization temperature was mostly a function of plastic strain, and was independent of initial grain size. It was also observed that the microstructural morphology was similar after two ECAE passes, and that fine(<20 micron), equiaxed grains were developed after four passes for all three starting grain sizes.

9:35 AM

Tensile Properties and Microstructure of the Electroformed Rhenium: *Alexander Smirnov*[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

Rhenium has high a melting point, high density, ductility and good strength at elevated temperature. A number of manufacturing methods are being applied to rhenium to produce near-net shape products. Electroforming process in molten salts is used by Engelhard-CLAL to produce high-density, porosity-free rhenium coatings for different applications. Electroformed rhenium must meet various requirements and the most impotent of them are tensile properties. It is shown that tensile properties of electroformed rhenium depend on the microstructure of the material. The influence of various conditions of electrolyses, annealing parameters and cold deformation on microstructure of the electroformed rhenium and tensile properties were studied. The stress-strain behavior and the microstructure of electroformed rhenium will be discussed.

9:55 AM Break

10:15 AM

Development, Characterization, and Li Compatibilty of an Electrically Insulating CaO Coating on V-4Cr-4Ti Alloy: *Mehmet Uz*[1]; K. Natesan[2]; [1]Lafayette College, Cheml. Eng. Dept., 341 AHE, Easton, PA 18042 USA; [2]Argonne National Laboratory, Energy Tech. Div., 212/G180, 9700 S. Cass Ave., Argonne, IL 60439 USA

As part of the US Department of Energy's Fusion Reactor Program, an electrically insulating and liquid Li compatible CaO coating is being developed on V-4Cr-4Ti alloy. This alloy is the primary candidate for various fusion reactor structural applications including the first wall structure/blanket. The first wall will be in contact with liquid Li coolant on the outside, and electrical conductivity across the wall will lead to magnetohydrodynamic, pressure losses during flow of the conducting liquid in the magnetic field. Hence, it is essential that a coating be applied to the alloy, and that it be electrically insulating and compatible with a liquid Li environment at temperatures of 500-700°C. This paper deals with the development and characterization of a CaO coating on V-4Cr-4Ti alloy specimens by a vapor transport process with double Ca-deposition/oxidation steps. The specimen surfaces were analyzed after each step during the process, and also after exposure to liquid Li together with their cross sections. The analysis and characterization were done using one or more techniques, including scanning electron microscopy, energy dispersive X-ray analysis, X-ray diffraction, and measurement of electrical resistivity external to the Li. Results are presented with emphasis on the details of the experimental procedure used and the microstructural characteristics of the coated samples. This work has been supported by the US Department of Energy, Office of Fusion Science, under Contract W-31-109-Eng-38.

10:35 AM

Effects of Processing and Prolonged High Temperature Exposure: *Mehmet Uz*[1]; Robert H. Titran[2]; [1]Lafayette College, Cheml. Eng. Dept., 341 AHE, Easton, PA 18042 USA; [2]NASA-GRC, 27146 Winwood Way, Olmsted Falls, OH 44138 USA

A systematic study was carried out to evaluate the effects of thermomechanical processing and prolonged high-temperature exposure on the microstructure of Nb-1 wt.% Zr-0.1 wt.% C alloy. All samples were from sheet stocks fabricated by cold rolling of separate billets that were single-, double-, or triple-extruded at 1900 K with a 4:1 ratio. Samples were annealed for 1 h at 1755 K and/or 2 h at 1475 K and subsequently exposed to 1350 K for up to 34,500 h with and without applied load. A combination of various analytical and metallographic techniques was used to characterize the microstructures of sheet samples, as well as the residue extracted from them after each step. The results show that the carbides, which are relatively coarse orthorhombic Nb2C in the as-rolled samples, transform upon subsequent heat treatment to rather fine cubic carbide of Nb and Zr with various Zr/Nb ratios. Also, the relative amount of cubic carbides and the Zr/Nb ratio increase as the number of extrusions increase before cold rolling. Furthermore, prolonged exposure to 1350 K gives rise to complete transformation of Nb2C to (Zr,Nb)-C regardless of the processing history. These and other observations are presented with the emphasis on the effects of processing on microstructure.

10:55 AM

Undercooling Experiments on the Zr-Ta System from Drop-Tube Processing: *Bernard Vinet*[1]; [1]CEA, DTEN/SMP/LESA, 17 rue des Martyrs, Grenoble 38054 France

Deep undercooling on refractory metals and alloys can be obtained during a few seconds in the 48-m high drop tube facility built in Grenoble. A first class of materials has been considered by alloying hcp Re with bcc W, Ta, Nb and Mo metals. With the support of first-principle calculations, this study was focused on nucleation and phase phenomena involving tcp phases [see PRL 83(1999)1621]. In the course of this new program, undercooling experiments are realized on a new class of materials where the above bcc metals are alloyed with elements from the group IVB. Characteristics results obtained on Zr-Ta alloys will be described including microstructures, exploitation of recalescence profiles and microhardness tests. The undercoolability of pure Zr as well as the formation of the metastable omega phase in solidified samples will be also discussed.

11:15 AM

Anomalous High Content of Strongly Bound Water in Plastically Bent Tungsten Single Crystals: *Oleksandr Illich Dekhtyar*[1]; Victor Ivanovich Patoka[1]; Mykola Yakimovich Shevchenko[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

The chemical composition of tungsten single-crystalline plates with the surface orientation {110} by 1.2-1.5 mm thickness plastically bent in vacuum around axis <112> with the help of electron beam heating at 1170-1270 K temperatures up to 20-25% strain have been studied by secondary ion emission. There was found the anomalous high content of water in the bulk of tungsten. This content is essentially higher than one of usual insert impurities (C, O, N) and other elements and compounds. There was studied the water amount after high temperature annealings. It is found that high water amount conserves up to 2700 K. Such strongly bound state of water molecules is caused by existing of the high excess density of one sign dislocations which conserves up to near tungsten melting point. The water molecules have to be finding way into the dislocation cores as ionized molecules.

Second International Symposium on Ultrafine Grained Materials: Processing and Structure - I

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

Monday AM — Room: 210
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Yuntian T. Zhu, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; Terry C. Lowe, Metallicum, LLC, 1207 Callejon Arias, Santa Fe, NM 87501 USA

8:30 AM Keynote
Continuum Mechanics Approach in Severe Plastic Deformation: *Vladimir M. Segal*[1]; [1]Honeywell Electronic Materials, STAR Ctr., R&D, 15128 E. Euclid Ave., Veradale, WA 99216-1895 USA

The presentation considers some aspects of severe deformation processing: 1. Early development in 1970's-1980's. 2. Deformation mode and loading history. 3. Simple shear versus pure shear. 4. Structure evolution during simple shear. 5. Processes of simple shear. 6. ECAE: slip line solutions and deformation mode. 7. ECAE: technological opportunities and challenges. 8. Conclusion

8:55 AM Invited
Powder Consolidation by Equal Channel Angular Extrusion: *Ricardo B. Schwarz*[1]; Jose I. Archuleta[1]; Jenya Macheret[2]; Tom Lillo[2]; Gary Korth[2]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MS G755, Los Alamos, NM 87545 USA; [2]DOE-ID, 850 Energy Dr., MS 1225, Idaho Falls, ID 83401 USA

Equal-channel angular extrusion (ECAE), an innovative deformation process capable of introducing elevated levels of shear while preserving the cross section of the product, has been applied to the consolidation of metal powders. A mixture of spherical copper and silver particles was loaded into square-cross-section copper cans, which were then extruded at 300°C. After three successive ECAE passes (equivalent extrusion ratio of 32), the powder becomes a filamentary Cu and Ag composite. The composites are fully dense and crack free. Their compressive yield strength is 330 MPa and their ductility exceeds 20%. The Young's moduli along and perpendicular to the filaments are 106 and 96 GPa, which equal the values calculated using appropriate averages of the elastic constants of pure copper and silver. The mechanical tests and elastic constant data show that ECAE is a viable method for the consolidation of powders.

9:15 AM
Grain Refinement in Beryllium by Equal Channel Angular Extrusion: *David J. Alexander*[1]; Robert D. Field[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-6, MS G770, Los Alamos, NM 87545 USA

Fine-grained beryllium alloyed with 6 wt% copper is the material of choice for fabrication of targets for the National Ignition Facility. Beryllium is a difficult material to process, because of its limited ductility and its environmental and safety concerns. Equal channel angular extrusion (ECAE) of Be has been performed at elevated temperatures to determine if ECAE is a suitable processing method for production of this material. Be samples 5 mm in diameter and 30 mm long were canned in mild steel to create a billet 10 mm square by 60 mm in length. These billets were extruded through 90° tooling. Several different processing routes were also investigated. Significant grain refinement occurred, even after only a single extrusion pass, with additional refinement for subsequent passes. The microstructures and textures resulting from the different processing routes will be compared, and the suitability of ECAE for production of fine-grained Be and Be alloys will be considered.

9:30 AM
Grain Refinement of Medium Carbon Steel with Controlled Thermo-Mechanical Deformation: Jongmin Park[1]; Dae Hyun Song[1]; Duklak Lee[2]; Wung Yong Choo[2]; Ik-min Park[1]; *Kyung-mox Cho*[1]; [1]Pusan National University, Matls. Sci. & Eng., San 30 Jangjeondong, Kumjungku, Pusan 609-735 Korea; [2]Posco, Ltd., Pohang, Kyungbuk 790-785 Korea

Microstructure of 0.45%C medium carbon steel was tried to control through thermo-mechanical treatment. The purpose is to obtain fine grained microstructure of the cold heading quality steel. With refined microstructure, the heat-treatment time for spheroidization could be reduced greatly. For the thermo-mechanical treatment, deformation temperature, deformation rate and cooling rate were controlled. The steel was deformed in austenite region (above A3) as well as austenite+ferrite region (between A3 and A1) and the resulting microstructure was compared. The grain size of thermo-mechanically treated 0.45%C medium carbon steel up to sub-micro-meter (less than 1 micro-meter) was obtained. The spheroidization heat-treatment time with the refined microstructure was reduced to about 3-5 hours comparing the conventional steel needed 15 hours for spheroidization.

9:45 AM
Heterogeneous Microstructural Evolution and Reactions during Repeated Intense Deformation: *Rainer J. Hebert*[1]; John H. Perepezko[1]; [1]University of Wisconsin, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

With the recent improvements in severe plastic deformation and cold rolling, deformation processing has become an alternative to rapid-solidification processing for the synthesis of bulk metastable structures. The systematic study of the deformation patterns that develop during repeated rolling of multilayer based on a stacked elemental crystalline array including the analysis of interface areas and the evolution of interface roughening, offers an effective approach to the examination of the mechanism involved in deformation alloying, nanostructure synthesis and amorphization. A key-factor for deformation-induced phase transformations is the occurrence of atomic scale mixing effects at interfaces. The results from cold rolling experiments on Al-Pt and Al-Hf alloys demonstrate that the overall multilayer refinement represents an essential component for the prediction of amorphization by cold rolling in addition to the heat of mixing and a deformation enhanced mixing kinetics at individual interfaces. In light of cyclic crystalline-to amorphous reactions reported for ball-milled Co-Ti and Al-Zr alloys, the effect of cold rolling on the devitrification behavior of Al-based glasses has also been addressed in a systematic study. For marginal glass formers, such as melt-spun Al92Sm8, cold rolling induced a complete crystallization without annealing while for melt-spun Al85Ni10Ce5, a crystalline phase has not been observed under the same deformation conditions. The structural changes are observed to develop heterogeneously throughout the sample over several deformation cycles due to the distribution of microstructural size scales. These developments highlight the importance of the localized microstructural response to the overall sample deformation. The support of the ARO (DAAD19-01-1-0486) is gratefully acknowledged.

10:00 AM
On the Development of Microstructure in a Metal Matrix Composite using Nano-Materials: Vladimir Popov[1]; *Donald Lesuer*[2]; Iouri A. Kotov[3]; Viktor V. Ivanov[3]; Andrej A. Aksenov[1]; Igor Khodos[4]; Galina Klimenko[4]; Oleg Smirnov[1]; Ajdar Murzakaev[3]; Sergei Zayats[3]; [1]Moscow State Institute of Steel and Alloys, Leninsky Prospect 4, Moscow 117936 Russia; [2]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA; [3]Institute of Electrophysics of Ural Branch Academy of Science, Ekaterinburg Russia; [4]Institute of Microelectronics Technology and High Purity Materials, Chernogolovka Russia

Metal matrix composites (MMCs) containing matrices with nanometer grain sizes provide opportunities for improved properties. In this paper, we report on MMCs prepared from pure aluminum nano-powders (particle sizes of 50-200 nm) with SiC reinforcement (particle size 3-10 μm). The volume fraction of SiC reinforcement was varied between 20 and 40%. The pure aluminum nano-powders were prepared using an exploding wire technique. The particles of the MMC were mixed and then dynamically compacted to high density using an explosive compaction method. The resulting microstructures were evaluated using optical and transmission electron microscopy. Microstructural analysis of the resulting MMC showed a highly uniform distribution of SiC particles with no visible defects or pores and the absence of deleterious phases (such as Al4C3) at the interfaces between the aluminum nano-grains and the SiC particles. The evolution of microstructure during dynamic compaction has also been studied using TEM and found to progress in three stages. The results of these microstructural evolution studies will be discussed as well as the resulting properties of the MMC. Work performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-74055-ENG-48.

10:15 AM Break

10:25 AM Invited
Fine Scale Microstructures Produced by Sliding Loads: *Darcy A. Hughes*[1]; [1]Sandia National Laboratories, Matls. & Eng. Sci. Ctr., POB 969, MS9405, Livermore, CA 94551-0969 USA

Frictional contact between metals under large sliding loads is a method for introducing extreme strains, thereby producing nano-scale deformation microstructures within the near surface regions. This microstructure consists of layers of finely spaced deformation-induced boundaries. The boundary spacing coarsens with increasing depth from the surface in correspondence to the decrease in deformation. This graded structure ranges in size from 10nm at the surface to a few µms at a depth of 200µm. The microstructural characteristics are quantitatively analyzed and compared to those developed by rolling. The structures in both cases evolve similarly with strain over this large range of scales. The similarity indicates that the deformation mechanisms controlling microstructural evolution are the same from 10 to 10,000nm. This work was supported by the Office of Basic Energy Sciences of the US DOE, Division of Materials Sciences under contract no. DE-AC04-94AL85000.

10:45 AM Invited
Grain Refining Mechanisms of Ti during Equal Channel Angular Pressing: *Yong-Seog Kim*[1]; Jong Youl Kim[2]; Inyoung Kim[2]; Dong Hyuk Shin[2]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Mapo Ku, Seoul 121-791 S. Korea; [2]Hanyang University, Metlgcl. & Matls. Sci., Sa 1 Dong Ansan Si, Kyunggi-Do 425-791 S. Korea

Microstructural development of titanium during equal-channel angular pressing was investigated to understand the mechanisms of grain refinement and strain accommodation. The samples were processed via three different pressing routes: A, B and C. TEM micrographs of the sample after the first pass showed that {1011} deformation twinning plays a primary role in accommodating the plastic deformation imposed by the pressing. During subsequent passes, plastic deformation was accommodated mainly by dislocation slip and its slip system was dependent on the pressing routes: a slip on prismatic planes when processed via route B, a slip on basal planes and a+c slip when processed via route A, and a slip on prismatic planes and deformation twinning when processed via route C. These differences between the routes were discussed based on the preferred orientation and the CRSS of each slip mode.

11:05 AM
Grain Refinement and Texture Development in Asymmetrically Rolled Aluminum Alloy Sheets: *Dong Nyung Lee*[1]; Su-Hyeon Kim[1]; Jong Kook Lee[1]; [1]Seoul National University, Matls. Sci. & Eng., Shinrim-dong, Seoul 151-742 Korea

Asymmetric rolling, in which the ratio of upper to lower roll diameter is different, has been used to introduce an intense plastic shear strain for the purpose of grain refinement and in turn strengthening and of obtaining higher plastic strain ratios of aluminum sheets due to shear deformation textures through the sheet thickness. Commercial purity aluminum sheets were asymmetrically cold rolled without lubrication and the strengths and microstructures of the sheets were investigated by tensile testing, transmission electron microscopy (TEM), and electron back-scatter diffraction (EBSD) analysis. After several passes of asymmetric rolling, a fine grain structure with large misorientations developed in the sheets and their strengths increased. Unidirectional rolling in which the rolling direction does not change between passes gave rise to more uniformly refined structures than alternating direction rolling in which the rolling direction is rotated through 180º about the rolling direction between passes.

11:20 AM
Effect of Vanadium Addition on Dynamic Gamma to Alpha Transformation during Hot Deformation of Low Carbon Steels: *Hu-Chul Lee*[1]; Jae-Young Cho[1]; Joo-Hee Kang[1]; Dong-Woo Suh[2]; [1]Seoul National University, Matls. Sci. & Eng., Shinrim-dong San 56-1, Kwanak-ku, Seoul 151-742 Korea; [2]National Institute of Materials Science, Matl. Creation Rsrch. Grp., 1-2-1 Sengen, Tsukuba 305-0047 Japan

The effect of 0.06-0.2% vanadium and 0.01wt% nitrogen addition on the dynamic gamma to alpha transformation in a low carbon steel (Fe-0.15wt%C-1.2wt%Mn-0.3wt%Si-0.02wt%S) was investigated. The 0.06-0.2wt%V steel and base steel were heated to 1080°C and 960°C, respectively, held for 5 minutes to produce an austenite grain size of about 100 microns, and deformed for 25%-70% in a single pass at 700°C, just above Ar3 temperature, using laboratory deformation simulator. The dilatation curve was measured for 10 minutes after deformation and normalized by the diameter of specimen just after deformation. The volume fraction of dynamically transformed ferrite was evaluated from dilation curves. The volume fractions of dynamically transformed ferrite were 21% and 52% in base steel and 35 and 54% in 0.2%V steel, respectively, after deformation of 25% and 40%. When the deformation was higher,the effect of vanadium addition was diminished. After deformation of 40% or higher, the base and 0.2wt%V steel show dual grain structure of fine ferrite grain (about 3-4 microns) which are formed and grown during deformation and coarse ferrites (about 7-8 microns) which are formed and grown during isothermal holding at 700°C. These results show that vanadium addition to low carbon steel enhances not only the static but also the dynamic transformation resulting in the refinement of ferrite grain size. The results of TEM analysis on the role of vanadium carbo-nitrides in dynamic transformation is also discussed.

11:35 AM
Nano-Nano Composites of Silicon Nitride and Silicon Carbide: *Julin Wan*[1]; Matt J. Gasch[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

This is an effort of bringing the grain size scale of silicon nitride and silicon carbide composites into the truly nanometric range, i.e., with the grain size of both silicon nitride and silicon carbide within 100nm limit. Amorphous Si-C-N bulk materials were first synthesized by a processing route based on pyrolysis of polymer precursor. By controlling the crystallization heat-treatment at relatively low temperature levels, and without oxide sintering additives, silicon nitride/silicon carbide composites with grain size of 30-50nm can be achieved. A second processing method of processing was high pressure sintering. Oxide additives were incorporated into precursor pyrolysis-derived powders, and sintering was conducted at 1400-1600°C under a pressure of 1-2GPa. Although in this case liquid phase sintering was involved to some extent, the combination of high-pressure, low temperature, short duration and the nature of starting powder effectively hindered grain growth during sintering, leading to nano-nano microstructures. This research is supported by the US Office of Naval Research (Grant No. N00014-00-1-0186).

11:50 AM Invited
Grain Refinement and Phase Transformations in Al and Fe Based Alloys during Severe Plastic Deformation: *Sergey Dobatkin*[1]; [1]Moscow State Steel and Alloys Institute, Dept. of Plastic Deformation of Special Alloys, Leninsky prospekt, 4, Moscow 119991 Russia

The present paper shows the possibility of additional refinement of the structure up to nanoscale size in Al and Fe based alloys during severe plastic deformation (SPD) by torsion under high pressure (HPT) due to phase transformations. 1. Al-Cu-Mg alloy. Severe deformation of oversaturated solid solution of Al-Cu-Mg-alloy after quenching allows to overcome the nanoscale of grain size (less then 100 nm) due to strain aging. 2. Al-Mg-Sc alloys. During hot SPD by HPT of Al-Mg-Sc alloy 01570 at T=400°C the UFG structure with grain size of about 150 nm is formed at the expense of decomposition of a solid solution. 3. Low-carbon steels. SPD of the initially quenched 0,09CMnSi and 0,1CMnVTi steels leads to a more dispersed nanoscale structure than that of initially hot rolled steels. The possibility of precipitation and dissolution of carbides are discussed. 4. Austenitic stainless steels. The martensitic transformations in austenitic steels during severe cold deformation by HPT promote the additional refinement of structure.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": From Fundamentals to New Projects

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

Monday AM	Room: 607
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Robert L. Stephens, TeckCominco Metals Ltd., PO Box 1000, Trail, British Columbia V1R 4L8 Canada; H. Y. Sohn, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112 USA

8:30 AM Plenary
Continuous Coppper Converting–A Perspective and View of the Future: *David B. George*[1]; [1]Kennecott Utah Copper Corporation, 8315 W. 3595 S., PO Box 6001, Magna, UT 84044-6001 USA
In spite of the many well known problems inherent in Peirce-Smith converting, it has remained the dominant copper converting process for nearly 100 years. Continuous copper converting and direct concentrate to blister smelting have been commercially available for over 20 years and now produce roughly 15% of the world's primary copper. The fully commercialized continuous converting processes, the Mitsubishi MI Process and Kennecott-Outokumpu Flash Converting, and several emerging converting concepts are reviewed with view to their potential, limitations, and environmental performance. Some myths regarding these technologies and Peirce-Smith converting are also challenged.

9:05 AM Plenary
Basic Principles of Sulfide Smelting and Converting with Oxygen-Rich Gas: Kimio Itagaki[1]; *Hong Yong Sohn*[2]; Manuel Pérez-Tello[3]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Katahira 2-1-1, Aoba-Ku 980-8577, Sendai Japan; [2]University of Utah, Dept. of Metallurgical Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [3]University of Sonora, Dept. of Cheml. Eng. & Metall., Hermosillo, Sonora 83000 Mexico
Thermodynamics and rate process fundamentals are two of the important principles involved in nonferrous production processes. Process configuration establishes whether thermodynamics or rate process behavior is most relevant in a particular application. In this paper, thermodynamic theory and rate process fundamentals are used to analyze the main characteristics of smelting and converting processes. The effect of increased oxygen potential is examined with emphasis, considering the increased use of highly oxygen-enriched gas in modern smelting processes. The phase equilibrium and the distribution of minor elements between the copper or nickel matte and the FeOx-SiO2 or FeOx-CaO slag are reviewed. The use of oxygen-rich gas has no serious effect on the recovery of valuable copper and silver in the matte phase, while the recovery of nickel and cobalt is adversely affected. The oxygen content in the gas also strongly affect the distribution behaviors of arsenic, antimony and bismuth. The rate processes occurring in the shaft of flash smelting and converting furnaces are analyzed by a three-dimensional computational fluid-dynamics model. The computer model is used to simulate the main features of an industrial flash converting operation. The role of basic principles in the engineering analysis of high-intensity matte smelting processes is discussed.

9:40 AM Cancelled
Considerations on Slag Chemistry of the Noranda Process Reactor: *M. Zamalloa*

9:40 AM
Kinetics of Sulfide Smelting in Mitsubishi Process: *Zenjiro Asaki*[1]; [1]Mitsubishi Materials Corporation, Central Rsrch. Inst., 1-297, Kitabukuro-cho, Saitama 330-8508 Japan
The copper concentrate particles dissolve quickly into the molten matte in the Smelting furnace of Mitsubishi Process, which indicates that the sulfide smelting is essentially gas/liquid reaction. In this paper, theoretical calculation has been made on the rate of oxidation of FeS component in the matte to form FeO by rising gas bubbles using the penetration theory proposed by Higbie. The results showed that all the oxygen in the bubble of 1.2 cm in diameter, which is the maximum stable bubble diameter, is consumed in 3 ms. This time required is sufficiently short, and it results in the actual operation of smelting furnace of 100% oxygen-efficiency. This also indicates that most smelting reactions are completed just under the lance except slag formation because the dissolution rate of silica particles is rather low.

10:05 AM Invited
The Ignition of Sulphide Flotation Concentrates in Flash Smelting: *Frank R.A. Jorgensen*[1]; [1]CSIRO Minerals, PO Box 312, Bayview Ave., Clayton, Victoria 3169 Australia
Flash smelting is used to smelt a large proportion of the world's copper and nickel and some lead/zinc concentrates. Understanding the factors controlling the ignition of these concentrates is an important consideration in designing burners and maximizing the throughput of the flash smelting process. The literature on the subject is briefly reviewed and techniques for measurement of ignition temperature are discussed. The mineralogy of the concentrates and the role of thermodynamics and phase diagrams in interpreting and predicting ignition behaviour are emphasized and applied to interpreting results obtained on a nickel concentrate.

10:30 AM Break

10:45 AM
Gresik Copper Smelter and Refinery–Current Operation and Expansion Plan: *Moto Goto*[1]; [1]PT Smelting Co., Desa Roomo, Kecamatan Manyar, PO Box 555, Gresik, Jawa Timur 61151 Indonesia
PT Smelting, located in Gresik, East Java, is the first copper smelter and refinery in Indonesia. Since the first charging concentrate in December 1998, many equipment troubles have been systematically eliminated, and the present operation is smooth and has accomplished the design capacity of 200,000 tonnes per year of LME, grade-A copper cathode. This paper describes troubles and countermeasures, which were experienced until now.

11:10 AM
Budel Zink Sets a New Standard for NO_x Reduction in a Sulphuric Acid Plant: J. van Driel[1]; B. Giesen[1]; *A. Berryman*[2]; S. Sampat[2]; S. Enevoldsen[3]; H. Jensen-Holm[3]; [1]Budel Zink B. V., Hoofdstraat 1, 6024 AA Budel-Dorplein The Netherlands; [2]Kvaerner Chemetics, A Div. of Kvaerner Canada, Inc., 1818 Cornwall Ave., Vancouver, BC V6J IC7 Canada; [3]Haldor Topsøe A/S, Nymollevej 55, DK-2800 Lyngby Denmark
NO_x is a growing concern for acid plant operators due to recent legislation reducing the allowable stack limits, as well as continually more stringent requirements for product acid quality. NO_x also causes a major problem during maintenance as NO_x rich acid residues fume following exposure to moisture in the air. Until recently, the solution for NO_x related issues was to treat the symptom, rather than the cause. NO_x in product acid was treated using chemicals and the NO_x in the plant stack was generally left untreated. As part of an environmentally driven change in concentrates, Budel Zink installed the first gas phase NO_x destruction process to be incorporated into a commercial scale acid plant. Nitrogen oxides contained in the process gas are reduced selectively with ammonia over a catalyst to form nitrogen and water. As a result, the NO_x reduction is in agreement with the permit requirements.

11:35 AM
An Overview on Operation of Jinlong's Copper Flash Smelting Since Start-Up: Bao Xian Cheng[1]; Song Xiu Ming[2]; *Jun Zhou*[1]; [1]Jinlong Copper Co., Ltd., Prod. Dept., Jinshan Rd., Tongling, Anhui 244021 China; [2]Jinlong Copper Co., Ltd., Tech. Dept., Jinshan Rd., Tongling, Anhui 244021 China
Jinlong's copper flash smelting process was started up in April, 1997. In the early days of production lots of equipment and process problems were encountered. But through continual improvements in equipment and operation, designed productivity was reached two years later, and smelting operation has been greatly upgraded since then. After more than 4 years of production, we have made great achievements, such as high flash furnace availability, high matte grate operation, optimization in concentrate burner's structure and operation, stable operation of converter and anode furnace, and etc. Smelter emission control has reached nation's standard since commissioning. This paper intends to outline Jinlong's operational achievements made after commissioning, analyze problems ever encountered and their remedies. Operational experience is also summarized.

Advances in Metallic Glasses: Glass Formation, Consolidation and Microstructure

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

Monday PM — Room: 212
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: En Ma, Johns Hopkins University, Dept. of Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218-2689 USA; Krishna Rajan, Rensselaer Polytechnic Institute, Dept. of Matl. Sci. & Eng., Troy, NY 12180-3590 USA

2:00 PM
Structure-Forming Principles for Amorphous Metals: *Kevin L. Kendig*[1]; Daniel B. Miracle[1]; Oleg N. Senkov[2]; W. Sanders[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., AFRL/MLLMD, Dayton, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

After extensive experimental and computational research over the past several decades, there is still a rather poor understanding regarding the structure of metallic glasses. The clearest picture exists for metal-metalloid glasses, yet this still provides only an incomplete understanding. The chemical and topological aspects of these systems differ, sometimes strongly, from many other well-established metallic glass systems, so that the fundamental structure-forming principles which operate in metallic systems are still not established. The purpose of this research is to propose specific ordering principles, which may govern the structural formation of metallic glasses. Earlier concepts of high atomic packing efficiency will be extended and applied to topological systems, which represent most metallic glass alloys. The concept of high packing efficiency has specific implications for both short- and medium-range atomic configurations, which will be presented. Possible atomic configurations will be explored, and consistency with experimental observations will be provided and discussed.

2:20 PM
Atomistic Mechanism of Bulk Metallic Glass Formation: *Takeshi Egami*[1]; Hao Chen[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., LRSM, 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Microscopic mechanism of bulk glass formation is discussed from a very general point of view, focusing on the size factor and interatomic potential. A key for the bulk glass formation is the "strong" glass behavior, as defined by Angel, that keeps the diffusivity of the melt low well above the glass transition temperature. A concept of distributed local glass transition is introduced to explain the strong glass behavior. In a multi-component system each element has a different local topological instability condition that defines the glass transition. This allows calculation of the local glass transition temperature for each component of the system. Widely distributed local glass transition temperatures result in the strong glass behavior. Molecular dynamics simulations to demonstrate this effect will be presented.

2:40 PM
Mechanical Modeling of the Structure of Amorphous Al: *Daniel B. Miracle*[1]; Kevin L. Kendig[1]; Oleg N. Senkov[2]; Robert Smith[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

A mechanical modeling approach has been applied to explore the structure of amorphous Al alloys. Polyethylene spheres were used to represent atoms within the structure. The number and relative sizes of the spheres were selected to represent typical amorphous Al alloy glass compositions. Arrays of 1000 atoms were used in the models. The spheres were loaded in an elastic container to minimize the influence of the free surface, and to allow introduction of arbitrary shear deformations to randomize the structure. Measurements of free volume for monosized spheres agree very well with accepted values from the literature, providing confidence in the methodology used. The coordinates of each atom in the "amorphous Al glass" arrays were measured, and provided the ability to obtain and study local atomic configurations and to determine partial and total radial distribution functions. The system free volume was also measured and compared with literature values. The results obtained from these models will be presented and discussed.

3:00 PM
Synthesis of $Ti_{34}Zr_{11}Ni_8Cu_{47}$ Bulk Metallic Glass by Warm Extrusion of Gas Atomized Powders: *Daniel Sordelet*[1]; Elena Rozhkova[1]; Matthew Kramer[1]; Paul Wheelock[1]; Matthew Besser[1]; Monique Calvo-Dahlborg[2]; Ulf Dahlborg[2]; [1]Ames Laboratory/Iowa State University, Metall. & Cer./Dept. of Matls. Sci. & Eng., 107 Metals Dvlp., Ames, IA 50014 USA; [2]LSG2M-UMR7584, Ecole des Mines, Nancy France

Bulk metallic glass was prepared by warm extrusion of $Ti_{34}Zr_{11}Cu_{47}Ni_8$ gas atomized powders. After consolidation near 723 K using an extrusion ratio of 5, the material retains between 88% and 98% of the amorphous structure found in the gas atomized powder. The onsets of the glass transition and crystallization temperatures of this extruded material are observed, respectively, at slightly higher and lower temperatures than those of the starting powders. Powders extruded at the same temperature, but using higher extrusion ratios of 9 and 13, exhibit substantial devitrification during consolidation, yet still deform homogeneously. A thin layer of a nanocrystalline phase rich in Ti and Zr was observed at some particle boundaries of extruded samples. Although the gas atomized powders contain approximately 700 ppm by weight O, parallel energy loss spectroscopy did not detect any O within this nanocrystalline phase. This devitrification may be related to localized frictional heating at the particle interfaces during extrusion.

3:20 PM
Consolidation of Metallic Glass Powders with Equal Channel Angular Extrusion: *K. T. Hartwig*[1]; I. Karaman[1]; Jae-Taek Im[1]; J. Robertson[1]; I. E. Anderson[2]; [1]Texas A&M University, Dept. of Mechl. Eng., MS-3123, College Station, TX 77843 USA; [2]Iowa State University, Ames Lab., Ames, IA 50011 USA

Previous research has shown that bulk amorphous metals can be fabricated by warm extrusion of rapidly solidified amorphous particulate. In the present study, the effectiveness of equal channel angular extrusion (ECAE) for consolidation of amorphous Zr-based powder is examined. ECAE subjects the workpiece to uniform simple shear but not area reduction and offers the opportunity for large cross section products. Powder, produced by gas atomization, was vacuum encapsulated in copper cans and extruded at a temperature above the glass transition temperature but below the crystallization temperature where the alloy behaves temporarily as a viscous liquid. The effects of extrusion temperature, strain rate, and strain level on consolidate porosity, degree of crystallinity and microstructure were examined. Results along with property comparisons to cast alloy are presented. Support for this work provided by DARPA under contract no. DAAD 19-01-1-0481.

3:40 PM Break

4:00 PM
Mechanical Properties of ECAE Consolidated Zr-Based Bulk Glassy Alloys: *Ibrahim Karaman*[1]; K. T. Hartwig[1]; J. Robertson[1]; [1]Texas A&M University, Dept. of Mechl. Eng., MS 3123, College Station, TX 77843 USA

Bulk metallic glasses (BMG) have received increasing interest during recent years because they are promising for use in engineering applications. Although many amorphous alloy systems have been known and studied for decades, their use for practical applications has been limited because of high cooling rate requirements and resulting product with at least one small dimension. Consolidation of particulates can circumvent this problem. The method involves viscous flow and atomic diffusivity in the supercooled liquid region with resultant fabrication of full density bulk glassy alloys. In the present work, a severe plastic deformation technique, equal channel angular extrusion (ECAE) is used for consolidation of atomized metallic glass powders. We will present a comparison of the mechanical properties of cast and conventionally extruded metallic glass powders with samples obtained by ECAE consolidation. The materials under study are Zr-based glassy alloys that are chosen for their high glass forming ability. Tensile, compressive and hardness properties are compared.

4:20 PM
Modeling of Thermal Tempering in Bulk Metallic Glasses: *Cahit Can Aydiner*[1]; Ersan Üstündag[1]; [1]California Institute of Technology, Eng. & Appl. Sci./Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

Bulk metallic glasses (BMGs) have recently been shown to possess good mechanical properties and are suitable candidates for many structural applications. One of the critical issues that influence their properties is the existence of residual stresses in them. In this case, they originate due to "thermal tempering". In this process, the viscoelastic nature of these materials and the high cooling rates necessary in their production lead to compressive surface residual stresses balanced with mid-tension. We have modeled the development of these stresses starting with a simple instant freezing model and progressing towards a more complicated and data intensive structural model. This talk will compare the predictions of these models. The results suggest the existence of large stress gradients on specimen surface.

4:40 PM
Internal Stresses in Tungsten Fiber Reinforced Bulk Metallic Glass Matrix Composites: *Bjorn Clausen*[1]; Seung-Yub Lee[1]; Ersan Üstündag[1]; Mark A.M. Bourke[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, MST-8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA

Bulk metallic glass (BMG)-matrix composites have been shown to increase the ductility of BMGs. However, the matrix-reinforcement interactions have not been fully understood. One important issue in such composites is the presence of internal stresses due to the mismatch in coefficients of thermal expansion between the matrix and the reinforcements as well as their elastic and plastic incompatibility under loading. In the present study, we have used neutron diffraction to measure the thermal residual strains in a series of bulk metallic glass matrix composites with varying volume fraction of tungsten fibers. As the neutron diffraction measurements only yield information about the fibers, finite element modeling has been used to predict the thermal residual strains in the BMG matrix. These composites have then been loaded in compression to study the in-situ mechanical deformation of the reinforcements and to understand their interaction with the matrix.

5:00 PM
Measurement of Residual Stresses in Bulk Metallic Glasses: *Cahit Can Aydiner*[1]; Ersan Üstündag[1]; [1]California Institute of Technology, Eng. & Appl. Sci./Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

Recent studies have suggested that large residual stresses exist in bulk metallic glasses (BMGs) due to "thermal tempering". This mechanism involves quenching a viscoelastic material from above its glass transition region to room temperature. During this process, compressive residual stresses are generated on the surface while the middle is under tension. While such stresses could be measured conveniently by photolelasticity in silica-based glasses, the opacity of BMGs prevent the application of this method. Similarly, the amorphous structure of BMGs precludes the use of diffraction. We have, therefore, employed mechanical relaxation methods in this investigation. In this presentation, results will be shown from relaxation methods including hole drilling, layer removal and crack compliance. These results will then be compared to modeling predictions.

Advances in Molten Salt Processing Technology: Process Fundamentals

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439-4837 USA

Monday PM	Room: 614
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div., Argonne, IL 60439 USA

2:00 PM Opening Remarks

2:05 PM
Determination the Free Energy of Formations of Na2Cr2O4 and Na2CrO4 using Na -b"-Al2O3 Solid Electrolyte: *Vilas D. Tathavadkar*[1]; M. P. Antony[2]; Animesh Jha[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK; [2]Post-Irradiation Studies Section, Fuel Chem. Div., IGCAR, Kalpakkam, TN 603 102 India

The thermodynamic properties of the Na2O-Cr2O3 mixtures were investigated using a Na -b"- Al2O3 solid electrolyte in the temperature range of 1100 to 1200 K. During the soda-ash roasting of chromite ores, both sodium chromite and sodium chromate are formed depending on the oxygen partial pressure and the amount of sodium carbonate. The experiments were carried out to measure the activity of Na2O in Na2O-Cr2O3 system using the following e.m.f cells: CELL-I :(-) Pt / Ar / Na2Cr2O4 + Cr2O3 || Na-b"-Al2O3 || Na2WO4 + WO3 / Air / Pt (+) CELL-II :(-) Pt / Air / Na2CrO4 + Cr2O3 || Na-b"-Al2O3 || Na2WO4 + WO3 / Air / Pt (+) The free energy of formation of sodium chromite and sodium chromate from solid Na2O and Cr2O3 was calculated using the e.m.f. values of cell I and II, and the activity of Na2O in Na2WO4 + WO3 reference electrode. The derived equations for DGo in the range 1100 to 1200 K for Na2Cr2O4 and Na2CrO4 are -213153 + 7.0172 T J/mole (+ 8 kJ) and -332173 + 64.164 T J/mole (+ 5 kJ) respectively. The relevance of controlling the oxygen partial pressure for the formation of water soluble chromium phases is emphasised. The results are compared with the available thermodynamic data from the literature. The experimental results obtained from the X-ray diffraction confirm the presence of sodium chromite and sodium chromate phases alone with Cr2O3 in the working half cells I and II respectively. The present investigation also compares the liquidus temperatures in the Na2CO3-Na2CrO4 system, which were determined by differential thermal analysis. The measured activities of Na2CrO4 and Na2Cr2O4 confirm the importance of controlling the oxygen partial pressure and soda-to-chromite ratio during the soda-ash roasting of chromite ores.

2:35 PM
Occlusion of Molten Chloride Salt by Zeolite 4A-Nature, Thermodynamics, and Application to Nuclear Waste Treatment and Disposal: *Dusan Lexa*[1]; Irving Johnson[1]; [1]Argonne, Cheml. Tech., 9700 S. Cass Ave., Argonne, IL 60439-4837 USA

Glass-zeolite ceramic waste forms are being developed at Argonne National Laboratory for the disposal of radioactive waste salts from electro-metallurgical treatment of spent nuclear fuel. The major component (70 wt%) of the process salt is the LiCl-KCl eutectic salt (58 mol% LiCl, 42 mol% KCl). In addition, the process salt contains NaCl, at least 16 other fission product chloride and iodide salts, and 2 mol% actinide chlorides, such as UCl3, NpCl3, and PuCl3. The melting temperature of the process salt is ~ 600 K. The selection of an optimal process salt treatment method requires detailed knowledge of the equilibrium ion-exchange zeolite/molten salt separation factors. These have been determined in a number of pseudo-ternary systems, including NaCl-LiCl-KCl, CsCl-LiCl-KCl, RbCl-LiCl-KCl, MgCl2-LiCl-KCl, CaCl2-LiCl-KCl, SrCl2-LiCl-KCl, BaCl2-LiCl-KCl, LaCl3-LiCl-KCl, CeCl3-LiCl-KCl, UCl3-LiCl-KCl, and PuCl3-LiCl-KCl, at 823 K, by chemical analysis of both the zeolite and the molten salt phases.

3:05 PM
Ion Exchange of Fission Products between Zeolite and a Molten Salt: *Mary Lou Dunzik Gougar*[1]; Michael F. Simpson[1]; [1]Argonne National Laboratory-West, PO Box 2528, Idaho Falls, ID 83403 USA

In an electrometallurgical process to treat spent nuclear fuel, a molten chloride salt solution accumulates fission products from the alkali, alkaline earth, lanthanide, and actinide families. One technique that has been identified for removing these fission products and extending the usable life of the molten salt is ion exchange with zeolite A. Salt fills the micropores of the zeolite, and ion exchange of fission products can then occur between the molten salt phase and the solid zeolite phase. Closed-system contact experiments have been performed using both radioactive, fission-product-loaded salt and non-radioactive surrogate salt. The time for the establishment of equilibrium has been determined, and initial data has been obtained in support of the development of a multi-component equilibrium model for the ion exchange of fission products between molten chloride salt and zeolite A.

3:35 PM Break

3:50 PM
Dissolution Kinetics of Silicon-Containing Sodium Fluosilicate in Stirred Bath of Molten Aluminium: Ibrahim Hamed Aly[1]; *Eng Ebrahiem Esmail*[1]; A. M. Omran[2]; [1]Minia University, Cheml. Eng. Dept., Fac. of Eng., Minia 61111 Egypt; [2]Aluminium-Company of Egypt, Matls. Sci. Eng., Nag-Hamady Egypt

In the present study an attempt was made to characterize the mechanism of silicon dissolution from sodium fluo-silicate lumps to molten aluminium. The experiments were carried out under the influences of various process parameters such as temperatures, particle size, Na2SiF6/Al ratio and dissolution time. The obtained results were explained in detailed, they showed that the silicon dissolution rate reached a maximum in the first 5 min., and then maintained constant. Silicon dissolution rate is also increases proportionally with increasing the particle size of sodium fluo-silicate, intensity of mixing and the Na2SiF6/Al ratio. The analysis of the experimental results suggested that the solute silicon atoms diffuse across a liquid boundary layer that was formed between solid phase (i.e. Na2SiF6 crystals) and liquid phases. Also the silicon dissolution rate is controlled by diffusion which has an activation energy ranged between 2.824 and 1.902 kcal/mole.

4:20 PM
Kinetics of Molten Chloride Salt Sorption into Zeolite-4A: *Michael Forrest Simpson*[1]; Sara Marie Lance[1]; Gregory Lee Moore[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83406 USA

A critical step in the EBR-II spent fuel treatment process involves blending fission-product loaded chloride salt with anhydrous zeolite-4A. The salt-loaded zeolite is a precursor for the final ceramic wasteform, which is comprised of glass-bonded sodalite. The blending is achieved by heating granular salt and zeolite to 500C in a rotating v-mixer for no more than 24 hours. The salt is ultimately occluded inside of the zeolite's alpha cages due to this process. Numerous salt sorption kinetics experiments using a laboratory-scale v-mixer have been performed in an effort to understand the factors that contribute to the rate of salt occlusion. A kinetic model for salt sorption in the zeolite has also been formulated with kinetic parameters estimated based on the experimental results.

4:50 PM
The Microreversibility of the System CuFeS2/Electrolyte: *Z. D. Stankovic*[1]; M. Rajcic-Vujasinovic[1]; Z. Stevic[1]; V. Fajnisevic[1]; [1]University of Belgrade, Techl. Fac. Bor, 19210 Bor Yugoslavia

In this paper results of the investigation of the microreversibility of the system CuFeS2/electrolyte are presented. The galvanostatic single pulse method has been used for investigation. Namely, the current pulses of the different amplitude and constant duration (40s) have been applied to electrode system composed of CuFeS2/electrolyte. The potential-time response in the form of the rising and decay curves were recorded. From the decay curves the conclusions of the reversibility of the system CuFeS2/electrolyte were evaluated.

5:20 PM
Molten Salt Separation from Uranium during the Processing of Spent Nuclear Fuel: *Brian R. Westphal*[1]; [1]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403-2528 USA

As part of the electrometallurgical treatment project at Argonne National Laboratory, a vacuum distillation process is being employed for the recovery of uranium following an electrorefining process. Distillation of a molten salt electrolyte, primarily consisting of a eutectic mixture of lithium and potassium chlorides with minor amounts of fission product chlorides, from uranium is achieved by a batch operation called cathode processing. The degree of molten salt separation from uranium at the cathode processor is termed distillation efficiency. This paper discusses the efficiency of the cathode processor via the use of representative isotopic tracers of the fission product chlorides. The relationship between distillation efficiencies, chloride vapor pressures, and relative concentrations is also discussed and applied to other chloride species of the system.

Alumina and Bauxite: Bayer Residue Handling

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

Monday PM	Room: 609
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Steven P. Rosenberg, Worsley Alumina Pty Ltd., Proc. Chem. Grp., PO Box 344, Collie, WA 6225 Australia; Jacques Mordini, Aluminium Pechiney, Direction Industrielle, Boite Postale 54, Gardanne Cedex France

2:00 PM
Behavior of Red Mud Agglomerated with Various Flocculants under Shear Conditions: *Michel Gagnon*[1]; Guy Simard[1]; Mélanie Normandin[1]; Guy Péloquin[2]; [1]Université du Québec à Chicoutimi, Appl. Scis., 555 boul Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, Limitée, Jonquière, Québec G7S 4K8 Canada

In the Bayer process, flocculating agents play an important role in the efficiency of the gravity settlers. They are added to the red mud slurry in the feed pipe and in the feed well. The flocculation process is certainly affected by the changing turbulent flow conditions existing in the pipe and at some point in the feed well. To gain a better understanding of the influence of flocculant properties and structure on the shear-flocculation occurring at these levels of the process, the behaviors of three commercially available flocculating agents were studied under shear conditions using concentric rotating cylinder (Couette system). The data obtained are expressed in terms of settling velocity, residual turbidity, and floc size distributions.

2:25 PM
Rheology Aids for Effective Mud Disposal: *Stephen John Adkins*[1]; Dana Therese Smith[2]; [1]Ciba Specialty Chemicals, Extractive Industries, PO Box 38, Low Moor, Bradford, W. Yorkshire BD12 0JZ England; [2]Alcoa World Alumina Atlantic, Point Comfort Operations, PO Box 101, Point Comfort, Texas 77978 USA

The process of red mud slurry stacking is widely used within the alumina industry. The physical and process variables which have a direct influence on this application, including solids content, particle size distribution and physical shear, have been previously investigated and documented. In many instances the physical properties of a final thickener underflow will meet the requirements for effective mud stacking. However, a significant proportion of the yield stress of the thickener underflow arises from the network structure imparted to it by the polymer added during the flocculation and thickening process. The necessity to transport the flocculated mud significant distances to a disposal area can result is a dramatic reduction in its rheological properties, caused by a break down of the network structure, and thereby reducing its ability to stack effectively. The physical and operational variables, together with operational constraints present within an alumina process, mean that it is difficult to maintain control of the physical properties of red mud for optimal disposal. The introduction of a rheology aid to the final mud could negate the impact of these variables, allowing optimum stacking performance to be maintained. This paper demonstrates the effect of such rheology aids in both laboratory and plant situations.

2:50 PM
Revised Application of Synthetic Flocculants in Red Mud Separation: *John Patrick Murray*[1]; [1]Alpart, Techl. Serv., PO Box 529, Arabi, LA 70032-0529 USA

Alumina Partners of Jamaica (Alpart) has used synthetic flocculants in Red Mud Separation since the late 1970s. These polymers were typically supplied to the vessels at low concentrations (0.05 to 0.10vol.%) following popular technical guidelines. During the late 1990s, Alpart was challenged to reduce flocculant consumption. An audit indicated that flocculant activity loss due to excessive degradation in the distribution piping was a major problem. Based on field experimentation, activity loss was eliminated at concentrations in the region of 0.4 to.5vol.%. Furthermore, concentrations on the order of 1.5 to 1.6 vol.% improved flocculant performance on the settlers (thickners). Since late 2000, Operations have been modified to reflect these findings, resulting in: 1) Flocculant usage reduced in excess of 30%, 2) Increased stability of the settlers and washers, 3) Reduced soda losses.

3:15 PM Break

3:35 PM Cancelled
Implementation of Turbidness Automatic Control in Thickener and Washer Tanks at C.V.G. Bauxilum: *Jesus Noya*

4:00 PM
Bauxaline: A New Product for Various Applications of Bayer Process Red Muds: *Valerie Martinent-Catalot*[1]; Jean-Michel Lamérant[1]; Gérard Tilmant[1]; [1]Aluminium Pechiney, Alumina Tech., BP 54, Gardanne 13541 France

Gardanne Pechiney plant produces 650000 tpy of alumina along with about 320000 tpy of red muds presently dumped into a deep submarine hole. For many years important R&D programs had been conducted to find possible ways of valorization of red muds, named as per december 2000 Bauxaline for commercial applications. Two main approaches are presently in progress: one consisting in thickening the muds so as to get a large amount for applications as for example dump rehabilitation, artificial reefs, filler for injection; one taking advantage of coloring properties, this application needs a more sophisticated preparation. The paper describes two particular uses of Bauxaline: arsenic fixation on co-disposal projects and coloring of various materials.

4:25 PM
CVG-Bauxilum Red Mud Neutralization: *Rodrigo R. Carneiro*[1]; Robert E. Keane[1]; Ricardo A. Galarraga[2]; Giao Nguyen[3]; [1]KD Engineering Company, 7701 N. Business Park Dr., Tucson, AZ 85748 USA; [2]CVG-Bauxilum, Ave. Fuerzas Armadas, Zona Industrial de Matanzas, Ciudad Guayana, Venezuela; [3]Atomaer Pty, Ltd., 14 Bell St., Canning Vale 6155 Western Australia

CVG-Bauxilum has been working toward the best alternative to neutralize and dispose the red mud and liquor generated from the alumina Bayer process. This has been done to eliminate any potential environmental impact on the near by Orinoco river. In June 2001, CVG-Bauxilum and ATOMAER-KD Engineering Co., Inc., conducted an on site pilot red mud neutralization study using carbon dioxide and the ATOMAER gas shearing technology. The pilot test program successfully reduced the red mud pulp pH from 12 to 7.60. The metallurgical results indicated that the process employed by CVG-Bauxilum and ATOMAER-KD Engineering Co., Inc., is feasible to neutralize the residue from the Bayer process.

Aluminum Reduction Technology: Environmental

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

Monday PM — Room: 6B
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Nancy J. Holt, Hydro Aluminium Metal Products, Tech. Ctr. Årdal, Øvre Årdal N-6882 Norway

2:00 PM
Can the Operator Take the Heat?: *Nancy J. Holt*[1]; Eldbjørg Saksvikrønning[2]; [1]Hydro Aluminium Metal Products, Tech. Ctr. Årdal, Øvre Årdal N-6882 Norway; [2]Hydro Aluminium Metal Products, Hydro Aluminium, Årdal Smelter, Øvre Årdal N-6882 Norway

All workers exposed to a hot and sometimes also humid environment may run the risk of heat stress and especially so if the work is strenuous. Work in the aluminium industry still includes several operations where the workers are exposed to potentially severe heat stress. The most widely accepted method used to evaluate heat stress is the Wet Bulb Globe Temperature (WBGT) index. The last couple of years Hydro Aluminium has gathered information on several work routines where the operator is exposed to heat stress. Although focus has been on potroom operations at several locations, measurements have also been done in the cast house and anode paste plant. This survey has been performed not only to find out if work routines are within international standards on recommended maximum heat stress levels. Another aspect of the measuring campaigns has been to gather information from different smelters with different technological solutions, and then compare these to see if there are possibilities for improvements that can reduce heat stress. Further, when e.g. a current increase section or new routines in anode change has been introduced, some of these have been evaluated with regards to heat stress. Heat stress evaluation is usually a natural part of exposure risk assessments. The information and experience with heat stress measurements have contributed significantly to a revised heat stress evaluation procedure for smelters in Scandinavia.

2:20 PM
Latest Developments on Dry & Wet Scrubbers Dedicated to Treatment of High Amperage Pots: *Andrew Haberl*[1]; Jean-François Langle[2]; [1]Procedair Industries, Inc., Techl. Div., 625 President Kennedy, Montreal, Quebec H3A, 1K2 Canada; [2]Procedair SA, Sales Dept., 25 Blvd. de la Paix, Saint Germain en Laye, Yvelines 78100 France

Major electrolysis technology providers offer today the capability for building large capacity primary aluminium smelters based upon a limited number of very high amperage pots. These smelters feature several particular parameters which are to be considered in the design of relevant modern Dry-Scrubbing Systems (DSS), such as i) requested increase of efficiency on HF, F particulate and total dust removal; ii) limited available area for DSS installation due to restricted distance between potrooms; iii) intensive magnetic field. Depending upon local regulations, wet scrubbers (WS) for SO2 removal together with optional waste treatment may be installed downstream above DSS. The aim of the present paper is to highlight the developments which have been recently implemented on DSS and WS in order to achieve the latest requirements from both environmental authorities and electrolysis plant operators.

2:40 PM
Retrofit of Dry Scrubbers Pays Back: *Geir Wedde*[1]; [1]ALSTOM Power Norway, Boilers & Environ., Ole Deviksvei 10, Oslo 0666 Norway

Since early 1970's the dry scrubbing process became the state of the art emission control and fluoride recovery technology for treating fumes from aluminium reduction lines. Many (most) of these scrubbers are still in operation 20-30 years later. Technology improvements and cost reduction programmes have ensured more efficient dry scrubbers. Retrofitting potlines with the latest dry scrubbing technology give healthy investment returns from reduced operating costs and other important benefits such as operators improved health and safety, reduced emissions and improved availability.

3:00 PM
Analytical and Experimental Study on Fluoride Evolution: Abdelhamid Meghlaoui[1]; Yussuf Ali Mohamed Al Farsi[1]; *Najeeba Hassan Al Jabri*[1]; [1]Dubai Aluminium Co., Ltd (Dubal), Tech. Dvlp. Dept., PO Box 3627, Jebel Ali, Dubai UAE

Aluminium fluoride is consumed within the electrolysis cell by hydrolysis reactions, reactions with alumina impurities and cathode reactions. It is difficult to calculate theoretically the AlF3 losses using physical and chemical equations because of the effect of other factors. Haupin considers the HF gaseous and particulate emissions as major and proposed a model to estimate them. The model is more oriented towards the environmental aspect than controlling the AlF3 additions. Entner proposed a multivariate regression model based on the AlF3 mass balance, which estimates the overall AlF3 losses, but doesn't specify their repartition. In this study, we implemented an AlF3 control strategy based on Entner model and fuzzy logic. Then calibrated Haupin model, using correlation equations from an experimental trial, and used it as soft sensor. A long-term run shows that AlF3 additions based on fuzzy-Entner control strategy is balanced with Haupin total emissions and reactions due to alumina impurities.

3:20 PM Break

3:30 PM
The Influence of Dry Scrubber Alumina and Humidity on COS Smelter Emissions: *S. J. Hay*[1]; M. M. Hyland[1]; J. B. Metson[2]; O-M. Bade[3]; [1]The University of Auckland, Dept. of Cheml. & Matls. Eng., PB 92019, Auckland New Zealand; [2]The University of Auckland, Dept. of Chem., PB 92019, Auckland New Zealand; [3]Alstom Power (Environment), Oslo Norway

Carbonyl sulfide (COS) emissions from aluminium smelting are modest compared to emissions of HF or SO2. However, the potential environmental effects of COS require that COS produced during aluminium smelting be considered. These studies aim to determine the activity of smelter-grade alumina used in dry scrubbing, in reaction with COS. In addition, the effects of humidity on this system have been considered. The major source of COS is from the 2-3wt% sulfur impurity present in anodes consumed during the production of aluminium. While COS is the main sulfur gas to be produced at the anode, it is largely oxidised to SO2 on contact with air in the headspace of the cell and in the ducting. Several groups have monitored COS entering and leaving the dry scrubber, but there are inconsistencies in their conclusions from these measurements, suggesting that local conditions in the dry scrubber affect the concentration of COS. Thermodynamic calculations were completed to predict the stable phases at equilibrium when starting with COS in the presence and absence of alumina and in different gas compositions. Results were compared with laboratory studies, performed by passing COS containing gases through a fluidised bed of alumina and measuring the outlet gas composition. Alumina type was varied, in addition to inlet gas composition, which allowed the effect of humidity to be considered. After reaction, alumina from the fluidised bed was studied using a variety of surface sensitive methods. Experimentally COS was found to be stable in the apparatus when in isolation, although the thermodynamic calculations predicted otherwise, pointing to the importance of kinetic factors. The presence of smelter-grade primary alumina resulted in partial hydrolysis of COS, producing H2S. A sulfur mass balance indicated some adsorption of COS on the alumina surface, which appeared to lead to saturation of the alumina. Secondary alumina from a typical dry scrubber was found to be unreactive towards COS, suggesting that the presence of F and S reduced the activity of the alumina surface. The presence of humidity altered the extent of conversion of COS to H2S and prevented saturation of the alumina surface for the duration of a typical experiment.

3:50 PM

Reduction of CF4 Emissions at the Smelter in Essen: *Martin Iffert*[1]; Regina Ganther[1]; Juergen Opgen-Rhein[1]; [1]Aluminium Essen GmbH, Electrolysis, Aluminiumallee 1, Essen D-45356 Germany

PFC emissions are harmful to the environment regarding their global warming potential and therefore it is a challenge to the aluminium industry to reduce the evolution of these gases. Based on this background we carried out two measurement campaigns of CF4 emissions from our smelter in Essen during spring 2001. Within the first campaign the current level of emissions was fixed and different strategies of automatic anode effect treatment were analysed and compared. We paid special attention to the success rate and the CF4 emission levels of the anode effect quenching strategies. Afterwards the most successful method with the lowest emissions was implemented in all process computers. In the second campaign the improvements were verified. This paper shows the correlation between anode effect over voltage and CF4 emissions that we have found in our campaigns. As well we point out our suggestions and trials about anode effect quenching strategies.

4:10 PM

Interaction Between Aluminium Process and Powder Technology: *Morten Karlsen*[1]; Are Dyroey[2]; [1]Hydro Aluminium, Tech. Ctr. Årdal, PO Box 303, Øvre Årdal 6882 Norway; [2]Tel-Tek, POSTEC, Kjoelnes Ring, Porsgrunn 3918 Norway

Historically, the development of the aluminium industry has been focused on the improvement of the electrolysis process. However, the largest volumes of handled materials are alumina and petrol coke, which are bulk solids. The interfaces between the pots and the fume treatment facilities often present challenges to which the application of powder technology may provide feasible and good solutions. The dusting problems of the open Søderberg pots represent a severe limitation with respect to fulfilling future environmental requirements. Based on correlations between dust and alumina fines content, promising test rig results initiated the development of a continuous de-dusting unit. This unit separates the alumina fines and reroutes this fraction into the feed of the dry scrubber section serving a prebake pot line. Increasingly strict environmental demands inevitably cause old fume treatment facilities to be replaced or retrofitted. If a retrofit is possible the harvest time of the initial investment may be prolonged for 5 to 10 years, giving the opportunity to plan and design a possible new expansion. By modifying the alumina injection points into the gas reactor, the cleaning efficiency of the dry scrubber can be improved. Implementation of the new injection technique has reduced the outgoing concentration by 72%.

4:30 PM

New Aerated Distribution (ADS) and Anti Segregation (ASS) Systems: *Bernt Nagell*[1]; Gisle G. Enstad[2]; Peter Hilgraf[3]; Morten Karlsen[4]; Are Dyroey[2]; [1]Norsk Hydro, Hydro Karmoey Aluminium, Haavik 4265 Norway; [2]Tel-Tek, POSTEC, Kjoelnes Ring, Porsgrunn 3918 Norway; [3]BMH, Claudius Peters Buxtehude, Schansenstrasse 40, Buxtehude 21614 Germany; [4]Hydro Aluminium, Tech. Ctr. Årdal, PO Box 303, Øvre Årdal 6882 Norway

Two tons of alumina is required to produce one ton of Aluminium. The logistics of alumina therefore represents an important part of the Aluminium production, including unloading of ships, internal transport and storage; fume treatment and finally distribution into the electrolysis pots. Over the last 20 years several automated distribution systems utilizing pneumatic conveying have been developed. These systems are closed, resulting in less dusting. However, the erosive properties of alumina are better countered by the use of low velocity conveying. Hence the development of an air-slide based system was implemented. Also low velocity and low-pressure drop reduces the ability of secondary alumina to form scales. We present a new system utilizing low conveying velocity air-slides with adequate capacities. The Aerated Distribution System (ADS) has been installed and tested in regular production and shows almost eliminated need for maintenance. The system also includes a separation unit to remove the scales, eliminating scaling problems. The difference in maintenance frequency, the lifetime cost and the "up-time" strongly favours low velocity systems. Another advantage of distribution systems is that several pots fed by these systems, more easily may be treated as one unit instead of several individual pots. When introducing automated distribution systems, improved homogeneity of the ingoing alumina is usually required to ensure stable logistic flow. A system to reduce segregation when filling silos and A-frames has been developed and operated in production, demonstrating a homogenizing factor of 1.47 on the alumina put through the system.

Automotive Alloys 2002 - I

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

Monday PM — Room: 611
February 18, 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

Assuring Continued Recyclability of Automotive Aluminum Alloys: Grouping of Wrought Alloys by Color Sorting and Chemical Composition-Based Sorting: *Adam J. Gesing*[1]; Tim D. Good[1]; F. Scott Costello[1]; Larry Berry[1]; Richard B. Wolanski[1]; Paul B. Schultz[2]; [1]Huron Valley Steel Corporation, R&D, 41000 Huron River Dr., Belleville, MI 48111 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

Although automotive aluminum is currently completely recycled, recycling technologies need to be improved to assure continued, high-value recyclability of all present and future automotive alloys–particularly wrought alloys, as the use of these alloys increases in the aluminum intensive vehicle. The Automotive Aluminum Alliance has designed a program to demonstrate the ability of new scrap sorting technologies to deliver high-value recyclability of aluminum scrap from end-of-life vehicles. Huron Valley Steel Corporation, the developer of these sorting technologies, is participating in this demonstration program. In this paper, we compare the alloy groupings for wrought scrap obtained using two techniques: color sorting of particles tinted by etching in an NaOH solution, and chemical composition-based alloy sorting.

2:30 PM

Filler Alloy Selection for Aluminum Welding: *Tony Anderson*[1]; [1]AlcoTec Wire Corporation, 2750 Aero Park Dr., Traverce City, MI 49686-9263 USA

When considering the welding of aluminum alloys, and the development of welding procedures one of the main considerations must be that of filler alloy selection. Typically there are a variety of filler alloys available which may be used to weld any given base alloy combination. However there are a number of variables associated with selection of the most suitable filler alloy to be used. These variables are largely based on product application and are as follows: Strength of the weld, Ductility, Corrosion Resistance, Sustained Temperature Service, Color Match After Anodizing, and Post Weld Heat Treatment. The understanding of these variables is a significant aspect in ensuring the correct design and development of a successful welding procedure.

2:55 PM

Effect of Quasi-Static Prestrain and Eddy Currents on Limit Strains in Electromagnetic Pulse Assisted Stamping of Two Aluminum Alloys: *Vincent Joseph Vohnout*[1]; Glenn S. Daehn[1]; [1]The Ohio State University, Matl. Sci. & Eng., 2041 College Rd., Watts Hall 177, Columbus, OH 43210 USA

The integration of electromagnetic pulse energy with conventional stamping shows promise as a method capable of overcoming the well known forming limit problems of stamped aluminum parts. The fundamental viability of this combined, hybrid process was demonstrated by conducting a series of experiments with 0.8 mm thick coupons of two aluminum alloys, 6111-T4 and 5754-0. The coupons were first quasi-statically strained to various levels terminating a few percent below the conventional forming limit. Each coupons was subsequently expanded rapidly, with an electromagnetic pulse, to failure. Records of the failure strains generated by the combined processes show a consistent increase in limit strains for this hybrid process when compared to either the conventional or high speed process applied singly. The effects of material type and expansion velocity on the extended formability limit was investigated as was the effect of eddy current joule heating of the coupon.

3:20 PM

The Influence of Surface Roughness on the Deforming Friction Behavior of Al-Mg Alloys: *Mark R. Stoudt*[1]; [1]NIST, Matls. Perfor. Grp., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

The inhomogeneous plastic flow exhibited by numerous aluminum alloys presents significant challenges to their use in automotive applications. The size and distribution of surface asperities influence the friction between mating die surfaces resulting in accelerated die wear and progressively reduced shape accuracy in the metal stampings. Finite element predictions of friction are often inconsistent with that observed in real measurements. This suggests additional studies are required to improve the understanding of the fundamental relationships between surface roughness and friction under deforming loads. Friction measurements typically draw metal sheets over a mandrel making evaluation of the material behavior under the deforming loads difficult. A measurement technique was developed to determine the frictional coefficients and to enable detailed assessments of the material response to the deforming loads. Measurements were performed on thin Al-Mg alloy specimens with controlled levels of initial surface roughness under varying normal forces. The results will be presented and discussed.

3:45 PM Break

4:00 PM
Using the Method of an In-Situ Thermal Analysis Array in a Cast Section of an Engine Block Casting to Diagnose the Cause of Shrinkage Porosity: *M. Djurdjevic*[1]; R. I. Mackay[1]; J. H. Sokolowski[1]; D. Cusinato[2]; A. Mueting[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, Windsor Aluminum Plant, 4600 G. N. Booth Dr., Windsor, Ontario N9C 4G8 Canada

Hot spots are undesirable occurrences during the cast solidification process since they lead to a higher level of porosity in the cast structure. Many techniques have been developed to identify the propensity of hot spots and they include using thermocouple arrays in cast moulds. This work was done in order to determine the specific solidification kinetics, which occurred in the cast section of interest that yielded the observed shrinkage porosity. The observation of, what will be described in this work as, a latent heat generated hot spot phenomenon was made. This phenomenon resulted in cooling in rates for the primary a-Al dendritic phases and for the Al-Si eutectic to change from one cast section to another. This was the result of the accumulative heat extraction by the mould wall and the partially solidified metal and the latent heat release of the solidifying Si crystals.

4:25 PM
Formability Performance of 5XXX Series Aluminum Alloys Produced with Twin-Roll Casting Technique: *Murat Dundar*[1]; Abdullah Soner Akkurt[1]; Chris Romanowski[2]; [1]ASSAN Aluminum, E5 Karayolu 32. Km., Tuzla, Istanbul 81700 Turkey; [2]FATA-Hunter, 6147 RiverCrest Dr., Riverside, CA 92507 USA

The ultimate goal of the present study was to produce twin roll cast 5XXX series alloys and related processes suitable for products involving severe press shop operations, with similar, or preferably better, properties than the corresponding DC-cast material. In connection with the previous paper of the authors that has involved microstructural and mechanical characterization of twin roll cast AA5052, AA5754 and AA5182 alloys aiming to automotive applications, formability performance of these materials were investigated by constructing their Forming Limit Diagrams (FLD) in the present study. Tendency of the materials to the localized necking and its plastic anisotropy were correlated with the FLD results through the material properties of n and r values. These results were compared with those of similar alloys produced by the DC casting route. Influence of microstructural features, that were either developed during processing route or intrinsically existing from casting, on failure limits of the alloys were elucidated by utilizing different characterization techniques. Contribution of grain structure and intermetallic particles on surface cracking and strain inhomogenities that could be developed by centerline segregation during biaxial loading were investigated.

4:50 PM
Effect of Second Phase Particles on Bendability of Al-Mg-Si Alloy Sheet: *Hidetoshi Uchida*[1]; Mineo Asano[1]; Hideo Yoshida[1]; [1]Sumitomo Light Metal Industries, Ltd., R&D Ctr., 3-1-12 Chitose, Minato-ku, Nagoya, Aichi 455-8670 Japan

Recently Al-Mg-Si alloy sheets are applied to automotive closure panel for good paint bake response and better recyclability. Improvement of bendability on Al-Mg-Si alloy sheet requires for hemming performance. In this study, effect of second phase particles on bendability of Al-Mg-Si alloy was investigated. Solution heat treatment time and quenching rate were changed to obtain several conditions of second phase particles. In regard to quench rate, fast rate is better for bendability because less the second phase particles on grain boundary. In regard to the solution heat time, level of crack after bending was becoming worse in less than 90 s and more than 300 s the level was becoming better. The reason of the phenomena was considered that combination of formation of microband by matrix deformation and size of the second phase particles.

5:10 PM
Effect of Casting Defects on Mechanical Properties of Magnesium Alloy Castings: *A. M. Gokhale*[1]; G. R. Patel[1]; [1]Georgia Institute of Technology, Sch. of Matl. Sci. & Eng., Atlanta, GA 30332-0245 USA

Die cast AM60 and AZ91D magnesium alloys show a large variation in tensile strength and ductility within a group of castings cast under the same average process conditions and chemistry. It is, therefore, important to understand the factors that affect such variability in the mechanical properties. In an earlier contribution, we presented quantitative microstructural and fractographic data on AM60 alloy castings and their correlations with the variability in the mechanical properties. In this contribution, we present our quantitative data on variability in mechanical properties of AZ91D magnesium alloy castings to establish quantitative correlations with microstructural and fractographic parameters. For this purpose, the fracture surfaces of the tensile test specimens and the bulk microstructures have been quantitatively characterized. In these cast microstructures, porosity (shrinkage and gas and/or air) is a dominant type of defect. A method to quantify porosity on the fracture surface and in the bulk microstructure has been developed and quantitative relationships between porosity on the fracture surface and the tensile ductility as well as UTS are established. A comparison is made between these data and the earlier data obtained on AM60 alloy castings.

Carbon Technology: Anode Raw Materials and Anode Property Testing

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

Monday PM	Room: 602-603
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Robert H. Wombles, Koppers, Pittsburgh, PA 15238 USA; J. Tom Baron, Koppers, Pittsburgh, PA 15238 USA

2:00 PM
Production of Low-PAH-Pitch for Use in Söderberg Smelters: *Winfried L. Boenigk*[1]; Gord H. Gilmet[2]; Dirk Schnitzler[1]; Jens Stiegert[1]; Mike Sutton[2]; [1]RUETGERS VFT AG, Kekuléstr. 30, Castrop-Rauxel 44579 Germany; [2]VfT, Inc., 725 Strathearne Ave. N., Hamilton, Ontario L8H 5L3 Canada

The legal understanding of PAHs differs from its scientific definition. Therefore the composition of low-PAH-pitches may vary from country to country. Strategies to produce low-PAH-pitches using coal tar and petroleum feedstocks will be discussed. The quality characteristics of low-PAH-pitch depend on the applied production strategy. Advantages and disadvantages of several production options will be pointed out. Industrial use of low-PAH-pitches for Söederberg applications has started.

2:25 PM Cancelled
Average Molecular Weight Measurement of Binder Pitch by Thermogravimetric Analysis(TGA) Method: *Kwang Eui Yoon*

2:50 PM
Alcan Characterization of Pitch Performance for Pitch Binder Evaluation and Process Changes in an Aluminum Smelter: *Amir A. Mirchi*[1]; Gaby Savard[1]; Jean-Pierre Tremblay[1]; Michel Simard[2]; [1]Alcan International, Ltd., Reduction Tech., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Alcan Primary Metal Group, Info. Tech., 1955 Mellon Blvd., PO Box 1500/1640, Jonquiere, Quebec G7S 4L2 Canada

Coal tar pitch is the main raw material used as a binder for the anode fabrication. The characteristics of the pitch binder affected the paste plant operation and anode performance during electrolysis. Over the last two decades, the coke oven design and technology have been improved to minimize the environmental emission and to increase the coke oven productivity. The properties of the coal tars used as raw materials for pitch production have been affected by this change. The pitch producer also uses more and more different tar sources. In the same period, the aluminum industry increased its requirement in terms of anode quality to improve the cell control, productivity and to increase the cell amperage. The impact of the pitch property evolution on the paste and electrode performance has been studied. A new experimental device has been developed by Alcan to measure the pitch wetting capability for pitch binder evaluation.

3:15 PM
The Characterization of Pre-Baked Carbon Anodes using X-Ray Computed Tomography: *Angelique N. Adams*[1]; [1]The Pennsylvania State University, 401 Academic Activities Bldg., University Park, PA 16802 USA

Carbon anodes represent about 25% of the production cost of aluminum, therefore quality control is important to the smelting operation. Typical quality control methods involve testing cores from production and scrap anodes. One parameter of particular interest is density. The aim of this research is to determine the density profile of carbon anodes through non-destructive analysis. X-ray computed tomography (CT) is a technique that can determine the density of materials and represent the information visually at high resolution. A bench-scale green anode was scanned using X-ray CT, over the length of the anode. The anode was baked in an industrial laboratory and re-scanned at the same registration. The density data were converted into a series of images. The density difference between the green and baked sample is apparent. The presence of additional features such as butts and localized areas of inadequate amounts of binder were also observed.

3:40 PM Break

3:50 PM
New Method for Measuring Thermal Conductivity of Coarse Grain Carbon Materials: *Peter S. Gaal*[1]; Heng Wang[1]; Silviu P. Apostolescu[1]; [1]Anter Corporation, 1700 Universal Rd., Pittsburgh, PA 15235 USA

Thermal conductivity and thermal diffusivity are two very important physical properties of carbon materials that can substantially impact upon the life cycle of anodes. The two properties are interrelated through density and specific heat capacity. The role of thermal conductivity in the performance of anodes is well known, while the role of thermal diffusivity, a dynamic heat transport parameter, is largely unknown. This is partly due to the complexity of measuring it, and the historically very small samples used in laser flash measurements, which in case of coarse grain materials, may not always be representative. This paper describes work aimed at extending the inherently fast flash technique to measure these thermal properties of carbon anode materials, by using larger samples (up to 30 mm diameter x 8 mm thick). Data is presented comparing results obtained with using the new larger size samples and conventional small samples. The results are also compared to those obtained using conventional steady-state heat flow methods.

4:15 PM
The Use of Image Analysis for the Optimization of Pre-Baked Anode Formulation: *Angelique N. Adams*[1]; [1]The Pennsylvania State University, 401 Academic Activities Bldg., University Park, PA 16802 USA

Carbon anodes represent about 25% of the production cost of aluminum. The direct cost is that of the consumable raw materials. The indirect cost is electrode resistance, which contributes to energy consumption. It has been well established that anode life and electrical resistivity are optimized when the binder quantity is optimized. The aim of this research is to determine if semi-automated image analysis can be used to determine the appropriate binder quantity for a given coke particle size distribution. Samples of anode mix were taken from a bench-scale anode production process, and characterized using a microscope with semi-automated image analysis. The thickness of the pitch film around coarse coke particles was determined. The results show good agreement between the quantity of pitch measured using image analysis and the amount added in the production process.

4:40 PM
Air Reactivity Factors Affecting Anode Performance: Frank R. Cannova[1]; *Bernard C. Vitchus*[1]; [1]BP, 300 Oceangate, Long Beach, CA 90802 USA

Anode reactivity is used to monitor anode quality and explain anode performance. Data will be presented which shows how anode air reactivity varies in anodes made from the same calcined coke but with different reactivities due to sodium content and air permeability properties. These reactivity results were obtained on cores taken from the center of commercial anodes. However, the air and CO2 reactivity of the anode occurs at the anode surface. The reactivity of the anode surface can be different than a core taken from the center of the anode. Air reactivity data is presented for surface anode and butt specimens. These data provide insight of how anode baking, effect of soft butts and cryolite from cells can affect the surface reactivity of anodes.

5:05 PM
Gas Reactivity Inside Industrial Anodes: *Marianne Aa. Engvoll*[1]; Harald A. Øye[1]; Morten Sørlie[2]; [1]Norwegian University of Science and Technology, Inst. of Chem., Trondheim N-7491 Norway; [2]Elkem Aluminum Research, PO Box 8040 Vaagsbygd, Kristiansand N-4675 Norway

Bath compounds, especially cryolite, sodium fluoride and cryolite, act as strong catalysts towards the air and CO2 gasification reactions. To prebaked anodes, considerable amounts of fluorides are introduced via the addition of butts. Dust accumulation on the anode top and penetration of NaAlF4 gas through the open porosity appear to be important contamination sources in Söderberg anodes. By use of electron microscopy, this work examines the extent of catalyzed gasification inside industrial prebaked and Söderberg anodes. The results are related to the gas permeabilities, contamination profiles and laboratory reactivity experiments.

Cast Shop Technology: Modeling II

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

Monday PM	Room: 6A
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Yogesh Sahai, Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; Bjørn Rune Henriksen, Elkem ASA, PO Box 8040 Vaagsbygd, Kristiansand 4675 Norway

2:00 PM Keynote
Mathematical Modeling in Aluminum Casting and Molten Metal Treatment: *James William Evans*[1]; W. Kinzy Jones[2]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Motorola, Adv. Products Tech. Ctr., 8000 W. Sunrise Blvd., Ft. Lauderdale, FL 33322 USA

The paper reviews the mathematical models that have been developed in recent years, at Berkeley and elsewhere, to simulate the DC and electromagnetic casting of aluminum, along with models for other cast shop operations such as melting or gas fluxing. Wherever possible the predictions of the mathematical models are compared with experimental measurements, either on actual plant operations or on physical models.

2:30 PM
Inverse Analysis of Space and Time Variations in the Boundary Heat Flux during Water Film Cooling: *Einar Arne Sørheim*[1]; Dag Mortensen[1]; Steinar Benum[2]; Christian Stette[2]; [1]Institute for Energy Technology, Matls. & Corr. Tech., PO Box 40, Kjeller N-2027 Norway; [2]Hydro Aluminium R&D Materials Technology, PO Box 219, Sunndalsøra N-6601 Norway

An inverse problem of recovering the heat flux, in a situation where an aluminium block is exposed to a sudden spray of water, is presented. The block is isolated on all sides but one; the water cooled as cast surface. The objective is to gain insight into the water film cooling mechanisms. Several thermocouple sensors are located in the interior of the block and their time history is recorded for several different water qualities and spray nozzle characteristics. A solution to the inverse problem is developed, by using a finite element model and the adjoint functions method combined with a conjugate gradient algorithm. Several different regularization strategies are applied and a connection between the resolution of the recovered heat flux and regularization, sensor locations, and number of sensors is discussed.

2:55 PM
Modelling of Grain Refinement in Directional Solidification: *A. L. Greer*[1]; T. E. Quested[1]; J. E. Spalding[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The modelling of grain refinement due to Bunn et al. [Light Metals 1998, pp. 963-968] was based on the Maxwell-Hellawell assumption that the melt remains spatially isothermal. A new model is presented for grain refinement in directional solidification where this assumption cannot apply. A simple heat balance is used evaluate the undercooling in the quasi-isothermal solidifying zone. This undercooling determines the grain size through the free-growth criterion in which the potency of an inoculant particle is assumed to be related only to its size. The predictions of the new model are compared with those from the isothermal-melt model and with measured grain sizes in TP-1 and Bridgman samples. Good predictions are obtained for the dependence of grain size on refiner addition level, solute content and cooling rate. The influence of temperature gradient on grain-refiner effectiveness is considered.

3:20 PM Break

3:35 PM
Modelling of Air Gap Development and Associated Surface Macrosegregation in DC Casting of Aluminium Sheet Ingots: *Mohammed M'Hamdi*[1]; Dag Mortensen[2]; Hallvard Fjær[2]; Asbjørn Mo[1]; [1]SINTEF Materials Technology, PB 124 Blindern, N-0314 Oslo Norway; [2]Institute for Energy Technology, PB 40, N-2007 Kjeller Norway

The interaction between thermally induced deformation and heat transfer in DC casting of aluminium often results in the formation of an air gap between the mould and the ingot. The metallostatic head can then force interdendritic liquid through the mushy zone, past the original casting surface, and into the air gap. Such exudation of liquid leads to a solute rich surface layer. A two dimensional mathematical model that quantifies surface macrosegregation due to exudation and solidification shrinkage has been presented elsewhere. A critical parameter of this model is the position of the air gap, and a thermomechanical calculation is needed for an accurate prediction of the development of this gap. For this purpose, the heat transfer calculation being input to the surface segregation model is carried out with an advanced model in which the air gap development is also taken into account by a model addressing the thermally induced deformations in the ingot. Periodic variations in the surface macrosegregation along the casting direction have been reproduced by the models simulating the DC casting of an AA5182 sheet ingot. These variations reflect oscillations in measured mould temperatures that are related to the periodic solidification/melting of the surface shell.

4:00 PM
Thermo-Mechanical Modeling to Predict Shrinkage, Shape and Mold Openings for DC-Cast Rolling Ingots: *Werner E. Droste*[1]; Gerd-Ulrich Grün[1]; Wolfgang Schneider[1]; Jean-Marie Drezet[2]; [1]VAW Aluminium AG, R&D, Georg-von-Boeselager-Str. 25, Bonn, NRW D-53117 Germany; [2]Calcom SA, Parc Scientifique, PSE-EPFL, Lausanne CH-1015 Switzerland

The traditional way to calculate mold openings of DC-cast rolling ingots is based on measured values of shrinkages and interpolation methods. Difficulties arise, when there are no measured values close to the shape and casting speed desired, as extrapolation on this basis does not work. On the other hand todays thermo-mechanical simulation tools for DC-casting are based on physical models. After validation using a given ingot size and casting speed such a model should be capable to predict the correct shrinkage even far away from the validation point at quite different ingot shapes and casting speeds. Here, a 3-D thermo-mechanical model based on the general purpose software Abaqus, which takes into account relevant material behavior, was tested for its ability to predict the ingot shrinkage for large and small ingot sizes and different casting speeds after evaluating the model for a medium size ingot. Resulting ingot shapes are compared with measured data and the influence of ingot size, casting speed and cooling conditions on the accuracy are discussed.

4:25 PM
Microporosity and Other Mushy Zone Phenomena Associated with Hot Tearing: Mohammed M'Hamdi[1]; *Asbjørn Mo*[1]; [1]SINTEF Materials Technology, PB 124 Blindern, N-0314 Oslo Norway

A two-phase continuum model addressing the two main mechanisms associated with hot tearing formation during solidification and subsequent cooling, namely melt feeding of solidification shrinkage and thermally induced deformation, has recently been formulated for the aluminium direct chill (DC) casting process. This mathematical model is in the present paper extended to include also the formation of microporosity because this phenomenon is often considered as related to hot tearing. The modelling equations have been solved for simplified one-dimensional conditions having some relevance for the DC casting process. Modelling results reveal that thermally induced deformations strongly affect the porosity level, and correspondence is pointed out between modelled porosity levels and hot tearing susceptibility in casting trials presented elsewhere.

4:50 PM
The Effects of Water Ejection and Water Incursion on the Evolution of Thermal Field during the Start-Up Phase of the Dc Casting Process: *J. Sengupta*[1]; D. M. Maijer[1]; M. A. Wells[1]; S. L. Cockcroft[1]; A. Larouche[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [2]Alcan International Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquière, Québec G7S 4K8 Canada

The control of the thermal cooling conditions at the start-up phase of the Direct Chill (DC) casting process for aluminum ingots is difficult, and is critical from the standpoint of defect formation. Firstly, boiling water heat transfer governs the secondary cooling experienced by the ingot surfaces as they emerge from the mould. This results in varying rates of heat transfer from the ingot faces as the surface temperature of the ingot changes with time during the start-up phase. Moreover, if the ingot surface temperature at locations below the point of water impingement is high enough to promote film boiling, the water is ejected away from the surface. This can result in a sudden decrease in heat transfer and the formation of local hot spots. Also, the chill water may enter into the gap formed between the ingot base and the bottom block with the evolution of the butt curl. This water incursion alters the heat transfer from the base of the ingot, and in turn affects the surface temperature of the ingot faces. This paper outlines the development of a 2D thermal model using the FEM code ABAQUS™ to study the effects of secondary cooling, water ejection, and water incursion on the evolution of the thermal field in the ingot during the start-up phase. Model predictions have been compared to measurements made on an industrial DC casting machine for validation.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: Grain Boundary Segregation and Fracture in Steels

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

Monday PM	Room: 307-308
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Clyde Briant, Brown University, Div. of Eng., Providence, RI 02912 USA; Vasek Vitek, University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

2:00 PM Keynote
Embrittlement Phenomena: Where Have We Been, and Where Are We Going?: *Charles J. McMahon*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

During the past four decades, I have been blessed with a large number of excellent collaborators: graduate students, post docs, and visiting scientists. Here I want to review some highlights of their work in the following areas: Deformation and brittle fracture of iron, Embrittlement of alloy steels, Hydrogen embrittlement of steels, Surface and interface segregation, Ti-Al alloys, Dynamic embrittlement of steels, Cu-based alloys, and Ni-based alloys. There is still work to be done on the dynamic-embrittlement problem, and I will describe our plans for the next few years.

2:35 PM Invited

Industrial Experiences in Relation to Intergranular Cohesion of Steels: *Hiroo Ohtani*[1]; Masaaki Igarashi[1]; [1]Sumitomo Metal Industries, Ltd., Corp. Rsrch. Labs., 1-8 Fuso-cho, Amagasaki, Hyogo 660-0891 Japan

The demands for high strength low alloy (HSLA) steels are increasing from the viewpoint of social needs, especially environmental conservation. There are, however, various types of intergranular embrittlement encountered during processing, fabricating, and also in service. Some examples of decohesion of boundary related to microstructural characteristics and impurity segregation are presented. (1) Intergranular cracking of cast steel: Surface cracking along austenite (γ) grain boundary of continuous cast slab is mainly due to large γ grain strengthened by alloy-carbonitrides with precipitation free zone or with thin ferrite transformed along the boundary. Rapid cooling followed by re-heating to refine γ grain size, or proper selection of the chemistry to avoid eutectic region whose γ grain size is largest is applied. Impurity segregation enhancing the weakness of grain boundaries is typically observed, for example, in cast 9%Ni steel for LNG storage tank. (2) Grain boundary embrittlement on tempering: Quench-temper is a conventional heat treatment to obtain high strength and toughness. Low temperature embrittlement is believed due to continuous precipitation of cementite and re-distribution of impurities along prior γ grain boundaries. In contrast to martensite structure, low temperature bainite is free from this type of embrittlement, and also shows good resistance to delayed fracture. High temperature temper embrittlement can be essentially eliminated by controlling the chemistry, impurities, and heat treatment. It is worth to know the rapid cooling after tempering causes another problem; dimensional change of the plates after cold forming due to residual stress. The processes that are less sensitive to such kind of embrittlement are presented. (3) Embrittlement due to center segregation of continuous cast slab: Hydrogen induced cracking has been seen in line pipe for transportation of natural gas and crude oil, and in petrochemical plants. Cracking path is usually along the boundary between ferrite and low temperature transformation product such as martensite. In addition to reduction of inclusions and shape control of them, TMCP process has been successfully applied to obtain uniform bainitic structure. (4) Grain boundary cohesion in heat resistant steels: Long-term creep embrittlement and the cracking due to reheating for stress relieving of the weldment have not fully been understood yet in heat resistant steels. They are considered to attribute to decrease in grain boundary strength due to combination of precipitation and growth of carbides formed along the boundary, and the resultant enhancement of impurity segregation into reformed boundaries. Some evidences of improving these kinds of embrittlement by the alloy elements will be discussed in the presentation. (5) Progress in steel making and analyzing technology: The precise control of chemistry and reduction of impurities in steel making together with the progress in the analyses of trace elements and microstructure of the steels will be presented.

3:10 PM Invited

Segregation Intergranular Fracture and Limits to Formability in Re-Phosphorised Steels: *David Embury*[1]; Kevin Boyle[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

Re-phosphorised steels represent an important method of producing formable steels for automotive applications. Under some conditions they can exhibit cold work embrittlement and this represents the basis of this presentation. The study has employed TEM and EBSP methods to examine the degree of P segregation as a function of heat treatment and the character of grain boundaries. Tests have been performed over a range of temperatures and prestrains to determine the conditions for intergranular fracture. A new model of the fracture process has been developed and applied to forming operations such as creep drawing. The presentation will cover both the fundamental aspects of intergranular fracture and the application of the fracture model to the complex strain paths used in forming operations.

3:45 PM Invited

Effects of Impurity-Element Segregation on Fracture Toughness and Fatigue-Crack Propagation in a 2.25Cr1Mo Steel: *John Frederick Knott*[1]; *Aminul Islam*[1]; Paul Bowen[1]; [1]The University of Birmingham, Sch. of Eng., Elms Rd., Birmingham, Edgbaston B15 2TT UK

The paper describes a number of effects produced by the segregation of phosphorus to grain boundaries. Reversible temper embrittlement is exhibited in fracture toughness tests carried out at both 153K and 77K, due to phosphorus segregation. As-quenched and one-step embrittlement are affected by both the pre-segregation of phosphorus in austenite and by the development of carbides during tempering, concentrating phosphorus by the carbide rejection mechanism. Stress-relief cracking and crack growth at approx. 775K are associated with the stress-driven segregation of sulphur to the region of high triaxial stress ahead of a stressed crack tip. Intergranular facets are observed during fatigue crack propagation in air at room temperature. It is suggested that this is a result of hydrogen uptake from the water vapour present in the air, but that the entry of hydrogen is facilitated by the prior segregation of impurity elements to grain boundaries.

4:20 PM

Quench Embrittlement: A Recently Recognized Mechanism of Intergranular Embrittlement in As-Quenched High-Carbon Steels: *George Krauss*[1]; David K. Matlock[1]; [1]Colorado School of Mines, Adv. Steel Rsrch. Ctr., Golden, CO 80401 USA

A number of intergranular embrittlement phenomena, including tempered martensite, temper, and hydrogen embrittlements, have long been recognized in hardened medium carbon steels. However, gradual characterization of intergranular fracture of as-quenched steels containing 0.5 mass pct carbon has now established another form of embrittlement, termed by the authors, in view of the fact that tempering is not required, as quench embrittlement. The mechanism of this embrittlement has been related to phosphorous segregation and cementite formation at austenite grain boundaries during austenitizing or during quenching after austenitizing. The embrittlement develops even in very low P-containing steels, and persists through tempering temperatures into the first stage of tempering, below temperatures that cause tempered martensite embrittlement. This presentation will describe the mechanisms, thermodynamics, and effects of steel chemistry associated with quench embrittlement, and identify processing approaches which are used to minimize the effects of quench embrittlement.

4:40 PM

Anomalous Fracture Behavior of AISI 4340 Steels Treated at Different Solution Annealing Temperatures: *Jaroslav Pokluda*[1]; [1]Brno University of Technology, Inst. of Physl. Eng., Technicka 2, Brno CZ-616 69 Czech Republic

Values of initial fracture toughness KIc and impact absorbed energy of martensitic ultra-high strength low alloy steels AISI 4340 were found to be inversely dependent on the prior austenite grain size (the solution annealing temperature). The increase in KIc values with increasing grain size can be explained in terms of the crack driving force reduction caused by the intergranularly induced crack tip branching in coarse grained steel grades. The paper shows the results of both the analytical and numerical calculations of this shielding effect and the HRAES and X-ray studies of impurity concentration at grain boundaries. These results are used to elucidate quantitatively the anomalous behavior. Moreover, the occurrence of a dimple intergranular fracture mode in samples treated for the temper embrittlement is qualitatively explained in terms of the Mo, P and C diffusion kinetics.

5:00 PM

Boronizing on Impurity Controlled Steels: C. Bindal[2]; *A. H. Ucisik*[1]; [1]Sakarya University, Dept. of Metlgcl. & Matls. Eng., Adapazari-Sakarya Turkey; [2]Bogazici University, Dept. of Matls. Inst. of Biomed. Eng., Saribal S. 40, Ortakoy-Istanbul 80840 Turkey

Boronizing being one of the simplest methods used for boride type ceramic coating has extensively applied by many researchers on commercial alloys to increase surface hardness, to obtain a layer resisting wear and chemical attacks. In this study, boronizing process was applied on impurity controlled steels of which all embrittlement problems have been solved by Professor Charles J. McMahon, Jr. Alloys with and without Mn and Cr had 0.02% P. Mn and Cr levels were 0.5, 1.0 and none. Only one alloy had both 0.5% Mn and 0.5% Cr. Boronizing bath consisted of Borax, Boric Acid and Ferro-Silicon. All alloys were boronized at 940°C for between 0.5hrs and 10hrs. After boronizing, each group of alloy was examined by using classical metallographic techniques, X-ray diffraction, hardness measurements, fracture toughness measurements, Scanning Electron Microscopy and Corrosion tests. It was revealed that hardness level on the layer depends on alloy and boronizing time and coated material are divided into three parts; (i) layer having borides, (ii) the region where boron makes solid solution, (iii) matrix. In addition to iron borides mangenese and chromium borides formed on the surface depending on alloying elements. Mangenese is the only element increasing fracture toughness. In severe conditions all boride layers showed good corrosion resistance.

Computational Modeling of Materials, Minerals & Metals Processing: Monday 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

Monday PM	Room: 619-620
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Mississippi River at Third Ave., Minneapolis, MN 55414-2196 USA

Keynote
Modelling and Process Optimization for Functionally Graded Materials: *Dan Tortorelli*[1]; [1]University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., 350 MEB, MC 244, 1206 W. Green, Urbana, IL 61801 USA

We optimize continuous quench process parameters to produce functionally graded aluminum alloy extrudates. To perform this task, an optimization problem is defined and solved using a standard nonlinear programming algorithm. Ingredients of this algorithm include 1) the process parameters to be optimized, 2) a cost function: the weighted average of the precipitate number density distribution, 3) constraint functions to limit the temperature gradient (and hence distortion and residual stress) and exit temperature, and 4) their sensitivities with respect to the process parameters. The cost and constraint functions are dependent on the temperature and precipitate size which are obtained by balancing energy to determine the temperature distribution and by using a reaction-rate theory to determine the precipitate particle sizes and their distributions. Both the temperature and the precipitate models are solved via the discontinuous Galerkin finite element method. The energy balance incorporates nonlinear boundary conditions and material properties. The temperature field is then used in the reaction rate model which has as many as 105° of-freedom per finite element node. After computing the temperature and precipitate size distributions we must compute their sensitivities. This seemingly intractable computational task is resolved thanks to the discontinuous Galerkin finite element formulation and the direct differentiation sensitivity method. A three-dimension example is provided to demonstrate the algorithm.

Computational Modeling of Materials, Minerals & Metals Processing: Track A - Optimization & Novel Methods

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

Monday PM	Room: 619
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Dan Tortorelli, University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., Urbana, IL 61801 USA

2:45 PM
Lattice Boltzmann Methods for Metallurgical Process Simulation: *Christian Redl*[1]; [1]Mining University Leoben, CD-Lab. Appl. Comptnl. Thermofluid-dyn., Franz-Josef-Strasse 18, Leoben 8700 Austria

The Lattice Boltzmann Method (LBM) solves fluid dynamics problems based on a discrete mesoscopic approach. It is based on the Lattice Boltzmann equation which is a special discretisation of the continuous Boltzmann equation. Its principle advantage is that it can handle complex physical phenomena (like interaction between different components and surface effects), complicated boundary and initial conditions very well, free from many gridding and stability constraints that plague conventional numerical methods used for fluid flow simulations. The LBM algorithm has a simple structure and acts locally, which is favourable for parallel computing. Complex structures like porous media can be resolved directly. Its potential for metallurgical process simulations is demonstrated in the present study which is concerned with the simulation of the flow in a copper winning electrolysis cell. The electrolyte is modelled as the carrier fluid and the oxygen is considered via a so called active scalar equation. The modelling of buoancy, momentum exchange and the free surface requires special effort. The results of the simulation are validated against experimental data obtained by Laser-Doppler-Anemometry. Despite the simplifications made, good agreement was found.

3:10 PM
Viscosity Estimation Model for an Oscillating Cup Viscometer: *Deming Wang*[1]; R. A. Overfelt[1]; [1]Auburn University, Dept. of Mechl. Eng., 201 Ross Hall, Auburn University, AL 36849 USA

Viscosity measurements of molten alloys become more and more important in modern metallurgy engineering. Lack of the viscosity data of many new alloys hampers many CAD computer codes to apply in casting manufactures. The oscillating cup viscometer has become a dominant technique to measure the viscosities of high temperature molten metals. Unfortunately, the viscosity estimation model from the observed logarithmic decrement and period of the oscillation is very complicated. There are still large discrepancies of viscosity estimation values between using different measurement facilities or using different viscosity estimation models for a same molten metal. The purpose of the paper is to evaluate the accuracy of an oscillating cup viscometer in Auburn University. Two well known viscosity estimation models, Roscoe's and Torklep's equations, are discussed and compared viscosity for different alloys. The theoretical literature for the fluid flow inside an oscillating cup is reviewed and a more accurate working equation for Auburn oscillating cup viscometer is developed. Some design parameters of the oscillating cup viscometer which also directly affect the accuracy of viscosity estimation by using the working equation are discussed. In addition, applications and experimental measuring data are presented in the paper for several different commercial alloys, such as aluminum alloys: A319, A356 and A201, nickel-base super alloys: In713 and In718, and casting irons, C40 Gray Iron and Ductile Iron.

3:35 PM Break

3:50 PM
Numerical Optimization of Magnesium Reduction in a Modified Pidgeon Process: *Alfred Yu*[1]; *Henry Hu*[2]; [1]Nanjing Welbow North America Office, 601-969 Felix Ave., Windsor, Ontario N9C 4C7 Canada; [2]University of Windsor, Mechl. & Matl., 401 Sunset, Windsor, Ontario N9B 3P4 Canada

A numerical model of heat and mass transfer in the retort was set up to simulate and optimize the reduction phenomena in the process of Pidgeon magnesium reduction. The simulations were run to determine the effect of varying processing parameters on the magnesium reduction time. The model predicted the temperature distributions, the heating curves, the recovery ratio of magnesium, and the total process time. The predictions were used to optimize the magnesium reduction process, the dimensions of retort, the shapes of materials, and reaction cycle. Demo operation shows that, with application of the optimizations, significantly production capacity increases in the same furnace, reduction period decreases, energy consumption decreases.

4:15 PM
Deterministic and Regression Models of Nickel Oxide Reducing Roasting Process: V. M. Paretsky[1]; *A. V. Tarasov*[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., 129515 Moscow Russia

A mathematical model of the low-temperature (soft) nickel oxide reduction (SNOR) in a tubular kiln has been developed including an equation describing changes in the nickel oxide content of the solid material along the length of the kiln; an equation describing the changes in the content of reagents ensuring nickel reduction (i.e., hydrogen and carbon monoxide) in the gas phase along the kiln length; as well as the temperature along the kiln length. The kinetic relationships used for the development of the model had been determined based on experiments conducted specially for this purpose. Model investigations were conducted to determine the distribution of such parameters along the length of the kiln as the contents of metals oxides in the solids, partial pressure of hydrogen and carbon monoxide, variations of the temperature depending on the SNOR process conditions, including the kiln rotation speed. Based on the data obtained, a simplified regression model of the reducing roasting process was developed.

4:40 PM
Integrating Computational Mechanics and Numerical Optimization for the Design of Material Properties in Electronic

Packages: *Stoyan Stoyanov*[1]; C. Bailey[1]; M. Cross[2]; [1]University of Greenwich, Sch. of Compg. & Mathl. Sci., Greenwich, London SE10 9LS UK; [2]The University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., Old Royal Naval College, Greenwich, London SE10 9LS UK

Silicon components containing transistor circuitry are at the heart of electronic products such as computers, mobile phones, etc. These components are connected to printed circuit boards (PCB's) using solder material that acts as the conductor for both electricity and heat. During the lifetime of a product it will undergo many thermal cycles where the chip becomes hot during product operation and then cools when the product is switched off. At the product design stage engineers undertake thermo-mechanical simulations to predict the thermal stress in the solder joints due to this thermal cycle and to ensure that the stress magnitude will not result in early fatigue-type failure. The design engineer's aim is to identify component parameters which ensure that the solder will survive beyond a certain number of thermal cycles. The aim of integrating computational mechanics and optimization tools together is to speed up dramatically the design process. From viewpoint of a design engineer in the electronics manufacturing sector, these tools can be used to quickly estimate key design parameters (i.e. material properties, product dimensions, etc.) that will guarantee the required product performance. In this paper a modeling approach coupling computational mechanics techniques with numerical optimization is presented and demonstrated. The integrated modeling framework is obtained by coupling the multi-physics framework–PHYSICA–with the design optimisation tool–VisualDOC. Thermo-mechanical simulations are presented that predict the creep strains in solder material. Different numerical optimization procedures: Direct and Response Surface based optimization plus Design of Experiments, are tested as a part of this modeling framework.

5:05 PM
Computer-Aided Modeling and Control of Autogenous Copper Smelting Process: *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

Based on theoretical and experimental studies into the specific features of the mechanism, kinetics, aerodynamics and heat transfer parameters of the flame-type oxidation of metal sulfides in a stream of technical-grade oxygen, a three-dimensional deterministic mathematical model of an oxygen-flame smelting furnace has been developed on the basis of the zonal method for computation of heat exchange. The model is presented in the form of equations taking into consideration the radiation component of the selective radiation, the convection component of mass transfer between the space and surface zones, heating of charge particles due to heat conductivity, accretions formation on a multi-layer refractory lining with inserted cooling elements. The adaptation of the model for a full-scale furnace has demonstrated the possibility for its use for computation of main smelting process parameters. Multifactor model investigations were conducted and the results obtained were applied using statistical methods for development of a simplified computational regression model describing the interrelation of the main output parameters: temperatures of flame, lining, slag and off-gas; heat fluxes to the lining; thickness of accretions; heat removed by cooling elements; copper content of matte. On the basis of this model, a block diagram of the algorithm was developed for control of the thermal conditions of the furnace, as well as the control algorithm itself.

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

Monday PM — Room: 620
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Vaughan Voller, University of Minnesota, Saint Anthony Falls Lab., Minneapolis, MN 55414-2196 USA; Matt Krane, Purdue University, Dept. of Matls. Eng., W. Lafayette, IN 47907 USA

2:45 PM
A Marker Chain Front Tracking Method for Modelling Meniscus Dynamics in the Al Ingot Casting Process: *Fionn Iversen*[1]; Jon Arn Bakken[1]; Stein Tore Johansen[1]; [1]Norwegian Institute of Science and Technology (NTNU), Matls. Tech. & Electrochem., A. Getz vei 2B, Trondheim N-7491 Norway

In conventional direct chill (DC) hot-top casting of aluminium extrusion ingot using 'gas-slip', poor surface quality of the cast ingot may appear. It is believed that these defects are related to instabilities such as periodic oscillations or folding of the meniscus (the interface between liquid metal and gas in the mould). The object of this work is to develop a stable and reliable model for simulation of the meniscus dynamics. A new 2D cylinder symmetric front tracking model has been developed for the meniscus propagation, and is implemented in a general Navier Stokes solver. The model is based on a finite volume cubic spline marker chain technique. The advantages of the model is its applicability to cases with large density ratios and its ability to calculate independent velocity fields for phases on separate sides of an interface, thereby making it possible to model the dynamics of thin films with a thickness scale smaller than the typical grid size. Effects of surface tension and wetting are also easily applied in the marker chain model. The model is applied to the DC casting process. Results are compared with data from plant test runs and suggestions are made on how to improve the casting process.

3:10 PM
Computational Modeling of Heat Mass and Solute Transport in Directional Solidification Processes: *Mohammed El Ganaoui*[1]; Patrick Bontoux[2]; [1]Université de Limoges, Phys./Numcl. Mod., 123 Albert Thomas, Limoges 87000 France; [2]CNRS, Numcl. Mod., IRPHE, Marseille, 13451 France

During directional solidification, absorption or rejection of latent heat or solute by the solidification front induce convective flows in the liquid phase. These convective motions affect heat and mass transfer in the vicinity of the solidification interface and is subject to many studies. In this work a numerical approach is presented. To avoid remeshing needed by front tracking methods a time-dependent homogeneous formulation is considered to verify implicitly thermal and solutal Stefan conditions at the interface. The numerical solution is based on finite volume approximation. The previous work show that the present method describe accurately the hydrodynamic transition and the interaction with the solid liquid interface in the case of pure material. This study focuses on the occurrence of solutal convection in gradient freezing applications. In a first step only the fluid phase is investigated and the results are validated with respect to spectral ones. In a second step the full solid/liquid model is investigated. A linear approximation of the equilibrium phase diagram is considered for establishing relations between mass fraction and temperature fields to close the set of conservation equations. The present work shows that the method is able to describe with accuracy close to spectral one complex phenomena occurring in reduced configuration to fluid phase. The global model with solid phase account correctly the interface displacement and its interaction with solutal field.

3:35 PM Break

3:50 PM
Computational Modelling of Freeze Layers in Smelting Processes: *Andrew P. Campbell*[1]; Koulis A. Pericleous[1]; Mark Cross[1]; [1]University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., 30 Park Row, Greenwich, London SE10 9LS UK

The use of computational modelling in examining process engineering issues is very powerful. It has been used in the development of the HIsmelt® process from its concept. It is desirable to further water-cool the HIsmelt® vessel to reduce downtime for replacing refractory. Water-cooled elements close to a metal bath run the risk of failure. This generally occurs when a process perturbation causes the freeze and refractory layers to come away from the water-cooled element, which is then exposed to liquid metal. The element fails as they are unable to remove all the heat. Modelling of the water-cooled element involves modelling the heat transfer, fluid flow, stress and solidification for a localised section of the reaction vessel. The complex interaction between the liquid slag and the refractory applied to the outside of the water-cooled element is also being examined to model the wear of this layer. The model is being constructed in Physica, a CFD code developed at the University of Greenwich. Modelling of this system has commenced with modelling solidification test cases. These test cases have been used to validate the CFD code's capability to model the solidification in this system. A model to track the penetration of slag into refractory has also been developed and tested.

4:15 PM
Mathematical Modeling of Heat Transfer and Microporosity Formation in Die Cast A356 Wheels: P. Vo[1]; *D. Maijer*[1]; S. L. Cockcroft[1]; M. A. Wells[1]; C. Hermesmann[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., Vancouver, British Columbia V6T 1Z4, Canada; [2]Canadian Autoparts Toyota, Inc., 7233 Progress Way, Delta, British Columbia V4G 1E7, Canada

Die cast aluminum wheels are one of the most difficult automotive castings to produce because of stringent cast surface and internal quality requirements. A mathematical model has been developed to predict heat transport and porosity formation in die cast A356 wheels as part of a collaborative research agreement between researchers at the University of British Columbia and Canadian Auto Parts Toyota, Incorporated. The heat transfer model, employing the commercial finite element code ABAQUS, is a three-dimensional, 30° slice of the wheel and die that describes forced air cooling, natural convection of the die to the surrounding environment, and interfacial heat transport between the wheel and die sections. Extensive temperature measurements in the die and in the wheel taken over several cycles in the casting process were used to fine tune and validate the model. Preliminary work on predicting porosity formation focused on using the Niyama parameter as a measure of the probability of porosity. The latest version of the model incorporates a new more fundamentally based porosity criterion, which takes into account the effect of hydrogen and inclusion content. The development of this model together with some early results will be presented.

4:40 PM
Modelling Filters in Metal Casting Processes: *Mark R. Jolly*[1]; Jean-Christophe Gebelin[1]; [1]The University of Birmingham, IRC in Matls., Edgbaston, Birmingham, W. Midlands B15 2TT UK

A number of different types of so-called filters are used on the metal casting industries to impart some cleaning effect and flow control on the liquid metal as it passes through them. The filters range from simple planar meshes through extruded channels to reticulated foam structures. It is most common that software packages used in the industry model the filters by a simple pressure drop associated with some area fraction and permeability parameters. Recent experimental work at the IRC in Birmingham has shown that filters of the same type can behave very differently depending upon the casting process in which they are employed. Modelling filter geometries for a range of different casting processes has indicated that the flow of metal and heat losses through the filters are rather complex and should be considered when using filters in the casting processes. This paper will present a number of cases of different types of filters modeled and different processes and indicate some of the sensitivities of the processes to boundary conditions imposed by the process.

5:05 PM
Computer Heat Transfer Model for Directionally Solidified Castings: *Deming Wang*[1]; R. A. Overfelt[1]; [1]Auburn University, Mechl. Eng. Dept., 201 Ross Hall, Auburn University, AL 36849 USA

Thermal transfer control is very important in directionally solidified (DS) castings. This paper presents a simple and efficient computer-aided heat transfer simulation method to predict the thermal characteristics of an alloy sample in a special furnace for directional solidification. A two-dimensional transient heat transfer by radiation combined with conduction is developed to calculate the energy exchange between the symmetric furnace and the sample. A control volume technique is used to obtain a set of highly efficient finite difference equations for heat conduction and heat radiation with changeable view factors. The model well simulates the transient process of DS castings. The simulation results are verified by a few measurable experimental results. Using the two-dimensional computer simulation model, many thermal properties of the samples can be obtained, such as temperature distribution, solidification velocity, the shapes and positions of the liquid/solid interface and thermal gradient at the inter faces. These are very important to analyze microstructure of DS casting alloy, avoid casting defects and control the quality of DS castings.

Computational Phase Transformations: Atomic Level Modeling of Interface Motion and Phase Transformations

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

Monday PM — Room: 201
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Jeff Rickman, Lehigh University, Matls. Sci. & Eng., #5 Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18105-3195 USA

2:00 PM
Impurity Effects on Interface Migration: *David J. Srolovitz*[1]; Mikhail I. Mendelev[1]; [1]Princeton University, Princeton Matls. Inst., Princeton, NJ 08544 USA

The migration of interfaces is central to all first order phase transformations and many types of coarsening phenomena. Even in high purity materials, the presence of impurities can dramatically effect how interfaces move. In this presentation, we will discuss the effects of impurities on moving interfaces both from the perspectives of a discrete, Ising-like model and continuum theory. We begin by reviewing the classical continuum theories of Cahn and Lücke and Stüwe and compare their predictions with the results of computer simulations. While these theories capture much of the physics, they do not lead to quantitatively reliable predictions. This is not surprising since the fundamental events associated with interface migration occur via atomic-scale mechanisms, which the impurities can modify. A more quantitative theory is presented based on the details of the atomic scale mechanism. Finally, we examine the effects of co-segregation on interface mobility. A number of new phenomena have been observed, including the identification of conditions under which increasing the impurity concentration can increase the interface mobility.

2:45 PM
Phase Transitions and Dynamical Behavior in Perovskite Ferroelectrics by Molecular-Dynamics Simulation: *Simon R. Phillpot*[1]; Marcelo Sepliarsky[1]; Marcelo Stachiotti[2]; Ricardo Migoni[2]; [1]Argonne National Laboratory, Matls. Sci. Div., Bldg. 212, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Instituto de Fisica Rosario, CONICET-UNR, Rosario Argentina

We use molecular-dynamics simulations to explore two aspects of the phase behavior and dynamical behavior of ferroelectric perovskites. In superlattices consisting of equal thickness layers of a perovskite ferroelectric (KNbO3) and a perovskite paraelectric (KTaO3) we find that the behavior is determined by the strength of the coupling between the KNbO3 layers, mediated by induced polarizations in the KTaO3 layers. Our results are consistent with recent experiments on this system. We also use molecular-dynamics simulations to explore the atomic-level details of the dynamics of polarization reversal in the tetragonal phase of a monodomain order-disorder perovskite ferroelectric. We find that the transition from an [001] orientation to an [00-1] orientation involves polarization rotation through intermediate states with polarization parallel to [011] and [01-1], accompanied by a strong transient piezoelectric response. This work was supported by the US Department of Energy, Office of Science under Contact W-31-109-Eng-38, by the DOE S&P Center Project on Nanoscale Phenomena in Perovskite Thin Films, and by CONICET-Argentina.

3:15 PM
Dynamical Self-Organization in Alloys Driven by Irradiation or Plastic Deformation: *Pascal Bellon*[1]; Jiwen Liu[1]; Raul A. Enrique[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

The microstructure of alloys driven away from equilibrium by ion irradiation or by plastic deformation can undergo spontaneous organization. In particular, as a result of the competition between external driving forces and internal relaxation processes, the composition field can develop patterns with a characteristic, finite length. In order to determine the conditions required to trigger such a self-organization, and to understand the wavelength selection process, we have developed kinetic Monte-Carlo simulations and continuum kinetic models. The main results is that the external driving force has to introduce a new length scale, different from that of the thermal relaxation process for patterning to take place, and that the structure of the external noise (external fluctuations) can play an important role in the wave length selection. Examples will be given on alloys that would undergo phase separation (e.g., Cu-Ag) or phase ordering and phase separation (e.g., Ni-Al) at equilibrium.

3:45 PM Break

4:00 PM
Atomistic Computations of Thermodynamic and Kinetic Properties of Solid-Liquid Interfaces: *Jeff J. Hoyt*[1]; Mark Asta[2]; Alain Karma[3]; [1]Sandia National Laboratories, PO Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA; [2]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [3]Northeastern University, Phys., 360 Huntington Ave., Boston, MA 02115 USA

The shape and velocity of a dendrite formed during solidification of an alloy depend critically on several thermodynamic and kinetic properties of the solid-liquid interface. In this talk we will review several molecular dynamics and Monte Carlo simulation techniques which can accurately determine many of the important interfacial properties. In each case the embedded atom method is used to model the interatomic potential and results are reported for Ag, Au, Cu, Ni and Pb and the alloys Al-Cu and Cu-Ni. The solid-liquid interfacial free was determined using the fluctuation spectrum method, a technique which is capable of resolving the very small anisotropy. The individual excess entropy and excess energy contributions to the interfacial energy will also be reported. The kinetic coefficient was found by monitoring the velocity of a planar solid-liquid interface as a function of undercooling for the three low index growth directions 100, 110 and 111. Simulation results will be compared to the crystallization rate model of Burke, Broughton and Gilmer. For the binary alloys we have computed the relative absorption of solute to the solid-liquid boundary using Monte-Carlo and thermodynamic integration techniques. Finally, the segregation coefficient as a function of velocity for Al-Cu will be presented and the results will be discussed in terms of various theoretical models and experiment.

4:30 PM
Monte-Carlo Simulations of Phase Separation in Binary Alloys: Peter Fratzl[1]; *Richard Weinkamer*[1]; Himadri Gupta[2]; Joel L. Lebowitz[2]; [1]Austrian Academy of Sciences and University of Leoben, Erich Schmid Inst. of Matls. Sci., Jahnstrasse 12, Leoben 8700 Austria; [2]Rutgers University, Busch Campus, Depts. of Math. & Phys., New Brunswick, NJ 08903 USA

Using three-dimensional atomistic Monte-Carlo simulations, we have studied phase separation in binary alloys with and without lattice misfit between precipitates and matrix. The elastic interactions are modeled by springs connecting the atoms. Our investigations concentrated on the influence of the misfit on the morphology of the precipitates, their size distribution and growth kinetics. Introducing a slight elastic inhomogeneity into the model, an additionally applied external stress resulted in directional coarsening ("rafting") of the precipitates. Neglecting elastic interactions, we have also studied the influence of vacancies and their effective interaction with the different atomic species on the phase separation kinetics. We found that when the vacancy is preferentially attracted to the precipitates, coarsening does not proceed via the usual evaporation-condensation mechanism, but according to a coagulation mechanism of moving precipitates.

5:00 PM
A Semi-Empirical Atomic Potential for Atomistic Approach to Phase Transformation: *Byeong-Joo Lee*[1]; [1]KRISS, Matls. Evaluation Ctr., Yusong PO Box 102, Taejon 305-600 Korea

Atomistic Simulation can be a useful tool to analyze and predict phase transformations, crystallographic changes and mechanical properties of metals. In order to obtain reliable results within reasonable computing time, it is important to use a well-assessed semi-empirical atomic potential for elements and alloy systems. In the present presentation, a recently developed atomic potential model, the second nearest-neighbor modified embedded atom method (MEAM), will be introduced. Various physical properties of elements (mainly bcc transition metals), including elastic constants, structural properties, point defect properties, surface properties and thermal properties will be calculated and compared with experiments or high level calculations. Some examples for description of alloy systems, and an atomistic approach to recrystallization will also be presented.

Creep Deformation: Fundamentals and Applications: Fundamental Behavior - II

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

Monday PM	Room: 214
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chair: M. E. Kassner, Oregon State University, Dept. of Mech. Eng., 414 Rogers Hall, Corvallis, OR 97331 USA

2:00 PM Invited
Rate Controlling Processes for Five Power-Law Creep: *M. E. Kassner*[1]; S. C. Bergsma[2]; [1]Oregon State University, Dept. Mechl. Eng., Rogers Hall, Corvallis, OR 97331 USA; [2]Northwest Aluminum Company, 3313 W. Second St., The Dalles, OR 97058 USA

The mechanisms for recovery-controlled-creep in pure and Class M (pure metal behavior) alloys at elevated temperature, usually within the five-power-law creep regime, will be discussed. Specifically, the roles of the dislocation substructure and the so-called internal stresses on the rate controlling mechanisms will be described in terms of recent TEM with convergent beam electron diffraction, in-situ TEM and dislocation modeling techniques.

2:25 PM Invited
Creep Behavior at Very Low Stresses: *Farghalli A. Mohamed*[1]; [1]University of California, Cheml. Eng. & Matls. Sci., 916 Engineering Tower Bldg., Irvine, CA 92697-2575 USA

This paper reviews recent information on two Newtonian deformation processes, Nabarro-Herring creep and Harper-Dorn creep, that may control the creep behavior of materials at very low stresses. In particular, the paper focuses on discussions and results related to the requirements for the occurrence of both deformation processes during the high-temperature deformation of materials.

2:50 PM
The Effects of H_2SO_4 on the Mechanical Behavior of Ice Single Crystals: *Ian Baker*[1]; Y. L. Trickett[1]; X. Li[1]; D. Iliescu[1]; P. M.S. Pradhan[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Tests were performed in compression at -20°C (0.93 T_m) at a variety of strain rates on three different orientations of ice singles with and without sulfuric acid (a naturally-occurring contaminant). Unlike the effect of impurities on most materials, the H_2SO_4 (as little as 100 p.p.b.) dramatically decreased both the peak stress and the subsequent flow stress of the ice single crystals at all strain rates. The stress exponent was determined to be ~2 and was unaffected by the dopant. The peak strength softening was shown to be proportional to the square root of the concentration of sulfuric acid. Synchrotron x-ray topography showed that the doped crystals had a grown-in dislocation density of 1.0 X 10^{10} m^{-2}. Tests were also performed at -10°C and -30°C for a single orientation and on ice from Byrd Station, Antarctica. Research supported by NSF grant OPP-9980379 and ARO grant DAAD 19-00-1-0444.

3:10 PM
A Framework for Modeling Creep: *Holger Brehm*[1]; *Michael J. Mills*[1]; *Glenn S. Daehn*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

We propose a method of modeling creep that couples traditional equations for dislocations overcoming obstacles in a thermally activated manner with those for diffusion-controlled microstructural coarsening, and microstructural refinement due to plastic flow. We find that with this model, over a wide range of input parameters, we can recover: a steady-state stress exponent near 5, a constant structure stress exponent which is much higher, a steady-state creep activation energy that is very close to that of diffusion, and the steady-state inter-obstacle spacing (which scales with subgrain size) which is nearly inversely proportional to the applied stress. One of the attractive features of this model is that it allows interrogation of the separate phenomena (slip, refinement and coarsening) that are essential to the creep process.

3:30 PM

Departures from Model Predictions for Creep in Pure Metals at Low Stresses Near their Melting Temperature: *Kevin Robert McNee*[1]; Vivek Srivistava[1]; Geoffrey Winston Greenwood[1]; Howard Jones[1]; [1]University of Sheffield, Dept. of Eng. Matls., Mappin St., Sheffield S1 3JD UK

Early experiments that verify the diffusional creep rate predictions of Nabarro and Herring were undertaken just below the melting temperature and on small diameter wires and thin foils with through thickness grains. Results were shown to give reasonable agreement with predictions at sufficiently long test durations but specimens of other geometries and grain shapes have shown more variability. Recently some of these results have been re-interpreted and the applicability of the Nabarro-Herring creep mechanism to them has been questioned. Further experiments have been carried out in the present work on a number of pure metals to determine possible sources of the experimental diversity and to re-evaluate operative mechanisms.

3:50 PM Break

4:00 PM Invited

Deformation Anomalies during Creep of Titanium Alloys: Matthew Brandes[1]; Thirumalai Neeraj[1]; Michael F. Savage[2]; *Michael J. Mills*[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]National Institute of Standards and Technology, Metall. Div., Matls. Perfor. Grp., Gaithersburg, MD 20877-8550 USA

Titanium alloys are attractive structural materials because of their high specific strength and excellent corrosion resistance. It has been recognized for some time, however, that Ti alloys must be employed conservatively because of their tendency to creep significantly at room temperature, even at stresses well below the macroscopic yield strength. This presentation will describe two additional, anomalous characteristics of these alloys under these conditions: (a) a distinct tension/compression (T/C) asymmetry and (b) dramatic recovery of strain hardening at room temperature. We will show that a T/C asymmetry is associated with deformation via a-type dislocations, and attempt to rationalize this finding via detailed examination of the core structure of these dislocations using high resolution TEM investigation. The room temperature recovery phenomenon appears to be correlated with the presence of dislocation pile-ups in the alpha-phase of these alloys, as revealed by diffraction-contrast TEM studies. The possible implications of these effects with respect to designing with Ti alloys will be discussed.

4:25 PM Invited

On the Mechanisms Controlling Steady State Deformation of Metals and Alloys: *Erik Nes*[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., N-7491, Trondheim, Norway

The fundamental problems of work hardening and steady state deformation relate to: (i) How to calculate the flow stress at a constant microstructure? (ii) How to define the mechanism of athermal storage of dislocations? And (iii) how to treat dynamic recovery? These are difficult questions, which have stimulated extensive research efforts over many decades. In an effort to reach an improved understanding, a new approach towards these problems has recently been proposed by the present author and co-workers. The model presented is based on a statistical analysis of athermal storage of dislocations. By combining the solution for the dislocation storage problem with models for dynamic recovery of network dislocations and sub-boundary structures, a general internal state variable description is obtained. The model includes effects due to variations in: stacking fault energy, grain size, solid solution content, and particle size and volume fraction. The result is a work hardening model, which in principle is capabl e of providing the stress-strain behavior for a given metal or solid solution alloy under condition ranging from deformation in the ambient temperature range to high temperature creep. In this presentation, the basic model concepts will be discussed and contrasted in relation to alternative interpretations found in the creep literature.

4:50 PM

Recent Advances in Modeling Creep Deformation and Damage: *James C. Earthman*[1]; [1]University of California-Irvine, Chem. & Biochem. Eng. & Matl. Sci., CBEMS, Irvine, CA 92697-2575 USA

Recent research accomplishments in the area of modeling concomitant creep processes and associated damage will be reviewed. Numerical models will be discussed which simulate different creep mechanisms for a range of materials and conditions. Experimental evidence will also be discussed which is particularly insightful for modeling the development of creep damage. For example, data for specimens tested under multiaxial stresses will be presented that are indicative of the principal damage mechanism. Results will be compared and contrasted in order to identify new insight and future research directions that will lead to better methods for predicting creep failure and improved materials performance at elevated temperatures.

5:10 PM

Equation for High Temperature Creep Rate of Pure Metals and Solid Solutions with Complete Structural Factor: *Alexander Illich Dekhtyar*[1]; [1]Kurdyumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Strength & Plasticity of Inhomogeneous Alloys, 36 Vernadsky Blvd., Kiev, Kiev Region 03680 Ukraine

The complete structure factor has found for equation of high temperature creep rate of pure metals and dilute solid solutions with FCC and BCC lattice. This factor involves the structural parameters of different structure scales: electronic and atomic structure, inside structure of individual dislocations, dislocation structure including dislocation distribution, grain structure. The structure factor includes the fundamental values of metals, such as atomic mass, melting point, shear modulus. The expression for the structure factor was deduced on the basis of experimentally found for a lot of metals and alloys strong dependence of creep rate on the widening of the reciprocal lattice poles. The reciprocal lattice pole widening is determined by X-ray diffraction investigations with high statistics as reflex widening in azimuthal direction. Obtained equation is proved both for primary and steady state creep stages.

5:30 PM

Interaction Between Dislocations during High Temperature Creep Process: *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

Interaction forces between pairs of straight dislocations in BCC lattice have calculated for the case of high temperature creep deformation. It was done for different combinations of screw, edge and mixed dislocations with different Burgers vectors in different slip planes. These forces were compared with the forces acting under external stresses on the dislocation straight parts determined experimentally by the electron transmission microscopy in single crystalline molybdenum undergone creep deformation. It is found that screw dislocations do not accumulate in subboundaries and all of dislocations do not intersect during creep process at low applied stresses. At high applied stresses dislocations might mutually intersect to create the jog concentration enough for realization the mechanism of intensive collective climb within subboundaries.

David L. Davidson Symposium on Fatigue: Fatigue Thresholds and Life Prediction

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

Monday PM	Room: 208
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Peter K. Liaw, University of Tennessee, Matls Sci. & Eng., Knoxville, TN 37996-2200 USA; Robert O. Ritchie, University of California-Berkeley, Matls. Sci & Eng., Berkeley, CA 94708-1238 USA

2:00 PM Keynote
High-Cycle Fatigue in Turbine Engine Alloys: *R. O. Ritchie*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94708-1238 USA

High-cycle fatigue (HCF) is a prime cause of military aircraft turbine engine failures. It results from fatigue-crack growth in blades and disks, initiated at small defects often associated with fretting or foreign object damage. Due to the high frequencies (>1 kHz) involved, design based on a HCF threshold would appear to be a preferred approach. In this work, the nature of the fatigue threshold is examined under representative high frequency and high load-ratio conditions, in a Ti-6Al-4V blade alloy with bimodal and lamellar microstructures, with emphasis on behavior following foreign-object damage (FOD) and under mixed-mode (modes I + II) loading conditions. It is shown that for all crack sizes, large or small, but of dimensions large compared to microstructural size-scales ("continuum-sized" cracks), a worst-case fatigue threshold can be defined (at load ratios where R approaches unity such that crack closure is minimized) which represents a lower-bound stress intensity for fatigue-crack growth. This holds for mixed-mode loading (studied over a wide range of mode-mixities at load ratios from 0.1 to 0.8), provided the threshold is characterized under worst-case mode I conditions in terms of the strain energy release rate. However, for crack sizes comparable with microstructural dimensions, i.e., < 10 microns in this alloy, as can be found in the vicinity of damaged regions due to FOD impacts, fatigue thresholds can be a factor of two lower. In such instances where the critical condition for HCF must be defined in the presence of such microstructurally-small cracks, the Kitagawa-Takahashi diagram is more appropriate, where the limiting conditions are defined in terms of the smooth-bar fatigue limit (at microstructurally-small cracks sizes) and the "worst-case" fatigue threshold (at larger, "continuum-sized" crack sizes).

2:30 PM Invited
Step Loading, Coaxing and Small Crack Thresholds in Ti-6Al-4V under High Cycle Fatigue: *Ted Nicholas*[1]; [1]US Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

The conventional method for determining fatigue strength at high cycle counts is to either extrapolate S-N data from lower cycle counts or to generate S-N data in the high cycle count regime using specialized high frequency test machines. As an alternative, a step-loading method may be employed. One question that arises from a step-loading test is any effect due to coaxing, a phenomenon that causes an elevation of the fatigue stress due to prior cycling below the fatigue limit. Data have been obtained which validate the step-loading methodology. Applicability of high frequency testing to the determination of fatigue crack growth thresholds will also be discussed. It will be shown that fatigue limits for small cracks are not drastically altered from uncracked material when using stress rather than stress intensity as the governing parameter with which to characterize threshold behavior.

3:00 PM Invited
A Damage Tolerance Approach for Predicting the Threshold Stress for High Cycle Fatigue in the Presence of Supplemental Damage: *Stephen J. Hudak*[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., San Antonio, TX 78238-5166 USA

The occurrence of foreign object damage (FOD) in fan and compressor blades and fretting-fatigue at blade-to-disc attachments can significantly reduce the high cycle fatigue (HCF) resistance of advanced turbine engines. Thus, both of these damage processes need to be explicitly accounted for in design and structural integrity assessments. The high stress concentrations associated with these damage processes often lead to the initiation of microcracks early in the fatigue life, particularly under service loading where low cycle fatigue is also present. Consequently, a fracture mechanics based threshold approach has been developed for treating the growth and arrest of microcracks within the steep gradients that exist at FOD notches and edge-of-contact regions in fretting fatigue. The basis of the methodology will be described and the viability of the approach will be assessed by comparing model predictions with available HCF threshold stress measurements on Ti-6Al-4V specimens containing notches, simulated FOD, fretting, and fretting-fatigue. The consequences of treating the crack growth threshold stress intensity factor as a crack-size-dependent property will be discussed. Key similarities and differences between the FOD notches and edge-of-contact regions will also be highlighted. This work was supported under US Air Force Contract No. F49620-99-C-0007 monitored by Dr. Jeff Calcaterra of AFRL/ML and managed by Dr. Joe Gallagher of UDRI.

3:30 PM Break

3:40 PM Invited
Fatigue Life Predictions in Small Volume Components: *William W. Gerberich*[1]; [1]University of Minnesota, Chem. Eng. & Matls. Sci., 151 Amundson Hall, Minneapolis, MN 55455 USA

Over the years there has been a gradual recognition that the one to one correspondence between crack advance per cycle and fatigue striation spacing broke down at low *K. In an attempt to resolve whether this might apply to small volumes, small diameter wires were evaluated in tension-tension fatigue. Fatigue striations on the order of 20 nm per cycle were able to be resolved at 100,000 and 200,000X magnifications at *K = 4MPa-m1/2 and R>0.5 for both Ta and MP35N wire 250 µm in diameter. From similar tests using runouts in the 10 million cycle regime, thresholds were established. From the fatigue crack propagation tests a Paris Law exponent of two was found and a threshold-dependents algorithm allowing for zero growth as *K approached threshold was established. Using standard integration procedures, it is shown that the fatigue life of sharply notched wires is divided into regimes where initiation is no greater than about half of the total fatigue life for samples with *K > 3MPa-m1/2. The tentative conclusion is that fewer grains traversing the crack front and more uniform stress states combine to allow fcp analysis to afford a reasonable first approximation of failure life in small volume components.

4:10 PM
Mixed-Mode Fatigue Crack Growth Thresholds of Ni-Based Single Crystals: *Kwai S. Chan*[1]; Jim H. Feiger[1]; Yi-Der Lee[1]; Stephen J. Hudak[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA

This investigation examines the effects of stress state and crystal orientation on the near threshold fatigue crack growth (FCG) response of PWA 1484 single crystals. Mixed mode FCG on (010) and (111) planes was characterized in air at 1100°F, using an asymmetric four-point bend test technique. Stress intensity solutions of the crack specimen were obtained using anisotropic fracture mechanics. The influence of mode mixity on the FCG threshold was determined as a function of stress state, ranging from pure Mode I to pure Mode II. Progress in applying the analytical and experimental results to develop a fracture mechanics approach for treating mixed-mode FCG in single crystal materials will be discussed. Work supported by AFRL through Contract No. F49620-99-C-0007.

4:30 PM
High Frequency FCP Thresholds in Nickel-Base Superalloys: *Walter W. Milligan*[1]; Amit Shyam[1]; Santo A. Padula[2]; [1]Michigan Technological University, Matls. Sci. & Eng., Houghton, MI 49931 USA; [2]NASA-Glenn Research Center, 21000 Brookpark Rd., MS 49-3, Cleveland, OH 49931 USA

High temperature, high frequency fatigue crack propagation thresholds were determined for a nickel-base turbine disk alloy. Effects of frequency, temperature, microstructure, and load ratio will be discussed. Relationships between thresholds and fractography and deformation mechanisms are explored. Sponsored by AFOSR MURI on High Cycle Fatigue.

4:50 PM
Cyclic Deformation Behavior and Damage Mechanism of HASTELLOY X Superalloy under Fatigue and Creep-Fatigue Loading: Lijia Chen[1]; *Peter K. Liaw*[1]; Gongyao Wang[1]; Robert L. McDaniels[1]; Kevin Liaw[1]; Scott A. Thompson[2]; James W. Blust[2]; Paul F. Browning[2]; Jose M. Aurrecoechea[2]; Rodger R. Seeley[3]; Dwaine L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The fatigue deformation behavior of a nickel-based superalloy, HASTELLOY X, was investigated at 816C and 927C in laboratory air. It was noted that at both temperatures of 816C and 927C, the alloy exhibited initial cyclic hardening, followed by a saturated cyclic stress response or cyclic softening under fatigue loading condition. For creep-fatigue tests, the alloy showed either cyclic hardening or cyclic stability, which is closely related to the test temperature and the duration of the strain hold time. It was also observed that the fatigue life of the alloy considerably decreased due to the introduction of strain hold times. In addition, the fracture surfaces of the fatigued specimens were observed using scanning electron microscopy to determine the crack initiation and propagation modes. The fatigue cracks was usually found to initiate in a transgranular mode, while both transgranular and intergranular crack growth were observed.

Flyash: Generation, Treatment, Metal Recovery and Disposal - II

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; I. Gaballah, Laboratoire Environmental et Mineralurgie, Associated to CNRS, ENSG-LEM, Vandoeuvre les Nancy 54501 France; David G. Robertson, University of Missouri-Rolla, Department of Metallurgical Engineering, Rolla, MO 65409-1460 USA

Monday PM — Room: 604
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: R. N. Singh, National Environment Engineering Research Institute, Nagpor India; Hideki Yamamoto, Kansai University, Dept. Cheml. Eng., Osaka 564-8680 Japan

2:00 PM Invited
Manufacture of Energy Efficient and Eco-Friendly Alinite Cements using Flyashes and Other Waste Materials: *Pradip*[1]; Maneesh Singh[1]; Anup Lakare[1]; P. C. Kapur[1]; [1]Tata Research Development and Design Centre, 54B Hadapsar Indl. Estate, Pune 411 013 India

We have been able to successfully convert a wide variety of fly ashes and other industrial waste materials into excellent quality hydraulic cements based on alinite cement chemistry. We present in this paper our recent results on the effect of various process variables optimized during our efforts on producing good quality alinite cements from a representative sample of chloride rich municipal incinerator fly ash obtained from South Korea. Most important process parameters include raw mix composition (represented in terms of lime index, silica modulus, iron modulus and chloride content), clinker grinding fineness and the clinkering schedule. It is possible to utilize up to 30% by weight of fly ash in the raw mix needed for the production of these cements. The quality of alinite cements thus produced are comparable in quality (in terms of physical properties and 1:3 cement/sand mortar cube compressive strengths) to portland cements and in fact, meet the Indian standard specifications for ordinary portland cements. Clinkering is carried out at 1150C for successfully achieving the desired proportion of alinite phase in the final cement. X-ray diffraction studies on these cements indicate alinite as the major phase with relatively minor quantities of other phases such as belite and calcium aluminochloride (11 CaO.7Al2O3.CaCl2). Addition of gypsum during clinker grinding is found to have a beneficial effect on the quality of cement produced by this low temperature clinkering route. This process thus offers an economically attractive and eco-friendly method of recycling chloride rich municipal incinerator ashes of considerable nuisance value.

2:30 PM Invited
Recycling Study of Korea's Fly Ash in Municipal Solid Waste Incineration Ashes for Cement Raw Material: *Ji-Whan Ahn*[1]; Hwan Kim[2]; [1]Korea Institute of Geoscience and Mineral Resources(KIGAM), PO Box 111, Yusung, Science Town, Taejon Korea; [2]Seoul National University, Sch. of Matl. Sci. & Eng., Seoul 151-742 Korea

The removal of chloride from fly ash in MSWIA (Municipal Solid Waste Incinerator Ash) by water-washing was investigated. The content of chloride in fly ash was 25-30% and the chloride compounds in fly ash were mainly KCl, NaCl, CaClOH and Friedel's salt (3CaO•Al2O3•10H2O). The effect of time and temperature in washing was surveyed. As a result, we found that the content of chloride in fly ash could be diminished to the content of approximately 2.0% by water washing on condition that washing time is 30 minutes, agitation speed, 300rpm and liquid/solid (v/w) ratio is 10. The removal of the chloride in fly ash can be used to improve the potential application of fly ash including cement raw material and building material and so on.

3:00 PM
Recovery of Lead and Zinc from Fly Ash from Municipal Incineration Plants by Means of Alkaline Leaching and Solvent Extraction: *Katsutoshi Inoue*[1]; Seham Nagib Tawfic[2]; [1]Saga University, Dept. of Appl. Chem., Honjo 1, Saga 840-8502 Japan; [2]Central Metallurgical R&D Institute Egypt

Fly ash from municipal waste incineration (MWI) plant is regulated as a hazardous waste because it contains considerable amounts of heavy metals and hazardous organic materials such as dioxin. Primary fly ash contains 0.8wt.% Zn and 0.12wt.% Pb, 1.37wt.% Fe while secondary fly ash contains 40.18wt.% Zn, 10.7wt.% Pb and 2.12wt.% Fe in addition to large amount of sodium and potassium chlorides. Therefore, it should be treated for detoxification or for the recovery of these metals as secondary resources. The recovery of by acid leaching using different kinds of acids such as sulfuric, hydrochloric and acetic acid was carried out. It was found that sulfuric acid leaching is effective where most of Zn was dissolved and also hydrochloric or acetic acid leaching was effective where most of Pb and Zn were dissolved. However, the acids dissolve also some impurities such as Ca, Fe and Al together with Pb and Zn from the fly ash. Therefore, alkaline leaching using NaOH as leachate was carried out; but, alkaline leaching was found to be suffered from low solubility of Zn. In order to improve Zn solubility, washing the solid residue using 2 or 5wt.% HCl was carried out which achieved the dissolution of almost all Pb and about 68wt.% Zn from secondary fly ash. For the purpose of separating and recovering Pb and Zn from the alkaline leach liquor, solvent extraction behavior of Kelex100 and D2EHPA for these metals from alkaline solution and from acetic acid media, respectively, were investigated.

3:20 PM
Recovering Metals in Fly Ash Derived from Heavy Oil by Combination of Leaching and Ion Exchange Processes: *Hideaki Tokuyama*[1]; Susumu Nii[1]; Fumio Kanaizumi[1]; Katsuroku Takahashi[1]; [1]Nagoya University, Dept. of Cheml. Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Oil fly ash contains such metals as V, Ni and Fe. Recovery of these metals makes the fly ash less poisonous with the result that the ash becomes a valuable resource. The purpose of present study is to develop a process which can selectively recover V and Ni from the fly ash. Experiment of leaching with water, HCl, H2SO4 and NaOH leads us to propose a two-step leaching process as an effective and mild operation. The first step is leaching with water for removal of Ni, Mg, Al and Zn and the second step is leaching with acidic solution for the recovery of V. Then the metals V and Ni are separated from other metals in each leached liquor by ion exchange. Further advantage of this separation process is that the metal recovery by ion exchange permit to reuse acidic leachant, which makes for saving separation energy.

3:40 PM Break

3:55 PM
Heavy Metals Recovery in Vitrification Process of MSW Residues: *Takuya Shinagawa*[1]; Keisuke Nakahara[1]; Hiroshi Yamamoto[1]; Tetsuo Akashi[1]; [1]NKK Corporation, Eng. Rsrch. Ctr., Keihin Bldg. 6F, 1-1 Minami-watarida-cho, Kawasaki-ku, Kawasaki-shi, 210-0855 Japan

Vitrification is considered a principal process for future waste disposal because the treatment can render harmless even wastes that include harmful substances, and it can recover resources in the waste. The products from vitrification, such as slag and metal, can be used as construction material and weights, and moreover, NKK developed a heavy metal recovery process in which zinc and lead in the dust are reclaimed as a raw material for smelting by making the best use of the characteristics of the dust. The NKK electric-resistance ash-melting furnace can volatilize the zinc and lead in residues at high rates using reduction melting and then concentrate these metals in the dust. The concentration of zinc and lead in the dust after washing is sufficiently high for the dust to be used as a raw material for the zinc and lead industry. The NKK electric-resistance ash melting furnace provides high performance in separating heavy metals and can produce dust advantageous for reuse as a raw material for smelting and safe slag containing less heavy metals. This system can ideally fulfill the function of vitrification.

4:15 PM
Spent Pot Lining (SPL) Treatment and Fluoride Recycling Process: *Ken Mansfield*[1]; [1]Portland Aluminium, PB 1, Portland, Victoria, 3305 Australia

Spent Pot Lining (SPL) is a hazardous waste product from the smelting of aluminium due to its fluoride and cyanide content. Disposal of SPL is a major problem for the aluminium industry due to disposal by landfill increasingly being banned. Between 1992-94 Portland Aluminium, Alcoa and Ausmelt conducted trials in Ausmelt's demonstration furnace to treat SPL. From these trials, and work with CSIRO to produce aluminium fluoride from the furnace offgas, Portland Aluminium and Alcoa authorized AUD$24,000,000 in 1995 to construct an SPL treatment facility. Output products from the process are aluminium fluoride, which has been successfully tried in the aluminium smelting process instead of imported material, and a vitreous granulated slag having leachability qualities acceptable to the EPA for specific end uses. The 'SPL Treatment and Fluoride Recycling Process' at Portland, Australia is successfully treating SPL and converting it into useful products.

4:35 PM
Improving Concrete Properties using Flyash: *Harovel G. Wheat*[1]; [1]University of Texas at Austin, Mechl. Eng., TX Matls. Inst., Austin, TX 78712 USA

It has been shown that the incorporation of flyash into concrete as a replacement for part of the cement can be beneficial. In some cases, the strength of the concrete can be increased and the permeability can be significantly decreased. This latter property can lead to a reduction in the migration and/or penetration of aggressive species into reinforced concrete. Some of these species, such as chlorides from deicing salts or marine environments, can subsequently result in accelerated corrosion of the steel reinforcement and a loss of structural integrity of the concrete. Care has to be taken so that the appropriate compositions of the components of the flyash are achieved in order to ensure maximum benefit to the concrete structures. This will be discussed as it relates to flyash generation and treatment.

Fundamentals of Advanced Materials for Energy Conversion: Magnets & Thermal Energy

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

Monday PM	Room: 613
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM USA; Ramana G. Reddy, University of Alabama, A129 Bevill Bldg., PO Box 870202, Tuscaloosa, AL 35487 USA

2:00 PM Plenary
Magnetic Materials for the Conversion of Magnetic Energy to Cooling and Heating: *Karl A. Gschneidner*[1]; Vitalij K. Pecharsky[1]; [1]Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

The application of a magnetic field to a magnetic material near its magnetic ordering temperature aligns the magnetic moments causing the material to warm up. When the field is removed the moments randomize thus cooling the magnetic material. This is analogous to a gas compressor which has been utilized for about 100 years to cool or heat. Recently it has been shown that magnetic refrigeration is a highly efficient technology, reaching 60% Carnot. Proof-of-principle apparati have achieved some notable firsts for magnetic cooling: a cooling power of 600 watts, a coefficience of performance approaching 15 and a temperature span of 38°C. It has been estimated that for large cooling devices, >50 kwatt, magnetic cooling is ~25% more efficient than conventional gas compression technology. Initial capital costs, however, are higher and it takes ~5 years to recover the higher costs. The status of magnetic heating and cooling will be summarized, including home air conditioners and refrigerator/freezers.

2:35 PM Plenary
Progress in Photovoltaics: A Success Story in Crystal Growth and Materials Science: *Thomas Surek*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

Photovoltaics (PV) is solar electric power-a semiconductor-based technology that directly converts sunlight into electricity. The manufacture and sale of PV has grown into a $2 billion industry worldwide, with nearly 300 megawatts (MW) of PV modules shipped in 2000. The majority of today's modules are based on crystalline silicon, building on advances in the growth of single crystal and polycrystalline ingots, as well as innovations in techniques to grow silicon sheets and ribbons directly from the melt. Some 25 years of research has led to the discovery and development of new PV materials and devices based on thin-film semiconductors and related alloys such as amorphous silicon, cadmium telluride, and copper indium diselenide. These materials can be deposited on low-cost substrates, such as glass, stainless steel, or plastics, by a number of potentially scalable, low-cost processes. These thin-film technologies are now entering the first-time manufacturing and commercialization stage. Very high efficiency devices have resulted form crystal growth advances of various III-V compounds, usually in multijunction device configurations where different portions of the solar spectrum are absorbed in successive layers of the device. Ongoing research is continuing to innovate new materials and device concepts which hold the promise of significantly higher solar conversion efficiencies and/or much lower costs. Based on the experience of the past 25 years, the successful development of these concepts will rely on continuing advances in crystal growth and materials science.

3:05 PM Invited
Bulk Ferromagnetic Glasses for Use in Energy Conversion Devices: *Ricardo B. Schwarz*[1]; T. D. Shen[1]; Ulrich Harms[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MST Div., MS G755, Los Alamos, NM 87545 USA

The efficiency of transformers and motors can be increased significantly by making the cores from amorphous (glassy) ferromagnetic materials rather than crystalline Fe-Si alloys. Thin ferromagnetic foils, typically 30-50 microns thick, have been commercially available since the 1980s but their use has been limited by the thinnest of gauge, post-anneal brittleness, and stress sensitivity. The recent development of bulk ferromagnetic glasses solves some of these problems. We discuss the synthesis and magnetic properties of bulk ferromagnetic glasses of the type Fe-(Co,Cr,Mo,Ga,Sb)-P-B-C. The new bulk ferromagnetic glasses have a large supercooled region, T_x-T_g, ranging from 35 to 61 K, within which the glass can be shaped under a relatively small applied load. These glasses have coercivity as low as 0.004 Oe and hysteresis losses at 50 Hz of 0.04 W/kg.

3:35 PM Invited
Advanced Thermoelectric Materials and Devices for Energy Conversion: *Jeff Snyder*[1]; Thierry Caillat[1]; Jean-Pierre Fleurial[1]; [1]Jet Propulsion Lab, JPL-Caltech 277-207, Pasadena, CA 91109-8099 USA

This talk will focus on the recent advances made in thermoelectric materials and devices, particularly at JPL, with an emphasis on power generation. Thermoelectric materials convert heat directly into electrical power. New thermoelectric materials, particularly the skutterudites and zinc antimonide developed at JPL, have higher figure of merit and therefore higher conversion efficiency than state of the art materials. These materials, when combined into a segmented thermocouple can achieve 15% conversion efficiency from temperature gradients found in automobile exhaust. New concepts for power generation utilizing automotive or other sources of waste heat, or radioisotope heat sources in space, will be discussed.

4:00 PM Break

4:10 PM
Thermoelectric Properties of $IrSb_3$ and Related Ternary Skutterudite System: *Sung Wng Kim*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., Interdisciplinary Grad. Sch. of Sci. & Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan

The binary antimonide compounds with the skutterudite structure have a potential for thermoelectric applications because of the high carrier mobility, high thermoelectric power, and relatively low thermal conductivity due to the complex crystal structure. However, the room temperature thermal conductivity of these skutterudite materials is as high as about 10W/mK with an estimated 80-90% contribution from the lattice thermal conductivity. Because the lattice thermal conductivity of related ternary skutterudite compounds can be substantially reduced due to the additional phonon scattering, a study of thermoelectric properties of these compounds is of interest. The binary $IrSb_3$, ternary $Ir_{1-X}Ru_XSb_3$, and $Ir(M_XSb_{1-X})_3$(M=Ge, Sn) compounds were prepared by hot pressing of pre-reacted powders. We have measured the electrical resistivity, thermoelectric power, and thermal conductivity, in a temperature range from room temperature to 973K. The binary $IrSb_3$ compound is found to have P-type electrical properties exhibiting a good Seebeck coefficient(up to 225μV/K around 773K), electrical resistivity($5*10^{-5}$Ohm M around 933K), and relatively low thermal conductivity(6W/mK around 950K). The lattice thermal conductivity, k_{lat} was calculated from the Wiedemann-Franz relationship.

4:35 PM

Thermal Stability of Ionic Liquids: *Zhijing Zhang*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metlgcl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487 USA

Long-term thermal stability of ionic liquids is essential for reliable performance of ionic liquids in any engineering applications. TGA studies were carried out on a series of ionic liquids with different cations and anions such as 1-butyl-3-methylimidazolium chloride ($[C_4mim]Cl$), 1-hexyl-3-methylimidazolium chloride ($[C_6mim]Cl$), 1-hexyl-3-methylimidazolium hexafluorophosphate ($[C_6mim]PF_6$), 1-octyl-3-methylimidazolium hexafluorophosphate ($[C_8mim]PF_6$), and 1-butyl-3-methylimidazolium bis (trifluromethanesulflonyl) imide ($[C_4mim][Tf_2N]$). The short-term thermal stability of ionic liquids was evaluated by measuring the onset temperature of decomposition of ionic liquids, and the long-term thermal stability of ionic liquids was also evaluated by weight loss of ionic liquids over a long period of time at various temperatures. The experimental results showed that the long-term thermal stability of ionic liquids decreased with increasing temperature and was significantly lower than the onset temperature of decomposition of ionic liquids.

5:00 PM

Solid State Thermal Energy Storage in Tris (Hydroxymethyl) Aminomethane, Neopentylglycol and their Solid Solutions: John Hansen[1]; *Dhanesh Chandra*[2]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA; [2]University of Nevada-Reno, Chem. & Metall. Eng., Mackey Sch. of Mines, Reno, NV 89557-0047 USA

A temperature-composition equilibrium phase diagram of the tris (hydroxymethyl) aminomethane (Tris)-neopentylglycol(NPG) binary system is developed to obtain various thermal energy storage materials with different transition temperatures. There are several space-related and other applications for this type of isothermal energy storage system. These organic crystalline materials undergo solid-solid phase transitions, and this property is used to store energy. The low temperature crystal structure of pure Tris is orthorhombic (α phase) and that of pure NPG is monoclinic (β phase). The high temperature "Plastic" (γ or γ') phases are cubic. The energy is stored in the γ or γ' phases primarily due to molecular motion of the O-H...O bonds. Guinier X-ray diffraction and differential scanning calorimetric data showed that there is virtually no solubility of Tris in NPG or visa-versa, below~43°C. A peritectic was observed at 150°C (L + γ —> γ') and also a eutectoid at 127°C (γ —> α + γ'). It is interesting to note that in certain regions of the phase diagrams, the energy is stored in two (γ + γ') plastic phases. Thermodynamic and crystal structure of solid solutions and components will be discussed.

5:20 PM

Phase Equilibria in Thermal Energy Storage Materials: Pentaerythritol and 2-Amino-2-Methyl-1,3-Propanediol: Renee Russell[1]; *Dhanesh Chandra*[2]; Wen-Ming Chien[2]; [1]Pacific Northwest National Laboratory, PO Box 999, MS K6-24, Richland, WA 99352 USA; [2]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

Organic molecular plastic crystals such as polyalcohols and amines reversibly store large amounts of thermal energy during solid-state phase transitions, which have many applications. A binary phase diagram for two such energetic plastic crystals, Pentaerythritol (PE) and 2-amino-2-methyl-l,3- propanediol (AMPL) is proposed. The crystal structure of AMPL was determined by single crystal method. This diagram was determined by high temperature Guinier x-ray diffraction and differential scanning calorimetric methods. The phase diagram in this study is rather complex with two eutectoids at 84°C and 147°C and a peritectic at 184°C. The low temperature phases of PE and AMPL are in equilibrium between 20°C and 84°C in the composition range of 12 to 98 mol% AMPL. The solubility of AMFL in PE is very high, up to 45 mol% AMFL in PE at 147°C. The solubility of PE in AMFL is very low, only up to 10 mol% PE in AMFL at 84°C. The structural and thermodynamic results, along with the amount of energy stored, will be presented.

Fundamentals of Structural Intermetallics: Nb and Mo Based Silicides

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

Monday PM — Room: 615-616
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Ian Baker, Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA; John Perepezko, University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

2:00 PM Invited

A Review of Very High-Temperature Intermetallic Based Composites: *Bernard P. Bewlay*[1]; Melvin R. Jackson[1]; J.- C. Zhao[1]; P. R. Subramanian[1]; [1]GE-CRD, PO Box 8, Schenectady, NY USA

This paper will review recent progress in the development of intermetallic based composites for very high temperature applications. Emphasis will be given to Nb-silicide based in-situ composites, but there will also be discussion of systems based on other intermetallics, such as Laves phases and Mo-based silicides. Nb-silicide based in-situ composites contain high-strength silicides that are toughened by a ductile Nb-based solid solution. Simple composites are based on binary Nb-Si alloys, more complex systems are alloyed with Ti, Hf, Cr and Al. In higher order silicide-based systems, alloying elements have been added to stabilize intermetallics, such as Laves phases and boron rich T-2 phases, for additional oxidation resistance. In general, alloying schemes have been developed to achieve an outstanding balance of room temperature toughness, high temperature creep performance, and oxidation resistance. This paper will review recent progress in the development of composite processing-structure-property relatio nships in Nb-silicide based in-situ composites and the methodologies that have been employed for Nb-silicide composite property optimization. The Nb-silicide composite properties will be compared with those of advanced Ni-based superalloys, and other intermetallic-based systems.

2:30 PM Invited

Processing, Microstructures and Properties of Nb-Base and Mo-Base Metal/Silicide Multiphase Alloys: *Madan G. Mendiratta*[1]; Sarath K. Menon[1]; Young-Won Kim[1]; [1]Air Force Research Laboratory (AFRL), MLLM, UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Mo + Mo_5SiB_2+ Mo_3Si and Nb + Nb_5Si_3 + Cr_2Nb systems are being developed for high temperature structural applications. These alloys are being processed using different methods. The Mo-based alloys are being made using powder metallurgy approach; the prealloyed powders are consolidated by cold isostatic processing + sintering in H_2 atmosphere + hot isostatic pressing + hot-extrusion. Prealloyed powders made by plasma rotating electrode process are being consolidated by hot-extrusion. The Nb-based alloys are being made by induction skull melting and casting into ingots and thin plates. The cast ingots are further processed by hot-extrusion. The microstructures at various stages of processing and heat treating are being characterized. Tensile properties and fracture toughness will be determined, as a function of temperature and failure mechanisms will be correlated with microstructures.

3:00 PM
Computational Design and Property Optimization for Niobium-Based In-Situ Composites: *Kwai S. Chan*[1]; David L. Davidson[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA

Niobium-based in-situ composites show potential for high-temperature structural applications, but they need simultaneous improvements in oxidation, creep, and fracture resistance. This paper describes the development of a material-science-based methodology that aids the design of alloy composition and microstructure of in-situ composites for optimized properties of oxidation resistance, yield strength, creep strength, and fracture resistance. Computational and analytical models are developed for describing properties of interest in terms of composition, microstructure, and properties of constituent phases in the in-situ composites. Using the property goals as the constraints, the range of constituent properties, volume fractions, and microstructures that would satisfy the imposed constraints are computed. Application of the proposed methodology to optimizing the oxidation, creep, and fracture resistance of Nb-based in-situ composites is presented and the results are compared against available experimental data in the literature. Work supported by the US Air Force Office of Scientific Research through Contract No. F49620-01-C-0016, Dr. Craig S. Hartley, Program Monitor.

3:30 PM Invited
High Temperature Properties of Laves Phase Two Phase Alloys: Dan J. Thoma[1]; *Robert J. Hanrahan*[1]; John D. Wittenberger[2]; [1]Los Alamos National Laboratory, TA 3, MS G770, Los Alamos, NM 87544 USA; [2]NASA, Glenn Rsrch. Ctr., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

An alloy design strategy has been developed and used to explore the utilization of Laves phases for high-temperature structural applications. NbCr2 was chosen as the base intermetallic constituent based upon existing high-temperature strength properties. Titanium was chosen as an effective addition to broaden the single-phase range of homogeneity and to optimize the deformation behavior of the NbCr2. Dual phase alloys consisting of a Nb/Ti bcc phase and the C15 (Nb,Ti)Cr2 phase were processed. The C15 phase, Nb20Ti20Cr60, had the optimum combination of properties in the ternary single-phase region, and all two-phase alloys examined were on the same tie-line with Nb20Ti20Cr60. 23Nb-34Ti-42Cr (at.%), has a maximum flow stress over 1GPa up to 1000°C, and accommodates compressive strains over 20% from room temperature to 1200°C. Additional studies were conducted of the oxidation and high temperature creep of the same alloys at 2E-4 to 2E-8/s and 900 to 1400 K in air.

4:00 PM
Microstructure Evolution, Thermal Stability and their Effect of Properties in a Multicomponent Nb-Si-X Alloy: *Young-Won Kim*[1]; Dennis M. Dimiduk[2]; Madan Mendiratta[1]; [1]UES, Inc., Matls. & Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433-7817 USA

The microstructural evolution and thermal stability was investigated for a multicomponent, hypereutectic NbSi-X alloy, 45.5Nb-22Ti-2Hf-7Cr-20Si-2Al-1.5Sn (at%), was investigated for. Alloy material was produced in cast-plate forms using the induction skull melting technique (ISM). The cast material consisted of large-size, primary silicides (Nb_5Si_3 type) which were surrounded by eutectic-structure regions. The eutectic structure was a mixture of relatively fine secondary silicides (Nb_5Si_3 type) and dendritic beta phase having small amounts of interdendritic Laves-phase particles. When annealed at 1400°C for 50h and then HIP'ed at 1450°C for 6h, the material showed significant microstructure changes; that is, transformation of the eutectic structure to a fine mixture of slicides and beta grains, partial decomposition of large Nb_5Si_3 type silicides to Ti_5Si_3 type silicides, and fine decoration of Laves phase particles along beta-grain boundaries. This process resulted in fairly uniform, refined microstructures. These microstructures have been tested for their thermal instability and/or phase redistribution happening when exposed to temperatures lower than 1400°C, which is near the upper limit of the desired use temperature. Measurements of yield stress and oxidation resistance are underway for selected microstructural forms, and their interrelationships will be analyzed.

4:30 PM
Development of High Temperature Coating Strategies for Mo-Si-B Alloy: J. S. Park[1]; R. Sakidja[1]; *J. H. Perepezko*[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

The development of protective coatings and the oxide scales on Mo-Si-B alloys were examined. Two types of approaches are employed; stable oxides and molybdenum disilicide (MoSi2) phase. The oxide coatings were produced through spray coatings, whereas the disilicide deposits were synthesized through the pack cementation process. Following oxidation in air at 1000-1200C for times up to 100 hrs, the substrate formed oxides composed of borosilicate, MoO2 and the metalloid-depleted zone with silica dispersions. With the oxide coatings, due to constricted oxygen diffusion, improved oxidation resistance was observed as reflected by the reduced thickness of the layered oxides. With disilicide coatings on the other hand, no layered oxides were observed. Instead, limited layered structures composed of Mo5Si3 and borosilicide phases were observed between disilicide coating and the substrate. The growth kinetics of layered structures and the oxides are presented. The support of ONR (N00014-92-J-1554) and AFOSR (F49620-00-1-0077) is gratefully acknowledged.

4:50 PM Invited
Cyclic Fatigue-Crack Growth and Fracture Behavior of Mo-Mo3Si-Mo5SiB2 Silicides at Ambient and Elevated Temperatures: *Heeman Choe*[1]; Joachim H. Schneibel[2]; Robert O. Ritchie[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., 463 Evans Hall, #1760, Berkeley, CA 94720-1760 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA

The quest for structural materials that can operate at higher and higher temperatures remains a persistent challenge in materials science. Among the potential candidates for higher-temperature advanced engine systems are multiphase Mo-Si-B intermetallics due to their high melting temperature (> 2000°C) and relatively good oxidation resistance. In this study, we investigate the damage-tolerant properties, i.e., fracture toughness and fatigue-crack propagation behavior, of several such multiphase Mo-Si-B intermetallics. Specifically, the alloys Mo-12Si-8.5B, Mo-16.8Si-8.4B, and Mo-10Nb-12Si-8.5B (at.%), consisting of alpha-Mo, Mo3Si, and Mo5SiB2 (T2) as their primary phases, are examined at temperatures from ambient to 1300°C, with the objective of discerning salient toughening mechanisms. The alloy with 16.8 at.% Si contains less alpha-Mo than that with 12 at.% Si. It is more brittle, but at the same time more oxidation resistant. It is found that Mo-12Si-8.5B (at.%) alloy in particular displays relatively high intrinsic (crack-initiation) toughness both at ambient and elevated temperatures, which is attributed to a crack-trapping mechanism at coarse alpha-Mo particles. Moreover, both fracture toughness and fatigue-crack growth properties in this alloy actually improve as temperature increases up to 1300°C. This is principally due to the enhanced ductility of alpha-Mo phase at elevated temperatures, which promotes some degree of extrinsic toughening, specifically crack bridging, which in turn results in rising resistance-curve behavior at 1300°C. This work was sponsored by the US Department of Energy, through the Office of Science, Office of Basic Energy Sciences, Materials Sciences Division of the US Department of Energy under Contract No. DE-AC03-76SF00098 (for HC and ROR), and the Office of Fossil Energy, Advanced Research Materials (ARM) Program, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed by UT-Battelle, LLC (for JHS).

5:20 PM
High Temperature Mechanical Behavior of A15 Mo_3Si Single Crystals: *Isai Rosales*[1]; Joachim H. Schneibel[1]; Lee Heatherly[1]; Lorenzo Martinez[2]; Bernardo F. Campillo[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]National University of Mexico, Fac. of Chem., Mexico D.F. Mexico

Mo_3Si single crystals were grown in an optical floating zone furnace, specimens with <111>, <110> and <100> directions were fabricated, and high temperature compression test were performed at 1400°C in vacuum (10-6 Torr). High stress values ranging from 600 to 1100 MPa were observed. Depending on the strain rate and stress, localized recrystallization occurred. Analysis of the slip traces on the surfaces of the deformed specimens was carried out in order to identify the active slip planes, which are {010} for the specimens with <110>, and {012} for the specimens with <111> compression axis. No slip traces were observed for the <100> orientation confirming that the resolved shear stress was zero. TEM study of the slip planes is in progress. Differences in cracking behavior were observed after conical indentation with a load of 0.5 kg. The preferential cleavage planes were {211} for the specimens indented on (111) and (110) surfaces, and {110} for the (110) surface. This work was sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory managed by UT-Battelle, LLC.

5:40 PM
Reducing the Thermal Expansion Anisotropy of Mo_5Si_3-Based Molybdenum Silicides: *Joachim H. Schneibel*[1]; Chong Long Fu[1]; Claudia J. Rawn[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831 USA

The intermetallic phase Mo_5Si_3 is of interest because of to its high melting point (2180°C) and potential oxidation resistance. However, its coefficient of thermal expansion (CTE) is highly anisotropic. Theoretical considerations suggest that this anisotropy can be reduced by stretching the Mo-chains in the c-direction of the tetragonal crystal structure, or by partial substitution of the chain atoms with smaller atoms. High temperature x-ray diffraction experiments confirm indeed that additions of atoms which are larger or smaller, respectively, than Mo reduce the CTE anisotropy from a value of 2 for Mo5Si3 to values as low as 1.3 for ternary $(Mo,Nb)_5Si_3$ compounds. The results are discussed in terms of the site occupation of the ternary alloying elements and the underlying electronic structure. The lower anisotropy is accompanied by a dramatic reduction in the density of microcracks in the material. This work was sponsored by (a) the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy, and (b) the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program. ORNL is operated by UT-Battelle, LLC, for the US Department of Energy under contract DE-AC05-00OR22725.

General Abstracts: Nickel and Molybdenum

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

Monday PM — Room: 209
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Carl Cady, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
Double Serpentine Diffusion Paths in Ternary Fe-Ni-Al System: *Mysore A. Dayananda*[1]; Junghan Kim[1]; [1]Purdue University, Sch. of Matls. Eng., MSEE Bldg., W. Lafayette, IN 47907 USA

Solid-solid diffusion couples assembled with (bcc) Fe-Ni-Al alloys were investigated for the development of uncommon diffusion paths. Selected couples were isothermally annealed at 1000°C for 2 days and analyzed for concentration profiles by electron microprobe analysis. Profiles of interdiffusion fluxes were determined and the couples were examined for the development of zero-flux planes and regions of up-hill interdiffusion for the individual components. Double-serpentine diffusion paths with two crossings of the straight line joining the terminal alloy compositions on the Fe-Ni-Al isotherm were observed for several couples. These unusual paths were characterized by the development of separate regions of up-hill interdiffusion on both sides of the Matano plane for at least one component. Diffusional interactions among the components were assessed at the compositions of the zero-flux planes developed by the various components. The paths are discussed in the light of interdiffusion and thermodynamic data for Fe-Ni-Al alloys.

2:25 PM
Ternary Interdiffusion and Accumulation of Al in Columnar-Grained $MoSi_2$: *Edward J. Ciecko*[1]; Mysore A. Dayananda[1]; [1]Purdue University, Matls. Sci. & Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA

Materials characterized by columnar structures (long, narrow grains) differ from those characterized by large, equiaxed grains because of the abundance of grain boundaries and the presence of relatively large number of triple junctions. Interdiffusion in such materials was investigated by means of a vapor-solid diffusion couple involving columnar-grained $MoSi_2$ in contact with Al vapor at 1000°C. The formation of the hexagonal (C40 type) phase $Mo(Si,Al)_2$ was observed. In addition, the development of relative maxima in the Al concentration profiles was observed to occur in the columnar-grained $MoSi_2$, indicating an accumulation of Al within the diffusion zone. The relative accumulation of Al is examined on the basis of the phenomenology of bulk ternary diffusion and is discussed in terms of diffusional contributions from grain boundaries and triple junctions.

2:50 PM
Pre-Precipitates in Ni-Rich Ni-Ti Alloys: *Gernot Kostorz*[1]; Michael Kompatscher[1]; Bernd Schoenfeld[1]; [1]ETH Zurich, Angewandte Physik, Zurich CH 8093 Switzerland

The stable precipitate phase forming in Ni-rich f.c.c. Ni-Ti solid solutions is the hexagonal η-phase, but metastable coherent precipitates are found at intermediate temperatures. In-situ small-angle scattering on single crystals of Ni-(10-12) at.% Ti show a complex sequence of decomposition for ageing temperatures around 600°C. An initial stage with precipitates containing about 17 at.% Ti is followed by an increase of the Ti content in the particles to about 22 at.% Ti. These conclusions stem primarily from the variation of the integrated small-angle scattering intensity as a function of ageing time. Further confirmation is obtained from transmission electron microscopy and diffuse scattering near the fundamental Bragg peaks and the superstructure peaks of the precipitates ($L1_2$ structure). A comprehensive view of the various stages of decomposition, the degree of order, the shapes and interfacial details of the precipitates, and the coherency strains due to elastic mismatch between particles and matrix emerges.

3:15 PM Break

3:40 PM
Fracture Toughness and Tensile Properties in Nanocrystalline Nickel Thin Sheets: Reza A. Mirshams[1]; *Parviz S. Razi*[2]; C. H. Xiao[2]; [1]University of North Texas, Eng., Denton, TX 76203 USA; [2]Southern University and A&M College, Mechl. Eng., Baton Rouge, LA 70813 USA

The potential engineering applications of nanocrystalline materials need more detailed study on deformation and fracture mechanisms at room and elevated temperatures under tensile loading. This paper reports results of a series of experiments carried out on nickel and carbon doped nanocrystalline nickel with different carbon concentrations from 500 to 1000 ppm at room temperature to 300°C. Grain growth was observed in nanocrystalline nickels as the testing temperature increases. A fast grain growth was noticed at 300°C. Pure nanocrystalline nickel experienced an abnormal grain growth at 500°C and its tensile properties reduced to a very low level. The addition of carbon exerted a potential effect to enhance the stability of the microstructure in nanocrystalline nickel at intermediate temperatures. However, carbon doped nickels exhibited lower tensile properties. Nanocrystalline nickels displayed a conventional Hall-Petch relationship. The results are discussed in relation to microstructural characteristics by using TEM and SEM.

4:05 PM
Measuring the Fracture Toughness of TZM and ODS Molybdenum Alloys using Standard and Sub-Sized Specimens: *Brian V. Cockeram*[1]; [1]Bechtel-Bettis, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

Oxide Dispersion Strengthened (ODS) and TZM molybdenum have excellent creep resistance and strength at high temperatures in inert atmospheres. The fracture toughness and transition temperature for brittle behavior must be known to evaluate the use of molybdenum for structural applications. Fracture toughness (Kic) testing in accordance with ASTM E399 methods was performed over a range of temperatures to characterize 0.25" thick plate material of ODS and TZM molybdenum. The transition temperature for brittle behavior of ODS molybdenum was below room-temperature. The transition temperature for TZM molybdenum in the longitudinal direction was slightly above room-temperature, while the transition temperature for the transverse direction was 150C. The use of sub-sized specimen geometries produced fracture toughness results that were comparable to standard-sized specimens.

4:30 PM
Molecular Dynamics Simulation and Experimental Proof of Hydrogen-Enhanced Dislocation Emission in Nickel: *Z. J. Li*[1]; J. X. Li[1]; H. Liu[1]; L. J. Qiao[1]; W. Y. Chu[1]; [1]University of Science and Technology-Beijing, Dept. of Matls. Phys., Beijing 100083 China

The molecular dynamic simulations indicated that hydrogen solubilized in nickel decreased the critical stress intensity for dislocation emission from $K_{Ie}(\theta = 45^0)$=1.0MPam$^{1/2}$ or $K_{Ie}(\theta = 70°)$=0.82MPam$^{1/2}$ to $K_{Ie}(\theta = 45°)$=0.9MPam$^{1/2}$ or $K_{Ie}(\theta = 70°)$=0.7MPam$^{1/2}$, respectively. Therefore, hydrogen could enhance dislocation emission. On the other hand, hydrogen decreased critical stress intensity for cleavage of a Griffith crack along the only slop plane in the quasi three-dimension model from $K_{IG}(\theta = 0°)$=1.03MPam$^{1/2}$ to $K_I{}^*{}_G(\theta = 0°)$=0.93MPam$^{1/2}$ and

MONDAY PM

then the surface energy from γ_{111}=2.2J/m^2 to γ_{111}=1.78J/m^2, resulting in facilitating dislocation emission. A constant deflection device designed for use within a transmission electron microscope (TEM) was used to study the change in dislocation configuration ahead of a loaded crack tip before and after charging with hydrogen. In situ observation in TEM showed that hydrogen enhanced dislocation emission and motion before initiation of hydrogen-induced crack.

General Abstracts: Aluminum and Titanium

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

Monday PM — Room: 310
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: George T. Gray, III, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
Deformation Microstructure Evolution in Single Crystal Aluminum Alloys: *Michael F. Savage*[1]; Donald E. Kramer[1]; Lyle E. Levine[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

Localized deformation and the formation of complex dislocation structures play a significant role in the room temperature flow behavior of high stacking fault energy FCC metals. In this study, the slip line evolution and underlying deformation microstructures of high purity single crystal Al and Al solid solution alloys have been investigated. Atomic force microscopy investigations of surface slip bands were performed during deformation of Al single crystals with an evolved dislocation structure. These results have been compared with the deformation microstructures observed in thin foils ex-situ, by Transmission Electron Microscopy. Relationships between the observed surface structures and the presence of Geometrically Necessary Boundaries (GNB's) and Incidental Dislocation Boundaries (IDB's) will be discussed. Effects of crystallographic orientation on the slip line evolution and the nature of these dislocation structures will also be presented. The impact of these results will be discussed with respect to an ongoing modeling effort of plasticity in dislocation cell-forming FCC metals.

2:25 PM
Fatigue Crack Growth in Al-7.5Mg Bulk Nanocrystalline Materials: *Peter S. Pao*[1]; Steve J. Gill[1]; Harry N. Jones[1]; Jerry C. Feng[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA

The fatigue crack growth of bulk nanocrystalline Al-7.5Mg was investigated. Nanocrystalline particulates were prepared by cryo-milling. These particulates were then compacted and extruded into rods. The fatigue crack growth rates of nanocrystalline Al-7.5Mg are significantly higher than those of ingot 7050-T7451. The fatigue crack growth thresholds of the nano alloys are significantly lower than that of 7050-T7451. Post-fatigue SEM investigation has revealed that the fracture surfaces of nano alloys are very smooth and the higher fatigue crack growth rates and lower thresholds may be attributed to the much smoother fracture surface morphology and lower roughness-induced crack closure in nano alloys. The fracture toughness of the nanocrystalline Al-7.5Mg is significantly lower than that of 7050-T7451. The reason for the lower fracture toughness in the bulk nano alloys is not understood yet but may be related to the lack of dislocation activities for energy dispersal in the fracture processes.

2:50 PM
Evaluation of the StressWave Fatigue Life Enhancement Process for Fastener Holes in Aluminum Alloys: *Brian D. Flinn*[1]; Christopher Meyer[1]; Eric T. Easterbrook[2]; [1]University of Washington, Matls. Sci. & Eng., Box 352120, Seattle, WA 98195 USA; [2]StressWave, Inc., 12932 S.E. Kent-Kangley Rd., MS 528, Kent, WA 98031 USA

The fatigue life improvement of the StressWave process was compared with existing split sleeve cold expansion technology and untreated specimens using aluminum 2024-T3 specimens 0.190" (4.82 mm) and 0.063" (1.6mm) thick. The StressWave method (patents pending) overcomes many of the limitations of the split-sleeve and split-mandrel cold working processes. Constant amplitude fatigue tests were conducted on zero load transfer, open hole specimens to evaluate the performance of StressWave-processed holes relative to holes treated with other methods. StressWave produces residual compressive stresses and fatigue performance comparable to, or better than, those produced by the existing processes.

3:15 PM
Fatigue Life Improvement of Fastener Holes in Titanium with the StressWave Process: *Brian D. Flinn*[1]; Scott Llitteras[1]; Milton Siegelmann[2]; Eric T. Easterbrook[2]; [1]University of Washington, Matls. Sci. & Eng., Box 352120, Seattle, WA 98195 USA; [2]StressWave, Inc., 12932 S.E. Kent-Kangley Rd., MS 528, Kent, WA 98031 USA

The fatigue life of titanium 6-4 open hole specimens was measured after treatment with the StressWave™ fatigue life enhancement process. The StressWave™ process induces beneficial compressive residual stresses in the material surrounding the fastener hole via plastic deformation prior to machining the hole. Specimens were tested at three StressWave™ process conditions. Fatigue life, crack initiation and growth are compared with untreated specimens. The StressWave™ process increased the fatigue life by four to ten times that of untreated holes. This life improvement is comparable to that of split-sleeve and split mandrel cold expansion processes.

3:40 PM Break

4:05 PM
Aluminum Titanate Formation and Densification under an External Electric Field: Influence of Precursor Powders: *Lia Stanciu*[1]; Joanna R. Groza[1]; Vladimir Kodash[1]; Carmen Plapcianu[2]; [1]University of California at Davis, Dept. of Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]National Institute for Materials Physics, Str. Atomistilor, 105 bis PO Box MG-776900, Bucharest 776900 Romania

Present work reports the results obtained by FAST (Field Activated Sintering Technique) sintering of three types of powders: cogelified alumina-titania powders, mechanical mixtures of individual alumina and titania gel powders, and alumina-titania precipitates obtained by a flotation method. The use of sol-gel cogelified alumina-titania powders led to a 98% formation of aluminum titanate, high density, and a grain size of 1-2 microns, at 1100°C. Mechanical mixture of individual alumina and titania powders, as well as the powders obtained by flotation method resulted in less aluminum titanate at this temperature and lower density. The sample obtained by sintering the powders obtained by flotation method has also a larger grain size, of about 5 microns. It has been concluded that sol-gel cogelified powders offer the best results in terms of grain size and composition of final ceramic due to the high homogeneity and initial nanometric particle size.

4:30 PM
Fatigue Behavior of Ti-64 Forged Alloy at Ambient and Elevated Temperatures: *Chih-An Yin*[1]; Malcolm C. Thomas[1]; [1]Rolls Royce Corporation, Matls. & Proc. Dept., PO Box 420, MC S-52, 2001 S. Tibbs Ave., Indianapolis, IN 46241 USA

Designing against fatigue failure involves so many uncertainties that it is usually necessary to test full-scale structures prior to service to prove their reliability. Such tests should be simple, short and inexpensive. At the same time, it is necessary to insure that the fatigue response of the laboratory test reflects the actual performance that can be expected of the structure in its service environment. For this reason, the material characterization testing for tensile, low cycle fatigue and fatigue crack growth responses were conducted on Ti64 forged alloy subjected to a specified heat treatment. Strain based mechanical hysteresis concepts have been used to establish cyclic stress strain behavior and fatigue life characteristics of the present material at ambient and elevated temperatures. The stress intensity factor concept of fracture mechanics has been used to establish the fatigue crack growth rate data at ambient and elevated temperatures. These selected test temperatures are corresponding to the conditions of normal operation in different stages of compressors for some aircraft engines. The analysis of the experimental results are based mainly on the phenomenological approach and predicted methods. So that a quantitative evaluation can be used to assess the effect of test temperatures on the cyclic deformation behavior, tensile mean stress relaxation resulted from the positive mean strains, and its effect of fatigue damage. In addition, the measured notch fatigue lives and lives predicted from smooth specimen fatigue data are illustrated based on Neuber's rule.

4:55 PM
Low Temperature Superplasticity of Ultrafine Grained 5083 Al Alloy: *Kyung-Tae Park*[1]; Deok-Young Hwang[1]; Ki-Seung Lee[2]; Dong Hyuk Shin[2]; [1]Hanbat National University, Div. of Adv. Matls. Sci. & Eng., San 16-1, Dukmyung-Dong, Yuseong-Gu, Taejon 305-719 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., 1271 Sa 1-Dong, Ansan, Kyunggi-Do 425-791 S. Korea

A ultrafine grained structure was introduced in a 5083 Al alloy by imposing an effective strain of 8 through equal channel angular pressing (ECAP). Tensile tests were performed on the as-ECAPed samples in the strain rate range of 10-5x10-2 s-1 at temperatures of 498-548 K. In order to examine low temperature superplastic behavior of the alloy. The maximum elongation of 315 % was obtained at 548 K and 5x10-4 s-1. In addition, the analysis of the mechanical data indicated that low temperature superplasticity of the present ultrafine grained alloy was attributed to grain boundary sliding that is rate-controlled by grain boundary diffusion with low activation energy associated with non-equilibrium grain boundaries. Necking instability during low temperature superplastic deformation of the alloy was also discussed by applying the Hart's necking instability criterion.

General Abstracts: Ferrous Materials: Carbon and Low-Alloy 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

Monday PM — Room: 211
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Robert Hackenberg, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM
The Effect of Surfactant and Environmental Parameters on Mild Steel Corrosion Inhibition: *Michael L. Free*[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E. Rm. 412, Salt Lake City, UT 84112 USA

The effects of parameters such as solution environment, hydrocarbon chain length, critical micelle concentration, surfactant concentration, and functional group on mild steel corrosion were evaluated by electrochemical testing. The results from the electrochemical testing were utilized together with theoretical considerations to develop a mathematical model for the prediction of mild steel corrosion inhibition using a variety of surfactants.

2:25 PM
Effects of Microstructure and Hardness on Machinability of 0.6% Carbon Steels for Machine Structural Use: *Yasuhiro Omori*[1]; Toshiyuki Hoshino[1]; Akihiro Matsuzaki[1]; Keniti Amano[1]; [1]Kawasaki Steel Corporation, Techl. Rsrch. Labs., Kawasakidori 1-Chome, Mizushima, Kurashiki, Okayama 712-8511 Japan

In order to improve the machinability of a 0.6%C steel in terms of the fatigue strength without using free cutting additives such as Pb, effects of both microstructure and hardness on machinability were investigated. Though the tool life measured by turning test was approximately controlled by hardness of steel, the tool life of bainitic steel was longer than that of the pearlite-ferrite steel, even though the hardness was the same. The tool life, on the other hand, had a good correlation with cutting force independently of microstructure of steel. Since coarse planar carbides which had random orientation in bainite played a big role as origins of stress concentration, shear angle increased and thus the cutting force decreased. The decrease in the cutting force also decreased tool temperature during cutting, which improved the tool life by preventing Co in the tool from diffusing into chips.

2:50 PM
Decomposition of Retained Austenite during the Coiling Process of Hot Rolled TRIP Steels: *Hyun Jo Jun*[1]; Sung Ho Park[2]; Chan Gyung Park[1]; [1]Pohang University of Science & Technology, Matls. Sci. & Eng., Hyojadong Namgu san 31, Pohang, Kyungbuk 790-784 Korea; [2]Technical Research Laboratory, POSCO, Pohang, Kyungbuk Korea

A good combination of strength and formability of TRIP steels is mainly attributed to the retained austenite transforming into martensite during the plastic deformation. However, the retained austenite in hot rolled sheet steels can be decomposed into other phases during the coiling process, which can produce serious inhomogeneity of the TRIP sheet steels. In order to evaluate this decomposing behavior of the retained austenite, the XRD and TEM in-situ heating observations have been made in the hot-rolled C-Mn-Si TRIP steels. It was found that the retained austenite was decomposed into various phases depending on the coiling temperatures ranging from 350 to 500C: carbide-free bainitic ferrite, ferrite associated with iron carbide and pearlite. The difference of these decomposition behaviors will be discussed in terms of the variation of carbon concentration and thermal stability of the retained austenite at high temperatures.

3:15 PM
Fracture Toughness Analysis in Transition Temperature Region of Mn-Mo-Ni Low Alloy Steels: *Sangho Kim*[1]; Sunghak Lee[1]; Bong Sang Lee[2]; Yong Jun Oh[2]; [1]POSTECH, Dept. of Matl. Sci. & Eng., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea; [2]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. Dvlp. Team, 150 Dukjin-dong, Yuseong-ku, Taejon 305-353 Korea

An investigation was conducted into the effect of the microstructures on fracture toughness in the ductile-brittle transition temperature region of various Mn-Mo-Ni high strength low alloy steels. Microstructures were analyzed using OM, SEM, and TEM. In order to evaluate the fracture toughness in the transition range, reference temperatures were determined according to ASTM E1921-97 standard test method. Fracture toughness in the transition region was interpreted by metallurgical factors such as yield strength, carbide size distribution, local fracture stress. It is shown that the critical nearest-neighbor distance between coarse carbides was an important microstructural factor affecting elastic-plastic fracture toughness, since it satisfied a linear relationship with the critical distance between a crack tip to a cleavage initiation site. These findings suggested that reducing the total number of carbides and M3C carbide fraction was useful to improve fracture toughness in the transition region.

3:40 PM
Change in the Precipitation Behavior of MnS with Ti Addition in Mn/Si/Ti Deoxidized Steels: *Han Soo Kim*[1]; Hae-Geon Lee[1]; [1]Pohang University of Science and Technology, Matls. Sci. & Metlgcl. Eng., San 31, Hyoja-dong, Pohang, Kyungbuk 790-784 Korea

Precipitation behavior of MnS on oxide inclusions was studied during solidification of Mn/Si/Ti deoxidized steels. The morphology of MnS precipitates accompanying oxides was varied with Ti addition, which might be attributed to the change in composition of oxide inclusions. Thermodynamic calculations were carried out to predict the change in the liquidus temperature of oxide inclusions with varying composition using computational thermodynamic method. Morphological change of MnS precipitates accompanying oxide inclusions could be interpreted using variation of liquidus temperature of oxide inclusions. Formation of Mn-depleted zone around oxide inclusions was also investigated. The change in the formation of Mn-depletion zone with Ti addition in the vicinity of oxide inclusions accompanying MnS could be explained with the help of understanding MnS precipitation behavior.

High Performance Metallic Materials for Cost Sensitive Applications: Lightweight Metals

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

Monday PM — Room: 213
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Eric Taleff, The University of Texas at Austin, Austin, TX 78705 USA; Theodore Reinhart, Boeing Military Airplanes, Seattle, WA 98124 USA

2:00 PM Invited
Weld Rework of Alloy D357 Castings: *Theodore L. Reinhart*[1]; Fred J. Feiertag[2]; [1]Boeing Military Airplanes, PO Box 3707, MS 4E-03,

Seattle, WA 98124 USA; [2]The Boeing Company, PO Box 3707, MC 5L-14, Seattle, WA 98124 USA

Weld rework of aluminum alloy castings is an integral part of the production process despite restrictions to noncritical/nondesignated/low stress regions of aerospace castings. This restriction, however, is a greater issue when larger, higher value parts are considered. Scrapping such a part for a relatively minor defect, one which could otherwise be reworked by welding, carries a painful economic penalty. In fact, the penalty may be sufficiently serious as to preclude consideration in some applications. This presentation covers the results achieved using improved welding procedures. These new procedures yielded results demonstrably better than those achieved using welding practices currently used by the casting industry.

2:30 PM Cancelled

Synthesis of Aluminum Alloys using Semi-Solid Thermal Transformations: *Michael E. Kassner*

2:30 PM

ALE-Finite Element Simulation of U-Shape Aluminum Profile Extrusion: *Zezhong Chen*[1]; Zhongxu Bao[1]; Hesheng Liu[1]; [1]Nanchang University, Sch. of Mechl. & Elect. Eng., Nanchang, 330029 China

Because the coefficient of extrusion (Ratio of billet and profile cross section area) is too large (more than 30), it is almost impossible to simulate aluminum profile extrusion process using Lagrange finite element method. It is hard to get satisfactory result since grid distortion level is exceptional and grid remeshing can not be conduct. On the other hand, Eulerian finite element method is widely used in fluid flow simulation since it is a good way for description of fluid flow. Therefore, Arbitrary Lagrange-Eulerian finite element method is adopted in the paper to simulate the “U” shape profile extrusion process. Lagrange method is used for mould and Eulerian method is used for metal flow. Satisfactory result, such as deformation process, stress and strain distribution, etc., are obtained.

2:50 PM

Spray Forming of Hypereutectic Al-25Si Alloy and Processing Condition for Hot Extrusion: *Woo-Jin Park*[1]; Tae Kown Ha[2]; Joseph Kim[2]; Sangho Ahn[1]; Young Won Chang[2]; [1]Research Institute of Industrial Science and Technology, Matls. & Proc. Rsrch. Ctr., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-600 S. Korea; [2]Pohang University of Science and Technology, Ctr. for Adv. Aeros. Matls., Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea

The fabrication of hypereutectic Al-25Si alloy via spray forming process, which are expected to be applied to the cylinder liner part of the engine block of an automobile due to excellent wear resistance, low density and low thermal expansion coefficient, has been reported in this article. The characterization of the microstructural and the mechanical properties were also carried out. The microstructure of the hypereutectic Al-25Si alloy appeared to consist of Al matrix and equiaxed Si particles of average diameter of 5~7 µm. To obtain the condition for hot extrusion, a necessary process for application as the cylinder liner, a series of load relaxation and compression tests have been conducted at temperatures ranging from RT to 500°C. The strain rate sensitivity parameter (m) of this alloy has been found to be very low (≤ 0.1) below 300°C and reached maximum value of about 0.2 at 500°C. The dynamic material model has been applied to construct the processing map of this alloy. The experimental extrusion has been successfully conducted at the temperatures of 300°C and above with the ratio of area reduction of 28 and 40.

3:10 PM Cancelled

Forming Properties and Mathematical Model of Semi-Solid AlSi9Cu3 Aluminum Alloy for Thixoforming: *Xie Shuisheng*

3:10 PM Invited

Materials for Antiterrorism and Other Critical National Needs: *Toni Grobstein Marechaux*[1]; [1]National Research Council, National Materials Advisory Board, 2101 Constitution Ave. NW, Washington, DC 20418 USA

High-performance and cost-effective materials are needed to address many of the nation’s high-visibility needs. Many technologies to battle terrorism, for example, can only be implemented if they are cost-effective. Such advanced equipment as night-vision goggles currently cost more than most local law-enforcement organizations can afford, and containers for checked baggage that are hardened against explosions are currently too heavy and too costly for widespread use. Other critical needs such as crime prevention, elder care, counterfeit deterrence, public health, education, and environmental protection can all utilize a variety of advanced materials–but only if they perform a highly specific function (or more likely, perform multiple functions) and are reliably and reasonably priced.

3:40 PM Break

3:50 PM

Hot-Formable, Age-Hardenable, Low-Cost Aluminum Alloys: *Eric M. Taleff*[1]; Jun Qiao[1]; [1]The University of Texas at Austin, Mechl. Eng., Matls. Sci. & Eng. Program, 160 E. Dean Keeton St., Austin, TX 78705 USA

A basis has been developed for engineering a new group of aluminum alloys which exhibit high tensile ductility at hot temperatures and good age-hardening at lower temperatures. High tensile ductilities, 150 to 300%, are achieved at high temperatures and low strain rates (ε. /D $\leq 10^{13}$ m^{-2}) with an alloying addition of Mg to Al, which induces solute drag creep. Solute drag creep provides a high strain-rate sensitivity of m = 0.3, which reduces the rate of neck formation and allows high tensile elongations to be achieved. Ternary alloying additions of Zn at concentrations up to 5 wt. pct. remain in solution during hot forming and do not significantly interfere with the increased ductilities from solute-drag creep. The ternary Zn additions provide significant age hardening at temperatures well below those associated with hot forming and yield a significant paint-bake response. Avoidance of the need for ultra-fine grain sizes is expected to keep the cost of these alloys low.

4:10 PM

Light Magnesium Constructions for Transportation Applications: *Adi Ben-Artzy*[1]; Illan Makover[1]; Gavri Cohn[1]; Avigdor Shtechman[1]; Arie Bussiba[2]; [1]Rotem Industries, Ltd., Metal Forming Grp., POB 9046, Beer-Sheva 84190 Isreal; [2]N.R.C.N, POB 9001, Beer-Sheva 84190 Isreal

In recent years the use of high-pressure die-cast magnesium components for automotive applications, has markedly increased. This casting technology became widely used due to high productivity, short cycle time of tools and longer tool life. In addition, Thixomolding and other new developments in casting processes allow thin wall and complex shapes parts to be produced in mass production. In cases where high strength to density ratio and long fatigue life are required (usually in automotive moving parts) wrought magnesium alloys become necessary. Wrought alloys also have suitable elongation, which is reflected by good energy absorption capacity-a property which safety engineers in the car industry are looking for. In order to manufacture transportation vehicles like a handicapped scooter, other small vehicles or light constructions, one must use hybrid combination of wrought and cast alloys, that would provide the appropriate combination of weight and strength. Extruded profiles (tubes and bars) and forged rigid parts can be joined together by welding to create such structures. The main difficulty is still to select the appropriate welding technology that would be suitable for different hybrid combinations and would provide the essential mechanical properties of the hybrid joints. Several hybrid combinations were tested, using different welding technologies. Extruded AZ31B and AZ80 wrought magnesium alloys where joined to forged AZ31B joints. High-pressure die-cast AM50 tubes were welded to AZ31B forged parts using various welding technologies. The joints were characterized by mechanical testing, by optical and scanning electron microscopy as well as by an acoustic emission method. It was found that reliable and proper mechanical response combined with good corrosion resistance, characterize some of the hybrid magnesium joints. The optimum combination of alloys, welding technology and process parameters, would establish the final needed properties of the hybrid light magnesium construction. The use of hybrid, light-magnesium structures opens a wide horizon for the use of magnesium in transportation applications.

4:30 PM

New Magnesium Alloys for Automobile Applications: *D. Eliezer*[1]; [1]Ben Gurion University of the Negev, Dept. of Matls. Eng., Beer Sheva 84105 Israel

Magnesium is the lightest of all the commonly used metals and is thus very attractive for transportation applications. It also has other desirable features including reasonable ductility, better damping characteristics than aluminium and excellent castability. Because of increasing pressures to improve automotive gas mileage in most parts of the world, in combination with the required property combinations of the automobile industry, use of magnesium in cars and light trucks is increasing significantly. Magnesium alloy components are usually produced by various casting processes. The most applicable methods are high-pressure die-casting and gravity casting, particularly sand and permanent mold casting. Other relevant production technologies are: Squeeze Casting, Thixocasting and Thixomolding. This paper will evaluate the necessity of magnesium alloys development for expanding the current magnesium applications in the automotive industry. The paper will also address the correlation between the alloy development and the production technologies of the actual components. Some achievement in alloys development will be described.

4:50 PM
Fabrication of Cu-Based 3-D Parts by Direct Laser Sintering: *Zhu Hai Hong*[1]; [1]National University of Singapore, Dept. of Mechl. Eng., 10 Kent Ridge Crescent 119260 Singapore

The direct laser sintering is a layered manufacturing process which can produce any desired three-dimensional parts with simple or complex shape. Recent research in this area has focused on the direct laser sintering of metal powder. Cu-based metal systems are the active research area in direct laser sintering because of its good thermal performance and high electrical conductivity. This paper reports on research work in direct laser sintering of a multiphase Cu-based powder in details. A composite metal powder consisting of Cu, Ag and P was successfully sintered in a self-developed Rapid Prototyping machine in ambient atmosphere at room temperature. 68.9% theoretical density and average surface roughness Ra 14-16mm were achieved with negligible distortion and shrinkage. Some 3-D parts have been fabricated successfully. Furthermore, the influence of the amount of liquid phase on mechanical properties, such as tensile strength and impact toughness were also investigated in this paper.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Thermodynamic Databases and Assessments

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

Monday PM — Room: 204
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Gerhard Inden, Max Planck Institut für Eisenforschung, GmbH, Max Planck Strasse 1, Postfach 140 444, Düsseldorf 40237 Germany; Frederick H. Hayes, University of Manchester/UMIST, Matls. Sci. Ctr., Grosvernor St., Manchester M1 7HS UK

2:00 PM Invited
Thermodynamic Tools for Alloy Design: *Joanne L. Murray*[1]; [1]Alcoa Technical Center, TPAL, 100 Technical Dr., Alcoa Center, PA 15069 USA

CALPHAD-type thermodynamic calculations of phase diagrams are now one of several computational tools for microstructure simulation. The eventual goal is to span the length scales from the atomic level to the macroscopic level and relate microstructure to properties and performance. Work on precipitation reactions during aging of 2xxx series aluminum alloys will be described.

2:30 PM Invited
Construction of a Thermodynamic Database for Ni-Base Superalloys: A Case Study: *Ursula R. Kattner*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

Ni-base alloys are the most widely used superalloys in applications involving high temperatures and severe environmental conditions and are also among the most complex. To model the various processing steps improvements in thermodynamic and diffusion databases are required. The first thermodynamic database for superalloys was developed by Larry Kaufman and co-workers in the 70's. Since then, computational technology has constantly improved resulting in advanced model descriptions and better reproducibility of the experimental observations. A database for Ni-base superalloys with emphasis on single crystal alloys has been assembled for 10 components: Ni, Al, Co, Cr, Hf, Mo, Re, Ta, Ti, W. Since the number of subsystems for such a database is enormous, it is desirable to utilize assessments from literature. These assessments must be reviewed for consistency and, if necessary, partially remodeled. Experience from assembling this database with discussion of more systematic estimation procedures for the unknown quantities will be presented.

3:00 PM Break

3:30 PM Invited
The State of the Art of Assessment Techniques, Part I: *Suzana Gomes Fries*[1]; *Bo Sundman*[2]; Hans Leo Lukas[3]; [1]ACCESS e.V., RWTH-Aachen, Intzestrasse 5, Aachen D-52072 Germany; [2]KTH, MSE, Stockholm SE-10044 Sweden; [3]MPI fuer Metallforschung, NMAM, Universitaet Stuttgart, Heisenberg Str 5, Stuttgart D-70569 Germany

The CALPHAD technique has reached maturity. It started from the vision to combine data from thermodynamics, phase diagram and atomistic properties like magnetism into a unified model. It is now a powerful method in a wide field of applications. The successful use of CALPHAD comes from the development of multicomponent databases which describe many different kinds of experimental data in a consistent way. The construction of these databases is still a very demanding task, requiring expertise and experience. There are quite a lot of subjective factors in the decisions when judging and selecting between redundant experimental data which are the most trustful. Furthermore the growing range of applications of these databases increase the feedback and several corrections and modifications are required. The development of new models and the rapid advancement of atomistic calculations make the assessments techniques very dynamic and challenging. The assessment experiences collected since many by the three authors, which have differnt backgrounds, will be reported.

4:00 PM Invited
The State of the Art of Assessment Techniques, Part II: *Bo Sundman*[1]; Suzana Gomes Fries[2]; Hans Leo Lukas[3]; [1]KTH, MSE, Stockholm SE 100 44 Sweden; [2]Access, Inc., Inzelstr 5, Aachen Germany; [3]MPI, PML, Heisenbergstr 5, Stuttgart Germany

The Calphad technique is now a powerful method in a wide field of applications where calculated Gibbs energies and derivatives thereof are used to calculate properties and simulate transformations of multicomponent phases. Chemical potentials and the thermodynamic factor, the second derivative of the Gibbs energy, is used in diffusion simulations. The driving forces of metastable phases are used to simulate the evolution of microstructures In solidification simulations the segregation and fractions of different solid phases as well heat capacities and entropies can be obtained during slow as well as rapid solidification. Whenever the thermodynamic description of a phase is required, the Calphad method can be applied. The construction of these databases is a demanding task, requiring expertise and experience and there is still quite a lot of subjective factors involved when judging and selecting between redundant experimental data which are the most trustful. The growing range of applications increase the feedback and several corrections and modifications are required. The development of new models and the rapid advancement of atomistic calculations makes the assessments techniques very dynamic and challenging. Some of the assessment experiences the three authors has collected, since many years, will be reported in this presentation.

4:30 PM Invited
An Associate Model and Thermodynamic Database for Dilute Solutions of Oxygen and Metals in Molten Iron: *In-Ho Jung*[1]; Sergei Degterov[1]; Arthur Pelton[1]; [1]Ecole Polytechnique, Matls. Eng., PO Box 6079, Sta. "Downtown", Montreal, Quebec H3C 3A7 Canada

In the classical interaction parameter formalism of Wagner, the solute atoms are assumed to mix randomly. However, for solutions of oxygen and highly reactive metals M = Ca, Mg, Ba, Al,.... in molten iron, there is a strong tendency for the formation of associated solute "molecules" MO. When this is taken into account, the model is greatly simplified. Usually only one temperature-independent parameter is required to reproduce all experimental deoxidation equilibria and oxygen activities at all measured compositions and temperatures. The deoxidation curves for M = Ca, Mg are elucidated for the first time. A database has been prepared with evaluated parameters for M = Al,B,Ba,Ca,Ce,Cr,Hf,La,Mg,Mn,Nb,Nd,Ta,Ti,Th,Si,V,Zr.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Interface Effects

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

Monday PM	Room: 617
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: John Cahn, NIST, Matls. Sci. & Eng. Lab., 223/A153, Gaithersburg, MD 20899-8555 USA; S. Ranganathan, Centre for Advanced Study, Dept. of Metall., Bangalore 560012 India

2:00 PM Invited
Interpretation of Surface Relief Effects in Terms of Interphase Boundary Structure: *Hubert I. Aaronson*[1]; Barrington C. Muddle[2]; Jian Feng Nie[2]; [1]Carnegie Mellon University, Matls. Sci. & Eng., Pittsburgh, PA 15213-3890 USA; [2]Monash University, Sch. of Phys. & Matls. Eng., Monash University, Victoria 3800 Australia

During a recent symposium on the mechanisms of the massive transformation (MT), the central focus was on the structure of micron-size planar massive:matrix boundaries formed by irrational orientation relationships and habit planes at which misfit-compensating defects could not be detected even with HRTEM. Whereas most participants considered these boundaries to be incoherent, some described them as partly coherent with interfacial structures yet to be resolved. Although surface relief effects (SRE) of the invariant plane strain or tent types are considered to be absent during the MT, such effects can be discerned in published micrographs of Fe-Ni and Ag-Cd alloys. On the view of Christian (1962) that such SREs result from passage of partially coherent boundaries, the presence or absence of such effects should be a useful test for boundary structure. However, this test must be applied with caution. Partly coherent boundaries should not yield such SREs when: (i) there is no change in stacking sequence across the boundaries; (ii) successive "shears" mutually accommodate; (iii) terraces of growth ledges are nearly parallel to the specimen surface; (iv) the terrace plane is of CSL type (Nie and Muddle, 2000); and (v) all interfaces of product phase crystals have their equilibrium interfacial structure (W. Z. Zhang, 1999).

2:25 PM Invited
Thermodynamics of Interfaces in Mechanically Alloyed Metals: *Harshad Kumar Dharamshi Hansraj Bhadeshia*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Cambridge CB3 7EQ UK

Normal thermodynamic theory for solutions begins with the mixing of component atoms. Many solutions are, however, prepared by mixing together lumps of the components, each of which might contain millions of identical atoms. In previous work, Badmos and Bhadeshia examined the way in which a solution evolves from these large clusters of components, from a purely thermodynamic point of view. They predicted that solution formation by the mechanical alloying of solid components cannot occur unless there is a gain in coherency as the particles become small. The nature of the barrier to mechanical alloying was discovered. There is also the possibility of a metastable state prior to the achievement of full solution, when the component atoms prefer like-neighbours. The evolution of the interface as mechanical alloying proceeds is investigated in this paper, together with implications on the formation of colloidal suspensions.

2:50 PM Invited
Diffusion at Zero-Flux Multicomponent Interfaces: *Martin E. Glicksman*[1]; Afina O. Lupulescu[1]; [1]Rensselaer Polytechnic Institute, MS&E Dept., CII-9111, Troy, NY 12180-3590 USA

Multicomponent diffusion gives rise to zero flux planes, ZFP's, where the flux of a component vanishes. The conditions for developing ZFP's in multicomponent alloys are now well understood through the works of Morral and co-workers. In this presentation we analyze dynamics of multicomponent diffusion near stationary ZFP's. In contrast to normal "global" component mixing that occurs in binary diffusion couples, the average equilibrium composition is achieved with a stationary ZFP by release of a pair of coupled diffusion waves. Curiously, each wave independently establishes the average concentration near the Matano interface. A stationary ZFP always prevents net transport of the component across the diffusion zone, which has both practically interesting implications for the design and lifetime of multicomponent films, coatings, and claddings that resist loss of a component into the substrate. Flux behavior is discussed for combinations of Cr-Al-Ni ternary alloys chosen near the composition 10at.%-Cr, 10at.%-Al, balance Ni.

3:15 PM Invited
Relationships among Interface Diffusion, Energies and Solute Segregations in Materials: *Devendra Gupta*[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

During the last several decades, a strong connection among the important properties of interfaces, notably, the energy, solute segregation and diffusion has been firmly established. We will discuss the methodology and the thermodynamical analysis of the diffusion measurements to obtain these parameters and illustrate them by results obtained in a variety of materials. Investigations carried out in pure polycrystalline metals have yielded grain boundary energies comparable to those directly measured. Furthermore, we will discuss the role of solute segregation at grain boundaries in alloys in altering diffusion. From the perturbations caused, solute segregation parameters,the enthalpy and the entropy of binding, have been extracted and levels of solute concentrations estimated. Finally, it will be shown that similar analyses when applied to complex materials' interfaces, e.g. the Ni_3Al intermetallic compound, the PbSn eutectic alloy and the Ag-piezo-electric ceramic system, also result in acceptable values of energies and segregation factors.

3:40 PM
Effect of Irradiation-Induced Interfaces on Thin Film Stability: *Paolo M. Ossi*[1]; [1]INFM-Politecnico di Milano, Dip. di Ingegneria Nucleare, via Ponzio, 34/3, Milano, MI I-20133 Italy

Dramatic amorphisability differences are observed in chemically similar compounds, irradiated under identical conditions. The atomistic Segregation-Charge Transfer (SCT) model for the structural stability of binary systems, bombarded under conditions such that dense collision cascades form is presented. At each cascade-matrix interface an off-equilibrium compositional profile develops, following interface enrichment in one compound constituent. Concurrently, a local electronic density profile develops, which can be non-equilibrium over the relevant cascade quenching timescale. Relaxation to (meta)stable equilibrium is schematised by charge transfer (CT) reactions, each involving two dissimilar atoms of the initial compound (IC), that generate an effective compound (EC) dimer. Every EC is compared to the corresponding IC, considering the CT associated energy cost, the enthalpy change at EC formation, and the local volume change, related to strain effects. For a meaningful set of compounds, both metallic and non-metallic, threshold parameter values separate amorphised materials from materials remaining crystalline upon irradiation.

4:00 PM Invited
The Role of Liquid-Solid Interface during Melting and Solidification during Surface Alloying and Dissimilar Metal Welding by Laser: *Kamanio Chattopadhyay*[1]; Gandham Phanikumar[1]; Pradip Dutta[2]; [1]Indian Institute of Science, Metall., Bangalore, Karnataka 560012 India; [2]Indian Institute of Science, Mechl. Eng., Bangalore, Karnataka 560012 India

Solidification is a first order transformation, where the transformation and the resulting microstructural changes take place by the movement of the transformation interface. The thermal and solutal conditions prevailing at the solid liquid interface and the thermodynamic constraints that exist at the interface control this movement and determine the evolution of the solidification microstructure. In the case of laser surface alloying and dissimilar metal welding, the situation is further complicated by the rapid heat transfer and fluid flow. The microstructure is often inhomogeneous and contains different concentration domains which are temporally varying. Thus the interface often experiences a changing condition which affects its motion and hence the microstructure. In this presentation, we shall give examples from the work done in our laboratory to elucidate these complexities. Attempts will be made to relate the origin of the complexities in the microstructure to the changing interface conditions.

4:25 PM
Interphase Interfaces and the Mechanisms of the α to γ_m Massive Transformation in Ti-Al Alloys: Ping Wang[2]; Veer Dhandapani[3];

Mukul Kumar[4]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Brown University, Div. of Eng., Providence, RI 29212 USA; [3]Motorola, Adv. Prod. R&D Lab., Austin, TX 78721 USA; [4]Lawrence Livermore National Laboratory, L-356, Livermore, CA 94550 USA

The massive-matrix interphase interfaces associated with the α to γ_m massive transformation in Ti-(46-48)Al alloys were studied. Special experiments were performed to arrest the transformation at an early stage. Nucleation and growth kinetics were determined and the former compared with calculations for various nuclei shapes using classical nucleation theory. Orientation relations between the γ_m and parent α (retained α_2) phases were determined using EBSD in an SEM and by electron diffraction, and the interphase interfaces characterized by two-beam bright-field/weak-beam dark-field TEM and HRTEM. The results reveal that the γ_m nucleates at grain boundaries with a low-index orientation relation and coherent interface with one parent grain, but grows into the adjacent grain with a high-index/irrational orientation relation. The growth interfaces between the two phases are generally free of misfit dislocations and consist of curved parts as well as planar facets whose macroscopic habit varies from high-index/irrational to low-index orientation. On an atomic scale the growth interfaces are often found to be faceted along low-index planes with steps, but are incoherent with respect to the parent grain into which growth occurs. The implications of these results on the nucleation and growth mechanisms associated with the α to γ_m massive transformation will be discussed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

4:45 PM

In-Situ Transmission Electron Microscopy of Combustion Synthesized TiC-NiTi Composites: *E. R. Strutt*[1]; T. Radetic[2]; M. A. Meyers[1]; [1]University of California-San Diego, Dept. of Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]National Center for Electron Microcopy, Lawrence Berkeley National Lab., Berkeley, CA 94720 USA

In-situ transmission electron microscopy was performed on combustion synthesized TiC-NiTi composites in order to observe how stiff ceramic inclusions affect the martensitic transformations in NiTi. The mechanisms of nucleation and growth of martensitic B19' have been elucidated. The effect of thermal cycling on the mechanism of transformation has been studied. The similarities and differences of the transformation mechanism between reinforced and unreinforced Ti-rich NiTi alloys are discussed in terms of a possible optimum dislocation density for the formation of the R-phase in TiC- NiTi composites. Research supported by the US Army Research Office under contracts DAAH04-95-1-0236 and DAAH04-96-1-0376, and by the Director, Office of Science, Office of Basic Energy Sciences, of the US Department of Energy under Contract No. DE-AC03-76SF00098. The transmission electron microscopy work was performed at the National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Interfacial Reactions and Intermetallics

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

Monday PM	Room: 612
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Srini Chada, Motorola, APTC, 8000 W. Sunrise Blvd., Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

2:00 PM Opening Remarks

2:05 PM Invited

Mechanical and Electrical Properties of Lead(Pb)-Free Solder Joints: *Sung K. Kang*[1]; M. J. Yim[2]; W. K. Choi[1]; D. Y. Shih[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA; [2]Korea Advanced Institute Science & Technology, Dept. of Matls. Sci. & Eng., Taejon Korea

The mechanical and electrical properties of several Pb-free solder joints were investigated in terms of their interfacial reactions, namely, the thickness and morphology of the intermetallic layers. In order to measure both the shear strength of a solder joint and its electrical resistance, a model joint was made by joining two “L-shaped” copper coupons. Three Pb-free solders investigated are Sn-3.5%Ag, Sn-3.8%Ag-0.7%Cu, and Sn-3.5%Ag-3%Bi (all in weight %), combined with two surface finishes, Cu and Au/Ni(P). By varying the reflow time and aging time, the thickness of the intermetallic layers grown at the joint interface was changed from a few micron to more than 10 micron. The morphology of the intermetallic compounds was significantly influenced by the solder composition as well as the choice of a surface finish layer. The correlations between the mechanical and electrical properties of Pb-free solder joints and their interfacial reactions are made. The microstructure of the solder joints has also been investigated to understand the characteristic behavior of the Pb-free solder joints.

2:30 PM

Nucleation Kinetics of Cu_6Sn_5 by Reaction of Molten Tin with a Copper Substrate: *Robert A. Gagliano*[1]; Morris E. Fine[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The nucleation kinetics of η-phase (Cu_6Sn_5) intermetallic compound is being investigated by hot dipping copper coupons in molten tin for several very short times, beginning with one second, at temperatures varying from 240ºC to 300ºC. In the SEM, the Cu_6Sn_5 phase appears as small, rounded bumps of varying size jutting out from the surface of the copper. There are generally more bumps present at lower reaction temperatures than at higher reaction temperatures. The role of coarsening is being examined by comparing the one sec. reaction time results with results for 2 and 3 sec. reaction time. Quantitative measurements of the experimentally determined nucleation rate/unit area will be presented and compared with theoretical predictions over the temperature range investigated. Activation energies, kinetic parameters, and nucleation models will also be discussed.

2:50 PM

The Effect of Cu on Interfacial Reaction in Lead-Free Systems: *Ka Yau Lee*[1]; Ming Li[1]; [1]Institute of Materials Research & Engineering, 3 Research Link, 117602 Singapore

The interactions of two lead-free solders: Sn-3.5Ag and Sn-3.0Ag-0.5Cu with Ni/Au metallization after liquid- and solid-state reaction have been investigated. Both systems were comparable in ball shear strength and performed better than SnPbAg system after solid-state annealing. Unlike SnPbAg system after solid-state reaction, re-precipitation of Au as $Au_{0.5}Ni_{0.5}Sn_4$ at the interface was not observed in both lead-free systems. Comparison was also made on interfacial morphologies of Cu-Sn-Ni intermetallic compound formed in SnAgCu system and Ni_3Sn_4 found in SnAg system. Thick layer of acicular Cu-Sn-Ni with large aspect ratio formed after liquid-state reaction. In contrast, the intermetallic compound became denser and more faceted with solid-state annealing time. The compound also grew slower than Ni_3Sn_4, with an activation energy of ~200 kJ/mol. The preferential formation of this Cu_6Sn_5-based compound was explained with the help of ternary phase diagram.

3:10 PM

A Study of the Effects of Solder Volume on the Interfacial Reactions in Solder Joints using Differential Scanning Calorimetry Technique: *Won Kyoung Choi*[1]; Sung K. Kang[1]; Da-Yuan Shih[1]; [1]IBM, T. J. Watson Rsrch. Ctr., PO Box 218, Yorktown Heights, NY 10598 USA

The interfacial reactions during soldering have been extensively investigated in the literature by employing various experimental techniques such as conventional cross-sectional metallography, scanning electron microscopy (SEM), electron microprobe analysis, X-ray diffraction and others. However, these techniques are time consuming and require special sample preparation. In this study, the differential scanning calorimetry (DSC) technique was employed to understand the interfacial reactions. In DSC, the simulation of a soldering process and the analysis of the interfacial reactions can be performed at the same time. The measurements of peak temperatures and heat involved provide useful information about the interfacial reactions such as the amount of intermetallic compounds (IMC) formed and that of Cu dissolved. Sn-plated Cu balls were prepared as samples for the DSC study. Cu balls with a different Sn thickness were used to investigate the solder volume effect. In addition, a Ni diffusion barrier layer (electroless or electrolytic) was introduced between Cu and Sn to examine its role on the dissolution kinetics of Cu into a molten Sn. The conventional metallography combined with SEM was also used to verify the DSC results.

3:30 PM

Isothermal Solidification of Cu/Sn Diffusion Couples to Form Thin Interconnects: *Judy Sooyean Kang*[1]; Robert A. Gagliano[1]; Gautam Ghosh[1]; Morris E. Fine[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The ever increasing demand for higher density of input/output (I/O) connections in electronic devices require smaller and smaller interconnects. Utilizing a lead-free approach for chip to chip carrier interconnects of a size scale that allows a very high level of integration is a viable approach to this problem. Recently, we have applied the concept of isothermal solidification to form a joint as thin as 30 µm between two Cu plates. Using pure Sn and a relatively low processing temperature, 275 to 325°C, we have been able to form intermetallic based joints containing only Cu_3Sn. The reaction between liquid Sn and Cu leads to formation of Cu_6Sn_5 and Cu_3Sn intermetallics which continue to grow until all Sn is consumed to form a solid bond. Depending on the extent of further reaction desired, the end product may be either Cu_6Sn_5+Cu_3Sn or only Cu_3Sn. The former requires less Cu. We will discuss the dynamics and morphology of Cu_6Sn_5 and Cu_3Sn intermetallic growth which are essential for controlling the isothermal solidification process.

3:50 PM Break

4:05 PM Invited

Textured Growth of Cu/Sn Intermetallic Compounds: *Kithva Prakash Hariram*[1]; Thirumany Sritharan[1]; [1]Nanyang Technological University, Sch. of Matls. Eng., Nanyang Ave. 639798 Singapore

The growth of Cu-Sn intermetallic compounds (IMC) at the interface of Cu and molten Sn-Pb solder alloys is studied over a range of temperature. X-ray studies show a very strong (10.2) and (10.1) peaks of η-phase (Cu_6Sn_5) when the Sn content was high, at all temperatures. In the low Sn solder (27Sn-73Pb), the η-phase peaks were absent at the two high temperatures but the (2 12 0) peak of ε-phase (Cu_3Sn) was prominent. (10.1) and (002) pole figures were constructed, using the conventional texture goniometer, for η-phase and ε-phase, respectively. The growth directions were identified to be <101> and <102> for η-phase and <102> and <031> for ε-phase, normal to the Cu surface. The growth directions do not change with the morphology and the layer thickness. The morphology of the η-phase layer varies gradually from a cellular film with rugged interface to a dense film with scalloped interface as the Pb content, temperature and reaction time increase. The ε-phase was dense and planar.

4:30 PM

Interfacial Reactions of In-Containing Solder Alloys on the Au Deposited Substrates Ni and Cu: *Chang Youl Lee*[1]; *Seung Boo Jung*[1]; *Chang Jae Shur*[1]; [1]Sung Kyun Kwan University, Metlgcl. & Matls. Eng., 300 Chunchun-Dong, Jangan-Gu, Suwon, Kyonggi-Do 440-746 Korea

The reliability of the solder joint is affected by type and extent of the interfacial reaction between solder and substrates. Therefore, understanding of intermetallic phase produced by soldering in electronic packaging is essential. Among many solder alloys, In-alloy solders are used as low-melting temperature solder. In the present work, the interfacial reactions of In solder alloy on the Au deposited substrates (Cu and Ni) are investigated. Also the wettability of liquid In solder on the various substrates was determined from meniscope method. Experimental results showed that the intermetallic compounds are observed Cu11In9, Ni10In27 and AuIn2 for different substrate respectively. Additionally, the growth rate of these intermetallic compounds were increased with the reaction temperature and time. We found the growth of this intermetallic compound in aging followed the parabolic law, which indicates that the growth is diffusion-controlled.

4:50 PM

Solder/UBM Reaction Characteristics in Optical Packages: Jong-Hwan Park[1]; Jong-Hyun Lee[1]; *Yong-Seog Kim*[1]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Seoul 121-791 S. Korea

Au/Pt/Ti UBM has been used frequently for Au-Sn solder pad in optical packages. Under a typical reflow condition, the diffusion barrier layer, Pt, was found to dissolve into the solder completely, exposing the adhesion layer of poor wettability to the solder. The dissolution is due to high reflow soldering temperature as well as formation of complete solid solution with Au. In this study, several metals and intermetallics were employed as the diffusion barrier layer and their dissolution and reaction kinetics at the solder/UBM interface were investigated. Preliminary investigations indicated that Ni, Cu, Cu6Sn5, and Ni3Sn4 layers dissolve into the solder readily and form intermetallics at the interface. Other diffusion barrier materials investigated include refractory metals such as W, Mo, and Nb. The dissolution and reaction kinetics of the metal layers with the solder were examined and fracture energy of the solder interconnection was measured by a ball shear tester.

5:10 PM

Failure Mechanism of Lead-Free Solder Joints for Flip Chip Packages: Fan Zhang[1]; *Ming Li*[1]; Balakrisnan Bavani[1]; William T. Chen[2]; *K. Y. Lee*[1]; [1]Institute of Materials Research and Engineering, Opto- & Elect. Sys. Cluster, 3 Research Link 117602 Singapore; [2]ASE (US), Inc., 3590 Peterson Way, Santa Clara, CA 95054 USA

The failure mechanisms of SnAgCu solders on Al/Ni(V)/Cu under bump metallization (UBM) were investigated after multiple reflows and high temperature storage using ball shear test, fracture surface analysis, and cross-sectional microstructure examination. The results were compared with those of eutectic SnPb solder. It was found that in the SnAgCu system, the failure mode changed with the number of reflow from ductile failure inside the solder ball to brittle failure near the interface between intermetallic layer and UBM. The change in the failure mode was found relating to the degradation of UBM structure due to the severe interfacial reaction between solder and UBM layer. After high temperature storage, the solder joints failed inside solder ball in a ductile manner in both SnAgCu and SnPb systems. The reduction of shear strength was observed with the increase of the aging time, which was explained by the microstructure evolution of the solder material.

5:30 PM

The Phenomena of Electroless Ni-P Stripping and Dissolving in the Eutectic Sn-Bi Solder: *Chien Sheng Huang*[1]; Bi Lian Young[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Interfacial reactions between electroless Ni-P (EN) and eutectic Sn-Bi solder was characterized. Joints of 42Sn-58Bi/Au/Ni-5.5wt%P/ Cu/ Al2O3 and 42Sn-58Bi/Au/Ni-12.1wt%P/Cu/Al2O3 were annealed at 145ºC and 185ºC for 30~180 minutes. EN deposited with a high pH value exhibited lower P content and larger nodules. After annealing, there are two interfacial intermetallics (Ni3Sn4 and Ni3P) formed between solder and EN. In the 42Sn-58Bi/Au/Ni-5.5wt%P/Cu joint, Ni3Sn4 was formed and stripped into the solder after annealed for long periods of time. The EN was first stripped from the initial interface and then dissolved into the Sn-Bi solder during annealing at 185ºC. In the 42Sn-58Bi/Au/ Ni-12.1wt%P/Cu joint, the growth of IMC was limited even annealed at either elevated temperature of 185ºC or long periods of 180 minutes. No EN stripping was found. It is believed that the tendency of EN stripping was related to the nodule size or surface roughness of EN. From the morphology of the interface between solder and EN, the mechanism of EN stripping and dissolving could be probed.

Magnesium Technology 2002: Magnesium Recycling and Environmental Issues

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

Monday PM	Room: 606
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: John N. Hryn, Argonne National Laboratory, 9700 S. Cass Ave., Bldg. 362, Argonne, IL 60439-4815 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Matls. Procg. Grp., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA

2:00 PM
Solubility of Fluorine in Molten Magnesium: *Kari Aarstad*[1]; Martin Syvertsen[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Alfred Getz vei 2b, Trondheim 7491 Norway; [2]SINTEF Materials Technology, Proc. Metall. & Cer., Alfred Getz vei 2, Trondheim Norway

As an alternative to the high global warming potential gas SF_6, oxidation and burning of liquid magnesium could be prevented by dissolving fluorine directly into the molten metal. The solubility of fluorine in molten magnesium was hence measured at temperatures between 700°C and 950°C. In these experiments, magnesium was equilibrated under argon in a MgF_2 crucible. Twice the furnace is evacuated to 1 mbar and then 99.99% argon is fed through. Samples of the equilibrated melt were taken using alumina tubes and a syringe. The fluorine solubility was determined employing the "Sintalyzer method", an electrochemical method developed at Sintef, Norway. As expected, the fluorine content in liquid magnesium is described by an exponential function of inverse temperature. Fluorine varies between 10 and 100 ppm by weight in the measured temperature interval.

2:30 PM
Progress towards Climate Friendly Magnesium Production and Casting: *Scott Charles Bartos*[1]; [1]US Environmental Protection Agency, Global Programs Div., 1200 Pennsylvania Ave. N.W., (6205J), Washington, DC 20460 USA

The US Environmental Protection Agency (EPA) is working cooperatively with the magnesium industry to reduce emissions of sulfur hexafluoride (SF6), a persistent and very potent greenhouse gas. EPA launched its domestic magnesium industry partnership in 1999 and is now proud to support the International Magnesium Association in its effort to identify and implement more environmentally friendly protective cover gas alternatives. This paper will discuss the impressive progress being made by EPA's US partners to protect the climate as well as other exciting initiatives that are taking place around the world.

3:00 PM
Recycling of Different Types of MG-Scrap: *Helmut Antrekowitsch*[1]; Gerhard Hanko[1]; Hubert Sommerhofer[1]; [1]University of Leoben, Non-Ferrous-Metall., Franz-Josef-Straße 18, Leoben, Styria 8700 Austria

The importance of magnesium recycling has become more evident in the last ten years. The more materials are recycled, the lower is the energy consumption and the fewer waste products will end up in landfills, in the water and in the air. Nowadays only high grade scrap such as gates, runners and drippings from die casting operations are used for the recycling process. For the coated and contaminated magnesium materials no economical and ecological processes exist. The department of nonferrous metallurgy at the university of Leoben, Austria, investigates different processes of melting and refining of these types of scrap. Concerning the reactivity of magnesium with other elements different techniques have to be tested. The European guideline on old automobiles, which gratifies that recycling processes considered in construction and design of new cars, relieve the winning of secondary magnesium.

3:30 PM Break

3:50 PM
The Successful Briquetting of Magnesium Chips and Turnings for Secondary Melting and Recycling: *David J. Roth*[1]; *Gerd Von Aschwege*[2]; [1]Altek, LLC, 314 Exton Commons, Exton, PA 19341 USA; [2]RUF, Gmbh & Co. KG, Tussenhausener StraBe 6, Zaisertshofen D-86874 Germany

The briquetting of magnesium chips and grindings has been successfully accomplished in Europe with the RUF two ram closed die system. Densities of 1.4-1.5 g/cm3 are achievable with this process allowing for safer handling by stabilizing and reducing the volatility of the fine magnesium particles. Government regulations and legislation on waste disposal have highlighted the importance of finding new, low-cost methods of recycling waste materials. Briquetting the fine material dramatically reduces the volume, making the shipping of the material safer, easier and more cost effective. This paper will review case studies on the recycling of the briquetted magnesium chips. It will discuss the equipment and the uses and remelt possibilities of the briquetted magnesium. It will also discuss the findings from an ongoing study partially funded by the European community to handle the magnesium chips produced by the automotive industry.

4:20 PM
Fluxless Refining of Clean Diecast Scrap for Noranda's AJ52 High Temperature Mg-Al-Sr Alloy: *Peter Forakis*[1]; Eric Richard[1]; Donald Argo[2]; [1]Noranda, Inc. Technology Centre, Metal Proc., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada; [2]Noranda, Inc. Technology Centre, Matls. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

The AJ52 high temperature Mg-Al-Sr alloy developed by Noranda has shown its potential for use in automotive powertrain components requiring elevated temperature creep resistance and other high temperature mechanical properties through extensive property evaluations and casting trials. A factor that must be addressed in using the alloy, as for other alloys, is the recycling of trim and scrap components (type 1 scrap). Many of the alloying elements (most notably Sr, Ca and to a lesser extent the rare earths) used in the design of alloys for improved high temperature properties tend to react with the common fluxes used for recycling and refining the typical AZ and AM alloys. This results in the loss of needed alloying ingredients, excessive dross generation and can affect the overall quality of the refined metal. This study investigates, through laboratory and pilot plant molten metal trials, the loss of these alloying ingredients when in contact with fluxes and specifically the potential of fluxless refining techniques for bringing AJ52 type 1 scrap to a high level of metal cleanliness with minimal Sr losses.

4:50 PM
Application of Magoxide Method for Cleanliness Evaluation of Magnesium Alloys: *Boris Bronfin*[1]; Nataly Polyak[1]; Eli Aghion[1]; Carl Carlton Fuerst[2]; David Barris[3]; [1]Dead Sea Magnesium, Magnesium Rsrch. Div., PO Box 1195, Beer-Sheva 84111 Israel; [2]General Motors, Global R&D Ops., PO Box 9055, Warren, MI 48090-9055 USA; [3]General Motors, Non Ferrous Metals Worldwide Purch., PO Box 9015, Warren, MI 48090-9015 USA

A new method named "MagOxide" aiming at evaluation of the MgO and Al-Mn-Fe intermetallics in magnesium alloys was developed by DSM's Research Division. The newly developed method is based on wet chemistry procedure and is compatible with the Fast Neutron Activation Analysis (FNAA) technique. The method was successfully implemented on a variety of primary and recycled Mg alloys. The present paper aims at presenting the systematic correlation of results obtained by FNAA and "MagOxide" methods when primary and recycled Mg alloys of AZ and AM series were analyzed.

5:20 PM
Use of Fluoroketones in Cover Gases for Molten Magnesium: Dean S. Milbrath[1]; John G. Owens[1]; [1]3M Specialty Materials Laboratory, St. Paul, MN, USA

Abstract unavailable

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Fabrication Methods and Principles - Session II

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

Monday PM Room: 205
February 18, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: David C. Dunand, Northwest University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; Iver Anderson, Iowa State University, Ames Lab., 122 Metals Development Bldg., Ames, IA 50011-3020 USA

2:00 PM
Stabilization Mechanisms of Metal Foams by Ceramic Particles in Liquid-State Processing: *Y. Q. Sun*[1]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

The stabilization mechanisms of metal foams by ceramic particles in their liquid-state processing are determined by experiments. The focus is on the relative foam stabilizing effects of liquid viscosity and the wetting properties of the solid particles in the liquid. The stabilization of liquid metal foams by partially wetting and non-dissolving ceramic particles is simulated experimentally with liquid solutions with controllable surface tensions and continuously variable wetting contact angles. The results show that liquid foams with micrometer pore sizes can be stabilized with partially wetting particles alone and that there is an intermediate wetting angular range around 80° in which the liquid foam has the optimum stability. The key steps in the liquid-state processing of metal foams stabilized by solid particles are also identified by the experimental simulations.

2:25 PM
Rheological Changes of Cellular Structure in Foaming Metal: *Zhenlun Song*[1]; Steven Nutt[1]; [1]University of Southern California, Composites Ctr., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA

Controlling melt viscosity is a key process parameter in the production of metal foams by melt processing. In this work, rheological changes of molten aluminum during foaming were investigated. In this process, the aluminum charge is placed in a crucible and heated to the melting point. Mechanical agitation is introduced, and when suitable viscosity is achieved, a granular foaming agent is injected. Foam expansion ensues rapidly, and the melt viscosity is critical to achieve stable foaming and avoid slumping. Three types of cell shapes in the foam are considered: (1) near the sidewall of the crucible, where cells experience shear deformation, (2) near the top of the foaming melt, where cells experience compression deformation, and (3) near the bottom of the foaming melt, where cells are well-developed. A qualitative model of the process is proposed explain the observations, and matched to experimental data.

2:50 PM
The Preparation of Highly Porous Structures from Filamentary Nickel Powders: *Alexandre Yu. Zaitsev*[1]; David S. Wilkinson[1]; George C. Weatherly[1]; Thomas F. Stephenson[2]; [1]McMaster University, Matl. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Inco Technical Services Limited, 2060 Flavelle Boulevard, Mississauga, Ontario L5K 1Z9 Canada

Porous nickel structures are in high demand for battery, catalyst and filter materials applications. Traditionally such structures are made by sintering of fine filamentary nickel powders. However, the strength of such structures is rather low, when compared for example with Ni foams of similar density. Our work is focused on increasing the strength through improved processing. In particular, colloidal processing techniques can be used to improve the powder particle distribution and, hence, the strength of the final sintered structure. By dispersing Ni powder in water prior to introducing a binder, better separation of particles and break-up of conglomerates is achieved. The addition of dispersant agents further improves the particle distribution. This method also improves control of the slurry viscosity and the green density of the nickel porous body. The structure of battery plaque prepared according to the new technique is much more uniform than the structure of the conventional plaque.

3:15 PM Break

3:35 PM
Novel Lightweight Open Cell Metal Foam Process and Resulting Properties: *Amit K. Ghosh*[1]; Xingcun Tong[1]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

A new low cost process has been developed for fabricating both stochastic and periodic open cell metal foam structures by direct deposition of solidifying liquid wires, droplets and spray, and simultaneously welding them in-situ. The process is capable of producing a wide range of pore spaces from microns to millimeters and amenable to computer-aided manufacturing technique. Open cell structures with closed outer surfaces and solid face sheets have been produced. The process is also capable of producing graded porosity, and various internal geometries when deposition is carried out over removable geometric inserts. Samples with a variety of internal geometries were produced from several aluminum alloys to create multiscale porosity levels, and tested under compression for elastic modulus and strength. The results show that plateau strength and modulus can be influenced by the choice of internal geometry, which are not uniquely predicted by ligament bending-based models. Simple models demonstrate that the orientation of internal columns and walls are directly connected with the measured deformation behavior. (This research was supported by the US Office of Naval Research, under grant: DOD-G-N00014-97-1-0510, Program Manager: Dr. S. G. Fishman).

4:00 PM
Processing of IN-718 Lattice Block Castings: *Mohan Hebsur*[1]; [1]NASA Glenn Research Center, OAI, Brookpark Rd., Cleveland, OH 44135 USA

Recently a low cost casting method known as lattice block casting has been developed by JAM Corp, Boston MA for engineering materials such as Aluminum and Stainless steels that has shown to provide very high stiffness and strength with only a fraction of density of the parent alloy. NASA-Glenn has initiated a research to investigate lattice block castings of high temperature Ni-base superalloys such as IN-718 for lightweight nozzle applications. Initially there were numerous problems in making and assembling the wax patterns of our design. It was decided to try a new wax, less fragile and a lot stronger than anything tried earlier. Not only did the molds filled easily with the new formula, also the parts came out of the molds without breaking and were easy to assemble. Totally ten Inconel-718 lattice block castings (each measuring 6 inch wide x 12 inch long x 0.5 inch thick) have been successfully produced by MCT Inc., Manchester NH using their patented counter gravity casting techniques. Details of the processing and resulting microstructures will be discussed. Post casting processing and evaluation of system specific mechanical property of these specimens are in progress. This work is in support of the Ultra Efficient Engine Technology (UEET) lightweight nozzle program.

4:25 PM
Processing of Controlled Porosity Titanium-Based Materials: *T. Dennis Claar*[1]; Ulf Waag[2]; Hartmut Goehler[2]; Donald M. Kupp[1]; [1]Fraunhofer USA, Ctr. for Mfg. & Adv. Matls., 501 Wyoming Rd., Newark, DE 19716 USA; [2]Fraunhofer IFAM, Powder Metall. & Composite Matls.-Dresden, Winterbergstr. 28, Dresden 01277 Germany

Alloys and intermetallic compounds based on titanium are used extensively in aerospace and biomedical applications, based on their high strength, light weight, and excellent corrosion resistance. To extend the range of applications, designers and engineers are now seeking titanium-based materials with controlled porosity. Ultra-lightweight, porous titanium alloys and intermetallics are being fabricated based on technologies being developed at the Fraunhofer Institute and Fraunhofer USA Center for Manufacturing and Advanced Materials, located in Dresden, Germany and Newark, DE, respectively. Powder metallurgy processing routes are being utilized to fabricate hollow metal spheres and open-celled foams in titanium, Ti-6Al-4V alloy, and TiAl and TiAl3 intermetallics. The processing, microstructures, and selected properties of these controlled-porosity materials will be presented, along with a discussion of potential applications in the aerospace, biotechnology, and other industries.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium

Poster Session: Monday 5:30 PM – 8:00 PM

Second International Symposium on Ultrafine Grained Materials: Processing and Structure - II

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

Monday PM	Room: 210
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: K. Ted Hartwig, Texas A&M University, Mechl. Eng., 319 Engineering Physics Bldg., Spence St., College Station, TX USA; Ke Lu, Institute of Metal Research, Shenyang Natl. Lab. for Matls. Sci., Chinese Academy of Sciences, Shenyang 110016 China

2:00 PM Keynote

Recent Developments of SPD Processing of Fabrication of Bulk Nanostructured Materials: *Ruslan Z. Valiev*[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia

During the last decade severe plastic deformation (SPD) techniques have been successfully used for the fabrication of nanostructures in various bulk metallic materials. This paper presents several results from recent investigations of SPD materials focussing on two main objectives: modelling and experimental works on SPD techniques, namely equal-channel angular (ECA) pressing, high pressure torsion (HPT) and their modifications aiming to produce homogeneous nanostructures in bulk large-size billets and to process hard-to-deform and low-ductile materials, e.g. Ti, W and their alloys; determination of critical SPD (strain amount, temperature, applied pressure etc.) and microstructure (types of grain boundaries, defect structures) parameters resulting in enhancement of properties in as-processed materials. Examples of obtaining very high strength and ductility, enhanced superplasticity and toughness in SPD materials are considered and their origin is discussed.

2:25 PM Invited

Equal Channel Angular Pressing of Steels (BCC), Al Alloys (FCC) and Pure Titanium (HCP): *Dong Hyuk Shin*[1]; Si-Young Chang[1]; Yong-Seog Kim[2]; Kyung-Tae Park[3]; [1]Hanyang University, Dept. of Metal. & Matls. Sci., Ansan, Kyunggi-Do 425-791 Korea; [2]Hong-Ik University, Dept. of Metal. & Matls. Sci., Seoul 121-791 Korea; [3]Taejon National University of Technology, Dept. of Adv. Matls. Sci. & Eng., Taejon 300-717 Korea

Several aspects of severe plastic deformation (SPD), such as grain refinement, microstructural modification, superplasticity and mechanical properties, etc, of steels, aluminum alloys and pure titanium, were examined. The grain refinement process of steels and aluminum alloys during SPD appeared to be due to slip band formation, but that of titanium seemed to be entirely different. In addition, SPD was found to be very effective in modifying microstructure. In the low carbon steels, for an example, a spheroidization of rod-like pearlitic cementite was enhanced and the spheroidized cementite particles were distributed uniformly in the ferrite matrix by SPD followed by static annealing treatment. The morphological change in the low carbon steels during SPD was discussed based on dislocation-cementite interaction. The mechanical properties of the severely deformed FCC and BCC metals were measured and discussed with slip systems operating in each class of metals. Finally, the grain refinement mechanism during severe plastic deformation was analyzed on the basis of observations regarding the formation of deformation bands associated with slip systems and their interactions.

2:45 PM

Nanostructures and their Evolution in Copper Processed by Repetitive Corrugation and Straightening (RCS): *Yuntian T. Zhu*[1]; Jianyu Huang[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87544 USA

A new Severe Plastic Deformation (SPD) process, Repetitive Corrugation and Straightening (RCS), has been developed to create bulk, nanostructured copper. In this presentation, the microstructures of copper processed by RCS will be discussed. HRTEM on RCS-processed Cu shows the existence of non-equilibrium grain/subgrain boundaries, a microstructural feature in SPD-produced nanomaterials that has been controversial. TEM observation revealed some unique microstructural features not observed in other deformation modes. The microstructural evolution during the RCS mode is compared with those during rolling and fatigue deformation modes.

3:00 PM Invited

Equal Channel Angular Processing of Magnesium Alloys: *Sean R. Agnew*[1]; G. M. Stoica[2]; L. Chen[2]; T. M. Lillo[3]; J. Macheret[3]; Peter K. Liaw[2]; [1]Oak Ridge National Laboratory, Oak Ridge, TN USA; [2]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996 USA; [3]Idaho National Engineering and Environmental Laboratory, Idaho Falls, ID USA

Equal channel angular (ECA) processing offers the potential to introduce very large strains into a workpiece without changing its cross-section. Hence, it is an attractive technique for developing homogeneous fine-grained metals with good forming characteristics. Magnesium alloys are ideal candidates to benefit from the ECA process, because they exhibit poor low temperature forming characteristics due to a hexagonal close packed crystal structure. Magnesium has a low melting point, which enables its alloys to be processed isothermally at temperatures of 325°C and below. An assessment of the technique has been made with three commercial magnesium alloys ZK60, WE43 and AZ31 and one experimental alloy Mg-4wt%Li. The ductilities of the former two alloys, which contain Zr, were substantially improved. The latter two alloys, which do not contain Zr, showed little change in their properties for the processing conditions explored. Relative to the as-received material, the ductility of ZK60 was improved by 2 to 3 times over the entire temperature range investigated (24-450°C); with up to 350% elongations. Explanations for the property enhancements are discussed in terms of microstructure observations and deformation mechanisms.

3:20 PM

Syntheses of Nd2Ti2O7/Al2O3 Nanocomposites by Spark-Plasma-Sintering and High-Energy Ball-Milling: *Guodong Zhan*[1]; Joshua D. Kuntz[1]; Julin Wan[1]; Javier Garay[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. & Matl. Sci., One Shields Ave., Davis, CA 95616 USA

Al2O3/3mol%Nd2Ti2O7 nanocomposites with different crystal structure of starting alumina nanopowders have been successfully consolidated to > 98% of theoretical density by SPS at relatively low temperatures. High-energy ball milling can decrease the agglomeration of nanoscaled powders and lead to the gamma-Al2O3 to alpha-Al2O3 phase transformation during milling and then enhance the densification at lower temperatures. The 3mol%Nd2Ti2O7/Al2O3 nanocomposite through high-energy ball-milling of gamma-Al2O3 nanopowder could be consolidated by SPS at a temperature as low as 1050°C and the resultant microstructure consists of ultrafine equiaxed grains with an average grain size of ~200 nm. However, the sintering temperature for 3mol%Nd2Ti2O7/Al2O3 nanocomposite without high-energy ball-milling of alpha-Al2O3 nanopowder was 50°C higher than that for composite by high-energy ball-milling and grain size was up to 280 nm. These results suggest that the combination of spark-plasma-sintering and high-energy ball milling could result in ultrafine matrix composites. This investigation was supported by a grant #G-DAAD 19-00-1-0185 from US Army Research Office.

3:35 PM

Evolution of Microstructure and Mechanical Behavior of Titanium during Warm Multiple Forging: *G. A. Salishchev*[1]; S. V. Zherebtsov[1]; R. M. Galeyev[1]; [1]Russian Academy of Sciences, Inst. for Metals Superplasticity Problems, Khalturina str. 39, Ufa 450001 Russia

Along with ECAP the multiple forging (MF) can be used in producing bulk ultrafine-grained materials. MF of titanium is carried out at temperatures of warm deformation (0.34-0.37Tm) in a die set of special design. The formation of ultrafine-grained structure in titanium during MF at 400°C (0.35Tm) and strain rate of about 10-3 s-1 was studied. The flow stress curve was plotted up to cumulative strains around 6. The curve shows maximum in stress at strains around 2 followed by a minor strain softening. The transformation in structure occurring at low to moderate strains is connected with formation of low angle grain boundaries inside of initial grains. As strain rises their misorientation increases up to high angle that results in formation of uniform structure with the grain size of about 200 nm. The mechanisms of ultrafine-grained structure formation as well as the relationship between microstructures, textures and mechanical properties are discussed.

3:50 PM Break

4:00 PM Keynote

Influence of Processing Route on Microstructure and Grain Boundary Development during Equal-Channel Angular Pressing of Pure Aluminum: *Terry R. McNelley*[1]; Douglas L. Swisher[1]; Zenji Horita[2]; Terry G. Langdon[3]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]Kyushu University, Dept. of Matls. Sci. & Eng., Fac. of Eng., Fukuoka 812-8581 Japan; [3]University of Southern California, Dept. of Matls. Sci., Los Angeles, CA 90089-1453 USA

High-purity (99.99%) aluminum was subjected to equal-channel angular pressing (ECAP) and then analyzed by orientation imaging microscopy (OIM) and backscatter electron (BSE) imaging methods. The analysis of microtexture and microstructure by OIM will be reviewed. The ECAP was conducted at room temperature with a die having a 90° angle between the die channels. Repetitive pressing operations were accomplished by either route A, BC or C. Billets were examined after one pass and after four or twelve passes by each of the ECAP routes. The microstructure after one pressing operation consisted of inhomogeneous, elongated grains that aligned with the shearing direction. The grain boundary disorientation distribution obtained by OIM, exhibited a peak at 2°-5°, indicating a deformation microstructure. After four pressings by each route, OIM and BSE data indicated the microstructures had become homogenized and exhibited a similar (sub)grain size (~1.3 mm). Elongation and alignment of the (sub)grains with the shearing direction of the last pressing operation was observed in all cases. The corresponding OIM disorientation distributions showed a significant decrease in the population of low (2°-5°) angle boundaries with a corresponding increase in the high (>15°) angle boundary fraction. The texture data from each route were similar in having only one or two dominant orientations, while the exact texture varied from route to route. Following twelve pressing operations, all routes produced a material with apparent further grain refinement (~1 mm) and an increase in high angle boundary population, while retaining a significant fraction (>10%) of low (2°-5°) angle boundaries. The preferred orientations in the material became more diffuse with repetitive pressings but all routes exhibited a <111> near the shear plane of the final pressing. After twelve ECAP passes, the microstructure comprised a mixture of deformation and recrystallization features regardless of the process route.

4:25 PM Invited

Mechanisms of Formation of Submicron Grain Structures during Severe Deformation Processing: *Philip Prangnell*[1]; Jacob Bowen[2]; Ali Gholinia[3]; [1]UMIST, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2] Risø National Labs, Matls. Dept., Roslkilde DK-4000 Denmark; [3]NIMR, Rotterdamseweg 137, PO Box 5008, Manchester 2600GA Netherlands

Severe deformation processing is an emerging method for the production of submicron grain structures in conventional alloys. By severely deforming metallic alloys to very high plastic strains materials can be produced with grain sizes as fine as 30 nm, containing mainly high angle grain boundaries (HAGBs). In this paper the structure of severely deformed alloys and the mechanisms by which grain refinement takes place are reviewed. Grain refinement occurs through grains subdividing by new HAGBs being formed discontinuously, on finer and finer length scales, until a limit is reached where the grain size merges with the subgrain size, which is the smallest available length scale within which dislocations can be emitted and adsorbed. The formation of new HAGBs is a natural consequence of crystal plasticity, that is initiated by grains splitting into deformation bands, and is promoted by heterogeneity and instability in plastic flow.

4:45 PM Invited

Surface Mechanical Attrition Induced Nanostructured Surface Layer on Metallic Materials: *Ke Lu*[1]; *Gang Liu*[1]; Jian Lu[2]; [1]Shenyang National Laboratory for Material Sciences, Inst. of Metal Rsrch., Chinese Acad. of Scis., Shenyang 110016 China; [2]LASMIS, Univ. of Tech. of Troyes, Troyes 10000 France

In the past decade, nanocrystalline (nc) materials have drawn much attention due to their novel properties and special microstructures. Meanwhile more and more experimental results indicated that nc materials possess some superior properties and performances compared with those of the conventional coarse-grained counterparts, synthesis of bulk nc materials for technological applications, nowadays, is still facing serious difficulties. In this work, we will present the synthesis of a nc surface layer on metallic materials (including pure metals and alloys) by means of the surface mechanical attrition (SMA) technique. Preliminary experimental results will be shown on microstructure characterization of the mechanically-induced nc surface layer and properties (mechanical, wear and friction, corrosion) for the treated materials. The micromechanism and processing techniques for realizing the surface nanocrystallization process will be discussed. Further development and prospects will be addressed with respect to the performance and technological applications of the engineering materials with a nc surface layer.

5:05 PM

The Effect of Strain Per Pass on the Microstructure Developed in Aluminum Processed by Equal Channel Angular Extrusion: *Pei-Ling Sun*[1]; P. W. Kao[1]; C. P. Chang[1]; [1]National Sun Yat-Sen University, Inst. of Matls. Sci. & Eng., Kaohsiung 804 Taiwan

The die angle used in equal channel angular extrusion (ECAE) determines both the shear plane orientation and the strain per pass. It is interesting to separate the effect of the shear plane orientation and the strain per pass on microstructural development. By rotating the billet 180° about the extrusion axis between passes (route C), the shear plane is kept unchanged during straining. Then the effect of strain per pass can be studied by using different die angles. In the present study, commercial pure aluminum was deformed by ECAE route C with two different die angles (90° and 120°). The deformed microstructure was characterized by TEM and the boundary misorientations were determined from TEM Kikuchi diffraction.

5:20 PM

Microstructure Evolution in Nanocrystal Formation during Ball Milling: *Zhiguang Liu*[1]; Jun Yin[2]; Yan Xu[1]; Koichi Tsuchiya[1]; Minoru Umemoto[1]; [1]Toyohashi University of Technology, Dept. of Production Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [2]Institute of Metal Research, 72 Wenhua Rd., Shenhe, Shenyang, Liaoning 110016 China

Nanocrystal formation by ball milling in pure Fe and eutectoid carbon steels has been studied. It was found that nanocrystalline ferrite (NF) first occurred as band structure at certain outer surface areas of powders at the early stage of ball milling due to deformation localization, while the interior remains the starting structure with much weaker deformation (coarse-grained region, CGR). The boundaries between NF and CGR were observed to be either quite clear or gradually changed. At least three types of microstructures were observed: coarse grains with either high density of dislocations or dislocation cells inside, layered nanocrystalline ferrite with a thickness ranging from 100 to less than 10 nm, and equiaxed nanocrystalline ferrite with a grain size of 5-10 nm. It has been suggested that a transition from dislocation cell wall created by work hardening during ball milling to grain boundary may be responsible to the observed nanocrystalline ferrite formation.

5:35 PM

Ultrafine Grain Formation during Equal Channel Extrusion in an Al-Mg-Sc Alloy: *Oleg Sitdikov*[1]; *Rustam Kaibyshev*[1]; Semeon Olenyov[1]; Gunter Gottstein[2]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia; [2]Institute of Metal Science and Physics of Metals, RWTH, Kopernikusstrasse, 14, Aachen D-52056 Germany

Microstructure evolution in an Al-6%Mg-0.3%Sc alloy during intense plastic straining has been studied at T=250°C. Equal channel angular extrusion (ECAE) was carried out to total strains above 12. TEM observations showed that plastic deformation to e=1 results in the formation of areas of equiaxed subgrains which alternate with bands of dense dislocation walls. Following strain leads to transformation of dense dislocation walls into low-angle boundaries and an increase in misorientation of deformation induced boundaries. Low-angle boundaries gradually transform into high-angle boundaries with strain. Ultra-fine grains were revealed after e=2 within areas of equiaxed subgrains. Volume fraction of recrystallized grains tends to increase with strain. It can be concluded that continuous dynamic recrystallization (CDRX) occurs. The main feature of CDRX in the Al-Mg-Sc alloy is low rate of dislocation rearrangements and low mobility of deformation induced boundaries. The role of Al3Sc dispersoid particles in CDRX is discussed.

Second International Symposium on Ultrafine Grained Materials

Poster Session: Monday – Wednesday

Deformation Induced Ferrite Transformation and Grain Refinement in Steels: *Han Dong*[1]; [1]Central Iron and Steel Research Institute, Div. of Structl. Matls., Beijing 100081 China

Austenite to ferrite transformation in steels is one of the typical diffusion transformation which is encountered in a variety of structural steels. For commercial structural steels, the diving force for austenite to ferrite transformation, free energy, is dependent on chemical composition and temperature. For deformed austenite, the accumulative deformation energy could contribute to transformation driving force which results in the promotion to austenite to ferrite transformation if deformation energy could not be released through recrystallization. This kind of transformation is named as deformation induced ferrite transformation (DIFT). Ferrite grain through DIFT is much finer compared with conventional transformed ferrite grain. The accumulative energy in austenite mainly depends upon chemical composition, deforming temperature, strain, strain rate, sample dimensions, and loading manner, etc. Uniformity dispersion of ultra-fine ferrite grains induced by DIFT can be controlled through variables to change accumulative energy. By adopting the theory of DIFT, mean grain size of 4m in diameter can be obtained in plain low carbon steel, and corresponding mean grain size of 1m in diameter can be achieved in microalloyed steel. It is proved that DIFT is a very effective method for grain refinement in steels.

Strength of Submicrocrystalline Severely Deformed Commercial Aluminum Alloys: *Mikhail Vyacheslavovich Markushev*[1]; Maxim Yurievich Murashkin[1]; [1]Institute for Metals Superlasticity Problems of the Russian Academy of Sciences, Sector 27, Khalturin St. 39, Ufa, Bashkortostan 450001 Russia

Understanding the mechanical behavior of submicrocrystalline (SMC) materials processed by severe plastic deformation (SPD) is of great importance. The data on ambient temperature tensile properties of a range of commercial aluminum alloys will be reviewed. The influence of alloy composition, deformation technique (complex and equal-channel angular extrusion) and treatment mode will be discussed. The properties of SMC deformed and annealed conditions will be compared with standard values for conventionally strengthened products. It is shown that SPD could be effective to improve static strength of non-heat treatable and low-alloyed heat treatable alloys. For better realization of the effect the alloy should be processed by complex treatment, involving angular extrusion, conventional work hardening and heat treatment. The combination of strength and ductility achievable is comparable or even better to that found with many high-strength precipitation-hardened alloys. For most heat treatable alloys the use of SPD is ineffective.

On the Properties of Severe Plastic Deformations under Twist: *Yan Beygelzimer*[1]; Viktor Varyukhin[1]; Dmitriy Orlov[2]; [1]Donetsk Physical & Technical Institute NASU, R. Lyuksembourg 72, Donetsk 83114 Ukraine; [2]Donetsk State Technical University, Donetsk Ukraine

One of the most effective methods for obtaining bulk ultrafine grained materials is severe plastic deformation that is typically done using equal channel angular pressing. In this work we describe and analyze a new method for obtaining severe plastic deformations proposed by the first author. The method is based on the direct extrusion and was termed "twist extrusion". The main idea of the method is to extrude a prism bulk through a matrix with a twist channel. The cross-section of the channel that is orthogonal to the pressing axis, is constant along this axis. In the general case, it is possible to have channels of quite arbitrary cross-section. Several variations of twist extrusion corresponding to known direct extrusion schemes are also possible; for example, twist extrusion in a container with a thin lubrication layer, hydrostatic or hydromechanical twist extrusion. We argue that in some cases twist extrusion has certain advantages. In particular, it allows to obtain not only bulks, but also ready-to-use products in the form of a prism with an arbitrary cross-section.

Friction Stir Processing to Achieve Fine Grains in Hexagonal Metals: *David J. Alexander*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-6, MS G770, Los Alamos, NM 87545 USA

Friction stir processing is being applied to several hexagonal metals, to determine whether this method can be used to achieve fine-grained material. The intended application is for Be alloys to be used for fabrication of targets for the National Ignition Facility. Because of the difficulties in working with Be due to environmental and safety concerns, Mg and Ti have been used as surrogate materials for preliminary investigations. Information about initial results of friction stir processing of these hexagonal metals will be presented.

Ultrafine-Grained Tungsten Produced by SPD Techniques: *Igor V. Alexandrov*[1]; Georgy I. Raab[1]; Vil U. Kazyhanov[1]; Larissa O. Shestakova[1]; Ruslan Z. Valiev[1]; Robert J. Dowding[2]; [1]Institute of Physics of Advanced Materials, Ufa State Aviation Tech. Univ., 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia; [2]US Army Research Laboratory, AMSRL-WM-MD, Aberdeen Proving Ground, MD 21005-5066 USA

The paper deals with the recent development of producing ultrafine grains in tungsten, using two SPD techniques, i.e. high pressure torsion (HPT) and equal channel angular pressing (ECAP). Tungsten is a typical low ductile and hard-to-deform metal and its SPD processing is a new and complex task. The application of HPT enables us to develop the ultrafine-grained (UFG) microstructure with a grain size of about 100 nm in the disk shape of a diameter about 10 mm. As a result, the microhardness has been increased almost twice. An investigation of the dependence of the accumulated strain, pressure and temperature on the features of the processed UFG microstructures and the final grain size has been performed. The regimes of equal channel angular pressing resulting in the formation of the UFG microstructures with the average grain size of about 1 mm in bulk rod shape ingots have been developed. It has been shown that further grain size refinement can be achieved by a variation of the strain rate and the temperature of ECAP as well as of the applied back pressure. The influence of the grain size on the strength and ductility of the commercial pure W samples has been established. A strong dependence of the brittle-to-dictile transition on the microstructure features has been revealed.

Effects of Hot Deformation on Austenite Transformation in HSLA Steel: *Yang H. Bae*[1]; Soon H. Hong[1]; Dong-Han Seo[2]; Jong-Kyo Choi[2]; Woong Y. Choo[2]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Kusung-dong, Yusung-gu, Taejeon 305-701 Korea; [2]POSCO, Tech. Rsrch. Labs., PO Box 36, Pohang 790-785 Korea

This study investigates the effects of hot working and isothermal heat treatment on austenite/ferrite phase transformation of Fe-0.2C-0.4Si-1.1Mn low alloy steel. The hot working process is known as the severe plastic deformation being employed for the purpose of refining the ferrite grain size of high strength low alloy(HSLA) steels. The alloy steel used in this study was manufactured by vacuum induction melting and followed by sizing process for the steel plate with a thickness of 15mm. The alloy steel specimens were austenized at the temperature of 950°C and then cooled with 40°C/s to several selected hot working temperatures ranged from 600°C to 800°C. It is shown from the experimental results that the volume fraction of the ferrite increased with increasing the isothermal heat treatment time and also with increasing the amount of hot working. It is also noted that the ferrite grain size decreased from 30μm to 5μm through the hot working process at the temperature of 775°C. By comparing the normalized TTT diagrams for both deformed and not-deformed specimens, it is concluded that the austenite/ferrite phase transformation kinetics mainly depend on the γ/γ grain boundary area.

Changing Titan Structure under Twist Extrusion: Yan Beygelzimer[1]; *Viktor Varyukhin*[1]; Dmitriy Orlov[2]; Boris Efros[1]; [1]Donetsk Physical & Technical Institute NASU, R. Lyuksembourg 72, Donetsk 83114 Ukraine; [2]Donetsk State Technical University, Donetsk Ukraine

We present some experimental results describing titan evolution during severe plastic deformation by twist extrusion (TE). In particular, we show that increasing the number of iterations of TE leads to the decrease of the grain size d in both orthogonal and parallel cross-sections of the bulk. For example, if in its original state titan had d~25mkm, then after three TE iterations, we had d<=1mkm. Furthermore, the bulk is deformed sufficiently uniformly across the entire cross-section. The micro-hardness H_mu of titan grows monotonically with the increase of the number of iterations. For example, if in undeformed state titan had H_mu~1720MPa on average, then already after the first iteration the average increased to 1990MPa. At the same time, the intensity of hardening gradually decreases and, after a large number of iterations, practically approaches zero. The structure and mechanical properties of titan after TE are compared against the analogous characteristics after equal-channel pressing.

Phase Transformations in Ultrafine Grained Fe and Fe-Mn Alloys: *Boris Efros*[1]; [1]Donetsk Physical & Technical Institute NASU, High Pres. Phys. & Adv. Tech., 72 R. Luxembourg St., Donetsk 83114 Ukraine

Influence pressure on structure-phase state of ultrafine-grained (UFG) iron and Fe-Mn alloys has been studied. Alloys with wide concentration of manganese (from » 0 to 55 wt. %), having in initial state BCC, HCP and FCC (stable and metastable state) phases were as the objects under investigation. The complex study of iron and Fe-Mn alloys structures, deformed by severe plastic deformation (SPD) under pressure, including the UFG structures, by methods of X-ray diffraction (XRD), transmission electron microscopy (TEM), Mössbauer spectroscopy (MOSS) and optical microscopy (OM) has been carried out. There is the range of pressure P » 0.1MPa - » 60 GPa and logarithmic deformation degree e = 0 - » 10 of studied alloys in experiments. In work is shown, that the influence the UFG structure has an effect in increase of forward-alpha-epsilon and reduction of reverse-alpha-epsilon on » 4 GPa in iron. Also is established, that the increase of pressure till 19-20 GPa in the UFG structure of Fe-Mn alloys in hcp of phase area lowers of reverse-gamma-epsilon so, that allows to stabilize 100% of a phase of high pressure under normal conditions and long endurance after removal of pressure. The research of temperature stability of a phase of high pressure has shown that the heating to temperature is higher 790 K initiates reverse transformation. For reception stable (under normal conditions) the FCC phase need to be sustained in an interval of temperatures 880-930 K during 5 minutes. Thus the obtained results of complex investigation (XRD, TEM, MOSS, OM) of studied metals and alloys in the UFG state have revealed the strong dependence of phase solid transformations on parameters of SPD and/or pressure and initial phase, structure state and concentration of Mn in alloys.

Determination of Dynamic Ferrite Transformation Kinetics during Deformation in Austenite: *Seung Chan Hong*[1]; Sung Hwan Lim[1]; Kyung Jong Lee[1]; Kyung Sub Lee[1]; [1]Hanyang University, Matls. Sci. & Eng., Haengdang-dong, Seoungdong-ku, 17, Seoul 133-791 Korea

The dynamic ferrite transformation during heavy deformation has been studied to obtain the ultrafine-grained structure in steel. In this study, the flow stress was measured at various temperatures with up to 1.2 of true strain and constant strain rate (10/s) by Gleeble 1500 in order to determine dynamic ferrite transformation kinetics quantitatively. The formation of dynamic ferrite decreased the flow stress during hot deformation in austenite. The flow stress was partly attributed by the flow stress of austenite and that of ferrite. Therefore, the fraction of dynamic ferrite was determined as follows; Xc = (óm-óa)/(óf-óa) Where, Xc is the calculated dynamic ferrite fraction, óm is the measured flow stress, óa is the extrapolated flow stress of austenite and óf is the extrapolated flow stress of ferrite. It was also confirmed that the critical strain of dynamic ferrite at high cooling rate to deformation temperature was shifted to lower strain compared to that at low cooling rate with the aid of increasing undercooling.

Nanostructure Formation and Carbides Dissolution in Rail Steel Deformed by High Pressure Torsion: Yu. V. Ivanisenko[1]; R. Z. Valiev[2]; W. Lojkowsky[3]; A. Grob[4]; H.-J. Fecht[4]; [1]IMSP RAS, Khalturin 39, Ufa 450001 Russia; [2]IPAM Ufa State Aviation Technical University, Ufa 450000 Russia; [3]High Pressure Research Centre PAS, Sokolowska 29, Warsaw 01-142 Poland; [4]Ulm University, Div. of Matls., Albert Einstein Allee 47, 89081 Ulm, Germany

The microstructure and the phase composition evolution of commercial UIC 860 steel during the high pressure torsion (HPT) were investigated by using transmission electron microscopy, thermomagnetic analysis and X-ray diffraction. Upon strain degree increasing, grain refinement up to 20 nm takes place and total cementite dissolution occurs. In the nanocrystalline state the steel is characterised by a maximum level of hardness (9.7 GPa), which is 2.5 times higher than that of the initial state. It was revealed, that the deformation is first concentrated in the soft phase-ferrite, where the cell structure forms. In the end a very high density of the defects, such as grain and cell boundaries, dislocations and vacancies achieves. The partial cementite dissolution starts already at the early stages of the HPT deformation and develops gradually after beginning of nanostructure formation in ferrite phase.

Formation of Nanocrystalline Structure in a Ni-20%Cr Alloy: *Rustam Kaibyshev*[1]; Nadia Dudova[1]; Vener Valitov[1]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Microstructure evolution resulting in nanocrystalline structure was examined during plastic deformation of a Ni-20%Cr alloy at a temperature of 500°C (0.46Tm) both in compression and torsion. It was shown that a low energy dislocation structure evolves at initial stage of plastic deformation. Nanoscale grains are formed at a strain of about 1.2. Their size was 80 nm and 50 nm in samples subjected by compression and torsion, respectively. Initial boundaries and areas of intersections of deformation bands play a role of nucleation sites. Following deformation leads to increase in volume fraction of nanoscale grains, and fully grained structure was revealed after e=5.5. Nanocrystalline structure evolved is characterized by high value of microhardness (4.6GPa) and increased internal elastic strain. An effect of deformation scheme on formation of such a structure is discussed.

Formation of Ultrafine Grained Structure in an Al-Li Alloy during Intense Plastic Straining at Elevated Temperature: *Rustam Kaibyshev*[1]; *Fanil Musin*[1]; Ksenia Saytaeva[1]; Yoshinobu Motohashi[2]; [1]Institute for Metals Superplasticity Problems of RAS, Khalturina 39, Ufa 450001 Russia; [2]Ibaraki University, Rsrch. Ctr. of Superplasticity, Nakanarusawa-cho, 4-12-1, Hitachi, Ibaraki 316-8511 Japan

Microstructural evolution of 1421 aluminum alloy (Al-4.1%Mg-2.0%Li-0.16%Sc-0.07%Zr in wt. pct) has been studied at T=400°C and at a strain rate of about 10-2 s-1. The 1421 alloy was subjected to equal-channel angular extrusion (ECAE) up to true strain of 12. It was shown that continuous dynamic recrystallization occurs in the 1421 alloy and results in the formation of ultrafine grained structure. After e=1, the arrays of low-angle boundaries are evolved. Gradual transformation of low-angle boundaries into high-angle boundaries takes place with strain. First recrystallization grains with a size of about 1µm were revealed after e=2. Following deformation leads to increasing volume fraction of recrystallized grains which attends about 80 pct at e=12. In the same time, the deformation induced microstructure is non-uniform. At e=8, the bands of recrystallized grains alternate with bands of elongated subgrains. Mechanisms of ultrafine grain formation are discussed.

Structure and Properties of Commercial Aluminum Alloy 1560 (Al-Mg-Mn) after Severe Plastic Deformation and Annealing: Maxim Yurievich Murashkin[1]; *Mikhail Vyacheslavovich Markushev*[1]; [1]Institute for Metals Superplasticity Problems of the Russian Academy of Sciences, Sector 27, Khalturin St. 39, Ufa, Bashkortostan 450001 Russia

Specific features of microstructure and crystallographic texture of the 1560 (Al 6.0Mg 0.6Mn) alloy after severe plastic deformation (SPD) via equi-channel angular extrusion and annealing are considered. The phenomenology and nature of processes occurring at transformation of the submicrocrystalline deformation structure to the grain one are discussed. Mechanical behavior of the severely deformed and annealed alloy at tension and bending at room temperature is investigated. The influence of a structural state on a serrated flow, strength, hardness, ductility toughness and crack resistance (crack formation and crack growth resistances) is shown. The reasons for improved strength and low crack resistance of the alloy after SPD are analyzed. Based on the evaluation of microcrack formation intensity, the analysis of deformation relief and fracture, the origin of plastic flow and failure of the alloy having submicrocrystalline deformation, submicrocrystalline grain and microcrystalline structure is discussed.

Structure Evolution of Ultrafine Grain Copper and Nickel at Plastic Deformation: *Eduard Victorovich Kozlov*[1]; Anatolii Nikolaevich Zhdanov[1]; Lina Nikolaevna Ignatenko[1]; Nina Alexsandrovna Koneva[1]; [1]Tomsk State University of Architecture and Building, Phys. Dept., Solyanaya Sq. 2, Tomsk 634003 Russia

The structure of nanocrystalline materials determines mechanisms of plastic deformation. With the grain size of hundreds of nanometers, the main deformation mechanisms are intergranular dislocation glide and grain boundary sliding. Under conditions of self-coordination of intergranular glide and grain boundary sliding, dislocations are not retained inside grains, but get pulled in the boundaries. In the work the structure evolution of ultrafine grained copper, nickel and alloys on their base at active plastic deformation and creep is investigated. The behavior of grain structure, grain boundaries and distributions of grain sizes on deformation degree is studied. The changes of scalar dislocation density, amplitude of internal stresses are measured. The estimation of shear value on grain boundaries and the contribution of this shear in total deformation are carried out. The behavior of second phase particles is watched. It is paid attention to contributions in plastic deformation of grains of different sizes.

The Use of SPD for the Fabrication of Nanostructured Materials for Ball-Milled Powders: *Georgy I. Raab*[1]; Nickolai A. Krasilnikov[1]; Robert Klemm[2]; Ellen Thiele[2]; Vladimir S. Zhernakov[1];

[1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Metals, 12 K. Marks Str., Ufa, Bashkortostan 450000 Russia; [2]Technical University Dresden, Inst. for Physl. Metal., Zellescher Weg 17, Dresden D-01097 Germany

Bulk nanostructured samples with high density were obtained from nanocrystalline Cu, Fe and Ni ball-milled powders using high pressure and severe plastic deformation (SPD). The process of the fabrication of the samples consists of a preliminary compacting and a following SPD, including such methods as high pressure torsion (HTP), equal channel angular pressing (ECAP) and multiforging under high pressure. The advantage of the suggested approach consists in the possibility of the fabrication of compacts with a density >98% and a nanocrystalline structure due to the decrease in the temperature of SPD consolidation under high pressure. Thus, the grain size of HTP Cu and Fe with a diameter of 20 mm and thickness of 2 mm was 100 mm. The Ni samples with a cross-section of 5x5 mm2 and a length of up to 50 mm having a grain size less than 100 nm were obtained by ECAP and multiforging. The samples obtained are characterized by a high strength, a homogeneous microstructure in the whole volume and high thermal stability. Meanwhile, some increase in die durability at low homologous temperature and elevated pressure remains the technical problem of the consolidation of nanostructured samples from powders. The technological aspects of the fabrication of high density samples from nanostructured powders by the applied methods, microstructural data and the mechanical behavior of the processed materials are discussed.

Formation of Nanocrystalline Structure in Two-Phase Titanium Alloys by Combining Thermohydrogen Processing and Warm Severe Plastic Deformation: *G. A. Salishchev*[1]; M. A. Murzinova[1]; S. V. Zherebtsov[1]; R. M. Galeyev[1]; O. R. Valiakhmetov[1]; [1]Russian Academy of Sciences, Inst. for Metals Superplasticity Problems, Khalturina str. 39, Ufa 450001 Russia

Warm severe plastic deformation realized via multiple forging can be used for formation of fine-grained microstructure with of a grain size of several hundred nanometers or less in titanium alloys. The less grain size the better superplastic properties resulted from lower temperature deformation. It has been established that the least grain size not only depends on the deformation temperature, but also the phase volume fraction, phase particle size and interparticle distance. The application of thermohydrogen treatment improves hot workability of titanium alloys at lower temperatures and permits to combine the deformation with metastable phases decomposition. Additional grain refinement is observed after hydrogen removal during vacuum annealing due to polymorphic transformation accompanied by recrystallization. The microstructure with a grain size of 25 nm was obtained in two-phase titanium alloy Ti-6.3Al-3.5Mo-1.7Zr wt.%. This alloy is superplastic at 550°C and e. =2¥10?4 s-1: m=0.52, relative elongation achieves 550%.

Nanostructured Metastable Alloys Processed by Severe Plastic Deformation: *Vladimir Vladimirovich Stolyarov*[1]; Ruslan Zufarovich Valiev[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., K. Marx, 12, Ufa 450000 Russia

Severe plastic deformation (SPD) during processing two-or multiphase alloys has an ability in both strongly refine microstructure till up nanometer range and change essentially a phase composition forming highly metastable states. As a result, there is a potential to achieve new and extraordinary properties in these alloys. This paper demonstrates examples of attaining such properties in several Ti, Al and Fe-Pr(Nd)-based alloys using SPD processing by high pressure torsion (HPT) and equal channel angular pressing (ECAP). It is shown that for microstructures of the SPD materials it is typical to have a presence of not only small grain sizes, but also a formation of supersaturated solid solutions and amorphyzation of intermetallic phases alloys. During further heating aging effects and reordering take place and processed alloys demonstrate unique mechanical (strength and superplasticity) and magnetic properties.

Tensile and Fatigue Properties of Al-Mg-Sc-Zr Alloy Fine-Grained by Equal-Channel Angular Pressing: *Alexei Vinogradov*[1]; Vladimir I. Kopylov[2]; Atsuo Washikita[3]; Kazuo Kitagawa[3]; [1]Osaka City University, Dept. of Intelligent Matls. Eng., Osaka 558-8585 Japan; [2]Physico-Technical Institute of the National Academy of Science, Minsk 220730 Belarus; [3]Kanazawa University, Dept. of Mech. Sys. Eng., Kanazawa 920-8667 Japan

A significant material hardening is observed after equal-channel angular pressing (ECAP) so that an apparent yield stress approaches an ultimate tensile strength leaving for the material a very limited number of ways to accommodate external strains during loading after fabrication. Although it has been emphasized that the ductility of ECAP materials is rather high, special conditions such as those at superplasticity are often needed to demonstrate that high ductility. At room temperature most ECAP metals are less ductile than their coarse-grain counterparts. Although the higher yield stress may result in the improved fatigue limit, the low-cyclic fatigue performance degrades owing to reduced ductility. Using the Al-Mg-Sc-Zr alloy, it is shown that ECAP is capable of fabricating materials with enhanced both low- and high-cyclic fatigue properties. The ECAP Al-Mg-Sc-Zr demonstrates notable strain hardening during tensile testing and significant improvement of fatigue life in comparison with other commercial middle-strength Al-alloys.

Microstructure of a Low Carbon Steel after Equal-Channel Angular Pressing: Jingtao Wang[1]; Xicheng Zhao[1]; Yuan Wang[1]; Lizhong Wang[1]; Terence G. Langdon[2]; [1]Xi'an University of Architecture & Technology, Sch. of Metlgcl. Eng., Yanta Rd., Xi'an 710055 China; [2]University of Southern California, Dept. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-Channel Angular pressing (ECAP) was successfully carried out on a low carbon steel at room temperature up to an equivalent true strain of ~11 via route C. Strongly banded structure was observed on the cross section after ECAP. The grain size was refined from 25 micrometers in the as-received hot rolled plate to a band width of ~0.2 micrometer after equal channel angular pressing to an equivalent true strain of ~11. The length of the grain fragment in the strongly banded structure also decreases with the increase of ECAP strain, and reaches a value of less then ~0.6 micrometer when the ECAP passage reaches or above. This paper reports investigations on microstructure characterization of the processed low carbon steel.

Synthesis, Microstructure and Characterization of Nanoscale Binary Aluminum Alloys: Jixiong Han[1]; Marty J. Pluth[1]; Jai A. Sekhar[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Cu and Al-Zn alloy particles. Rapid plasma ablation followed by rapid quenching was used to synthesize nanoparticles from Al-4.4 wt.% Cu and Al-15 wt.% Zn alloy ingots and the microstructure and transformation behavior of the particles were studied by transmission electron microscopy. The particles were found to be in the supersaturated state in both types of alloys, but displayed a variation in the individual particle composition when compared with the precursor bulk alloys. After the plasma ablation and quenching process, the particles were exposed to air and were found to contain a 3-5 nm thick adherent aluminum oxide scale which prevented any further oxidation. Several of the particles were faceted and bound by (111) planes. The nanoparticles of the alloys were heat treated in order to examine the precipitation sequence in the Al-Cu alloys and spinodal decomposition in Al-Zn alloys. Differences in the transformation behavior of these alloy particles when compared with bulk alloys of the same composition were observed. In the Al-Cu alloys, precipitates which were considerably enriched in copper were noted at the beginning of the transformation. These subsequently transformed to precipitates with lower copper content, as the heat treatment progressed. In the Al-Zn alloy, a spinodal structure was noted in the nanoparticles, as well as precipitates of pure zinc with a f.c.c. structure upon aging. In both the Al-Cu and Al-Zn alloy particles, the precipitates were observed to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

Shear Banding in Materials: Metals

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

Monday PM — Room: 303
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Todd Hufnagel, Johns Hopkins University, Dept. of Matls. Sci. & Eng., Baltimore, MD 21218 USA; Marc Meyers, University of California-San Diego, Appl. Mech. & Eng. Scis., La Jolla, CA 92093 USA

2:00 PM
Shear Banding in Different Classes of Materials: *Walter W. Milligan*[1]; [1]Michigan Technological University, Matls. Sci. & Eng., Houghton, MI 49931 USA

An overview of the phenomenon of shear banding in various types of materials, as well as the goals of the workshop, will be presented.

2:20 PM
Shear Banding as the Dominant Deformation Mechanism in Nanophase and Ultrafine-Grained Iron: *E. Ma*[1]; D. Jia[1]; Q. Wei[1]; K. T. Ramesh[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. & Eng., Baltimore, MD 21218 USA

Iron samples with a range of grain sizes have been processed by mechanical attrition followed by consolidation. The consolidation was a two-step process. In the first step, the powder was compacted under a pressure of 1.4 GPa at room temperature for about 10 hours using a WC die. In the second step, the compact was transferred into a larger WC die for hot consolidation. Temperatures in the range of 480-680°C and a pressure of 850 MPa were applied to obtain different grain sizes, ranging from sub-micron to ca. 100 nanometer. Samples for mechanical testing were cut from the fully dense compacts using electrical discharge machining (EDM). Compression tests under a quasistatic strain rate were performed with an MTS machine. High strain rate testing was conducted using a miniature Kolsky bar uint (or Desktop Kolsky Bar (DKB)). Strain rates up to ~5 x 10+4 s-1 were obtained with DKB. Shear banding was established as the predominant plastic deformation mechanism under both quasistatic and dynamic conditions when the grain size is below 300 nm. The development of multiple shear bands has been monitored as a function of plastic strain. Transmission electron microscopy (TEM) was used to study the microstructure inside and outside the shear bands. Nano-indentation measurements were carried out to investigate the nano-scale mechanical behavior inside and outside the shear bands.

2:40 PM
Shear Localization and Failure in Pre-Shocked Metals: *Geoffrey H. Campbell*[1]; Mukul Kumar[1]; James S. Stölken[1]; F. Xabier Garaizar[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, MS L-356, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, Computations Direct., PO Box 808 L-561, Livermore, CA 94550 USA

Strain localization is an important phenomena for understanding metal dynamics in general and many types of failure observed in practice. There exists much information on both localization and failure of ductile metals that start with a low defect density; however, less is known about highly defective metals. Shocks induce deformation microstructures with high dislocation densities (>10^12 /cm2) that are comprised, for the most part, of statistically stored dislocations. This highly defective state allows recovery mechanisms to begin at an earlier point in the deformation behavior, reducing the work hardening rate and increasing the propensity for shear localization. We have induced shocks using laser drives in pure Ta, Ta-2.5% W alloy, and pure Cu under a variety of conditions. We have measured their mechanical properties and characterized their microstructures in the pristine, shock processed and mechanically strained conditions with a variety of techniques, including EBSD mapping and TEM. We have also performed plane stress fracture mechanics tests with the double edge notched tension specimen. The mechanical response will be discussed in terms of the observed shear localization behaviors.

3:00 PM
The Shear Band in 7050 Aluminum Alloy: *Shih-Chin Chang*[1]; Yaw-Shing Wang[1]; Yuan-Yuan Lin[1]; [1]National Tsing-Hua University, Dept. of Matls. Sci. & Eng., Hsinchu 30043 Taiwan

In plane strain compressed 7050 aluminum alloy specimens with either T or L direction constrained, macroscopic shear bands were visible on the constrained plane surface of the specimen. The shear bands formed along planes of maximum shear stress which make an angle 45 degree to the S and the non-constrained plane. The maximum shear strain observed in a shear band is 2. It was noticed that the strength of specimens constrained in L direction were higher than that of their counterpart specimens constrained in T direction. For under aged specimen, the shear bands was sharply localized. Specimen will shear rupture along shear bands with a sudden drop of the stress. Similar but wider shear bands were observed in peak and over aged specimens. The width of over aged specimens was the largest. No rupture were observed in over aged specimens even when the true compression strain went up to 0.6 when the test ended. For under aged specimen with T direction constrained, the true strain of rupture is 0.32. For cold rolled specimen, the formation of shear bands occurred at a smaller strain and the true strain of rupture is 0.27. When constrained in L direction, the rupture strain, 0.29 for underaged and 0.125 for underaged and cold rolled specimens was smaller than those in T direction. In contrast to the underaged case, for over aged specimens the stain of shear band formation was larger when constrained in L direction than in T direction. A model based on the dislocation-precipitate interaction was proposed to explain these observations.

3:20 PM Break

3:40 PM
The Effect of Shear Localization on Ductility and Fracture: *Fereshteh Ebrahimi*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

There is a broad range of behaviors that lead to plastic strain localization. Usually, local softening due to microstructural weaknesses (e.g., particle or void shearing, short range disordering), hotness (e.g. high strain rate, cryogenic deformation), inhomogenity of microstructure/composition (e.g. grain size or hydrogen distribution) or crystal rotation (e.g., non-Schmid effects in single crystals) creates the conditions for shear banding. In this presentation examples of shear localization in single crystals (NiAl and a superalloy) and nanocrystals (nickel and copper) are presented. While a low resistance to ductile fracture can be explained by tendencies to shear localization, it is not clear why softening should encourage cleavage fracture. It is shown that indeed cleavage facets develop within the shear bands but on planes that experience the highest tensile stress components. An explanation based on strain incompatibility that leads to the development of high hydrostatic tensile stress components is suggested.

4:00 PM
Shear Band Phenomena in Extreme Deformation Processes: From Ballistic Penetration to Friction-Stir Welding: *Lawrence E. Murr*[1]; Sridhar Pappu[1]; Christine Kennedy[1]; Mario Guerra[1]; [1]University of Texas at El Paso, Metall. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

The interplay between strain and strain rate as well as strain saturation in the context of strain hardening can produce intermittent regions of shear instability or overlapping, broad regimes of adiabatic shear bands which facilitate superplastic, solid-state flow as a consequence of intrinsic, dynamic recrystallization (DRX). During the penetration of ductile targets such as copper by tungsten-heavy alloy (WHA) rods, the target material flows during cratering to form the entrance rim and jetting. Penetration occurs by overlapping shear bands composed of mostly equiaxed DRX grains. Similar features are observed for the penetration of tungsten single crystal rods into RHA steel targets. For 7039 aluminum targets strain-hardened to the maximum, intermittent shear bands combined with a very thin DRX regime at the channel wall facilities WHA rod penetration. Correspondingly, the friction-stir welding (FSW) of the same or dissimilar metals and alloys such as brass to copper or 6061 Al to itself produces intercalations or mixtures of narrow and very wide shear bands which flow and produce residual vortex and other complex flow patterns within the weld zone. Solid material between the shear bands can flow as large blocks. Detailed light and transmission electron microscopy studies confirm that in all of these flow phenomena involving various shear bands DRX provides the fundamental solid state flow mechanism.

Research supported by ARO-DAAG55-97-1-0238 and GSA Grant PF-90-018.

4:20 PM
The Effect of Shear Banding on Ductility and Bendability of Automotive Aluminum Alloys: *David S. Wilkinson*[1]; J. David Embury[1]; David J. Lloyd[2]; [1]McMaster University, Matls. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Alcan International, Kingston R&D Ctr., Kingston, ON K7L 5L9 Canada

Aluminum alloys such as AA5754 and AA6111 have been developed as sheet materials for automotive applications. One of the technical issues controlling the utility of these materials is their bendability, for example in hemming operations. Through extensive work on these alloys we have found that the interaction between shear bands and Fe-aluminide constituent particles plays a critical role in the failure process that controls both tensile ductility (as measured by the reduction in area) and bendability. As the level of Fe (and thus the constituent particle density) increases the failure mode can sometimes change quite dramatically. In this presentation we will present results in which the effect of Fe content has been studied at a range of temperatures in both tension and bending. We will also present data for tensile tests performed under superimposed pressure.

4:40 PM
TEM Study on Microstructural Evolution of Rolled Al-1%Mn Single Crystals of {001}<110> Orientation: Zhengjie Li[1]; [1]Tsinghua University, Dept. of Matls. Sci. & Eng., Bldg. 14# 208, Beijing 100084 China

Aluminum single crystals of {001}<110> orientation have been rolled to 18, 30, 50, 70, 80 and 90% reductions under controlled homogeneous rolling conditions. The evolution of the deformation structure during rolling was investigated using the Transmission Electron Microscopy (TEM) and the local orientations were measured by a simple semi-automatic TEM method. The regions investigated are characterized by two types of bands with sharply alternate orientations and strong TD crystal rotation was observed. Long and Straight dislocation boundaries parallel to the slip planes of {111} (i.e. crystallographic boundaries) are observed at low to medium strains whereas shortened and more or less equiaxed deformation structure are developed at higher strains. A theoretical analysis based on the idea of crystal slip geometry and Frank formula was proposed to explain the microscopic subdivision and local crystallography of crystals with the present orientation.

Surface Engineering: Science & Technology II: Surface Modification Technologies

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

Monday PM	Room: 203
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Ashok Kumar, University of South Florida, Dept. of Mechl. Eng. & Ctr. for Microelect. Rsrch., Tampa, FL 33620 USA; Yang-tse Cheng, General Motors, Matls. & Proc. Labs., Warren, MI 48090 USA

2:00 PM Opening Remarks

2:05 PM Invited
Laser-Based Micro- and Nanoprocessing: *Y. F. Lu*[1]; [1]National University of Singapore, Elec. & Comp. Eng., 10 Kent Ridge Crescent 119260 Singapore

Laser-induced micro and nanoprocessing haven investigated for potential applications in microelectronics, data storage and photonics. The following toptics will be addressed in detail: laser surface cleaning, laser deflash and plasma-assisted laser microprocessing, laser texturing, laser bumping and related technologies, laser deposition of hard coatings and thin films, real-time Monitoring of laser surface processing, laser-induced controllable periodic structures, laser etching and nanolithography by tip-enhanced laser irradiation and optical resonance in microparticle.

2:30 PM
Surface Modification of Austenitic Stainless Steels: *Howard Ocken*[1]; Roger Asay[2]; [1]EPRI, 3412 Hillview Ave., Palo Alto, CA 94304-1395 USA; [2]Centec XXI, 8870 Muraoka Dr., Gilroy, CA 95020 USA

Austenitic stainless steels are widely used as structural alloys in nuclear power plants. The passive films that form when in contact with high-temperature primary coolant incorporate activated corrosion products, which are responsible for the exposure of plant personnel to ionizing radiation. The industry's need to minimize such exposures has led to the development of various surface modification techniques. EPRI has supported development of a surface modification technique, the Stabilized Chromium Process (SCrP), that is more effective than electropolishing. This paper will address the following topics: [1] early experiments on small coupons, [2] developing approaches for applying SCrP to complex shapes, [3] evaluating the response of components installed in commercial nuclear reactors, and [4] evaluating SCrP for possible non-nuclear applications.

2:45 PM
Deposition of Metal Coatings Utilizing Ablation Plasma Ion Implantation (APII): *Ronald M. Gilgenbach*[1]; Bo Qi[1]; Yue Ying Lau[1]; Mark D. Johnston[1]; Michael C. Jones[1]; Gary L. Doll[2]; Alexander G. Lazarides[2]; [1]University of Michigan, Nucl. Eng. & Rad. Scis. Dept., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]The Timken Company, Timken Rsrch., Adv. Matls. R&D, 1835 Deuber Ave. S.W., Canton, OH 44706 USA

We present results on a new technique for deposition and implantation of metal coatings by means of ions generated by KrF laser ablation of metals and accelerated by pulsed bias to the substrate. Initial APII experiments have implanted iron ions into silicon substrates at bias voltages up to negative 10 kV. Materials have been analyzed by Transmission Electron Microscopy (TEM) and X-ray Photoelectron Spectroscopy (XPS). Results prove that ion implantation has occurred, consistent with a maximum effective ion energy of about 8 keV. The lower effective energy is due to voltage droop and limited penetration of the overlying Fe film. XPS performed during an argon ion etch shows a depth profile confirming Fe implantation and deposition on the Si substrate. Future research will concentrate on ion implantation and deposition of hard coatings over softer metals. This research was funded by the National Science Foundation.

3:00 PM
Surface Modification of P/M TiC Reinforced Iron Matrix Composites: *Omer N. Dogan*[1]; David E. Alman[1]; Jeffrey A. Hawk[1]; [1]US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

Surface modification of wear resistant Fe-M_xC_y-TiC composites using a surface melting technique was explored. Powder metallurgy (P/M) was utilized to fabricate the composite alloys. Iron powder was mixed with elemental titanium, chromium, and graphite powders, and then hot-pressed to full density. During the hot pressing cycle, an SHS-type reaction occurred, transforming the titanium and chromium powders to TiC and $(Fe,Cr)_xC_y$ in a steel (Fe-Cr-C) matrix. The influence of alloy composition and SHS process parameters on the microstructure and abrasive wear resistance of the TiC reinforced composites were discussed. It is demonstrated that during the surface modification, the TiC phase does not melt but the steel matrix and $(Fe,Cr)_xC_y$ phase melt. During solidification of the austenite and M_7C_3 eutectic, a finer distribution of M_7C_3 particles is obtained. The in-situ formation of TiC and $(Fe, Cr)_xC_y$ precipitates via SHS reaction processing and surface melting produce a composite with wear resistance superior to conventional wear resistant materials.

3:15 PM
Synthesis of Nickel Ferrite Nanosize Powder by Pulsed Wire Discharge: K. Ishizaka[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

Various metal, simple oxide and simple nitride nanosize powders have been synthesized by a novel pulsed wire discharge (PWD) method. However, synthesis of double oxides has not been attempted because of difficulties in discharging two different metal wires simultaneously. In order to investigate possibilities of synthesizing double oxides by PWD, synthesis of nickel ferrite ($NiFe_2O_4$) nanosize powder was carried out. Nickel and iron wires were discharged in a chamber filled with oxygen gas. The synthesized powders floating in the gas were collected by pumping the gas through a molecular sieve. X-ray diffraction results indicated that small amount of a nickel oxide phase was remained in the powders. However, the main phase was $NiFe_2O_4$ and essentially single phase $NiFe_2O_4$ nanosize powders were successfully synthesized. From the results, we demonstrated capabilities of synthesizing complex oxide nanosize powders by PWD.

3:30 PM Invited
Laser Surface Modification of Electronic Properties in Wide Band Gap Materials: *I. A. Salama*[1]; N. R. Quick[2]; A. Kar[1]; [1]University of Central Florida, LAMMP, CREOL, Mech., Matls. & Aeros. Eng. Dept., Orlando, FL 32816 USA; [2]Applicote Associates, 894 Silverado Ct., Lake Mary, FL 32746 USA

A direct-write laser conversion technique is used to induce variation in both electrical and microstructural properties on the surface of wide band gap materials such as silicon carbide (SiC), diamond and gallium nitride (GaN). The effects of various processing parameters, e.g., laser-matter interaction time, laser beam power, number of repeated irradiation and ambient gas in the irradiation chamber are examined. The microstructural variations induced by laser beam irradiation are assessed for different process parameters. Scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), X-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS) are used to study the laser-irradiated surfaces and to understand the improvement in electrical properties observed in different materials. Different applications of the laser conversion technology will be discussed.

3:55 PM Break

4:10 PM Invited
Modifying the Surface of Nanoparticles by Coating: *Dieter Vollath*[1]; Dorotheé Vinga Szabó[1]; Sabine Schlabach[1]; Bin Xu[1]; [1]Forschungszentrum Karlsruhe, IMF III, PO Box 3640, Karlsruhe D-76021 Germany

Using the microwave plasma process, it is possible to synthesize ceramic nanoparticles with narrow size distribution. In many instances, the application of this kind of materials requires a modification of the surface. The reasons may be the reduction of the particle interaction using a coating as distance holder or an alteration of the chemical properties of the surface. It is a special feature of the microwave plasma process that the particles leave the reaction zone with electric charges of equal sign. Therefore, the particles are repelling each other. This makes it possible to coat the particles individually in a second reaction step. Depending on the application, this coating may consist of a polymer or a second ceramic. This coating–in the simplest case–influences the dispersion behavior in water. Other typical examples of application are superparamagnetic composites, where the coating acts as a distance holder in-between the particles reducing interaction.

4:35 PM
The Spectral Emittance and Long-Term Thermal Stability of Coatings for Thermophotovoltaic (TPV) Radiator Applications: *Brian Vern Cockeram*[1]; Jim L. Hollenbeck[1]; [1]Bechtel-Bettis, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

Vacuum plasma spray coatings (ZrO2 + 18%TiO2 + 10%Y2O3, ZrC, Fe2TiO5, ZrTiO4, ZrO2 + 8%Y2O3 + 2%HfO2, and Al2O3 + TiO2) have been developed to improve the surface emissivity of materials under consideration for TPV radiator applications. These coatings have been shown to be thermally stable and have produced a desired increase in the surface emissivity of refractory metals and nickel-base materials. The spectral emissivity of these coatings is measured before and after long-term vacuum anneals to determine the power density that would be provided to a TPV cell. The thermal stability of the coatings is further evaluated by characterization of the coatings after long-term vacuum annealing. A kinetic model of the volatility of oxide phases is used to describe the excellent thermal stability of the coatings that possess high post-anneal emittance values (ZrO2 + 18%TiO2 + 10%Y2O3, ZrC, and Al2O3 + TiO2.

4:50 PM
Surface Treatment of Aluminum Alloys with Potasium Ferrate Solutions: *Myung K. Han*[1]; S. K. Kim[1]; [1]Korea Institute of Industrial Technology, Ctr. for R&D Assistance for SMEs, 472 Kajwa-dong, Seo-ku, Inchon 404-254 Korea

A new and environment-friendly surface treatment method for aluminum alloys has been studied, which appears to be applicable as an alternative to the chromate conversion coating. The major drawback for the chromate conversion coating process is the toxicity of the hexavalent chromium, which is classified as a "known" carcinogen. Surface treatment with potassium ferrate solutions appears to be an ideal alternative to this process in that (1)the quality of the coated surface is superior, (2)ferrate compounds can easily be reduced to ferrous compound the thus become environmentally benign substance, and (3)the process consists of a simple dip-and-dry procedure. In this paper we present (1)pilot scale production of potassium ferrate powder, (2)analysis of the purity of the powder, (3)salt fog tests of the aluminum alloys treated with potassium ferrate solutions, and (4)analysis of the ferrate-coated surface.

5:05 PM
Nanostructuring Diamond Polytypes using Direct Ion Beam Deposition: *Quan Li*[1]; X. F. Duan[2]; N. G. Shang[1]; I. Bello[1]; Y. Lifshitz[1]; S. T. Lee[1]; [1]City University of Hong Kong, Dept. of Appl. Phys., Tat Chee Ave., Kowloon Tong, Hong Kong China; [2]Beijing Laboratory of Electron Microscopy, Beijing China

Direct ion beam deposition using an ion source consisting of mixtures of hydrocarbon/argon/hydrogen ions is capable of producing different diamond polytypes during diamond-like-carbon (DLC) film growth. A large range of experimental conditions is investigated including ion energy, substrate temperature and gas concentration ratios. Transmission electron microscopy (TEM) is used as the major analytical tool to study the film/substrate interface and film microstructures. Bombardment of argon ions together with hydrogen ions can modify silicon substrate surface by creating hillock patterns, exposing different silicon surfaces and increasing the substrate surface roughness. These modified silicon surfaces are proved to serve as epitaxial nucleation sites for diamond crystallites. Polytypes of diamond, including cubic, rhombohedral and hexagonal structures are observed in two forms concerning the original nucleation sites, i.e., directly grown on silicon or embedded in the amorphous DLC films. While the former exhibits certain epitaxial relationship with the silicon substrate, the later does not show any preferential orientation.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Smelter Gas Handling

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

Monday PM	Room: 607
February 18, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Norbert L. Piret, Piret & Stolberg Partners, Im Licht 12, Duisburg 47279 Germany; Pekka Taskinen, Outokumpu Research Oy, PO Box 60, Pori FIN-28101 Finland

2:00 PM
Effective Design of Converter Hoods: *Paykan Safe*[1]; John Deakin[2]; Sam Matson[1]; [1]Gas Cleaning Technologies, 4950 N. O'Connor Rd., Ste. 250, Irving, TX 75062 USA; [2]H. G. Engineering, Ltd., 400 Carlingview Dr., Toronto, ON M9W 5X9 Canada

The primary source of fugitive gas emissions in most copper smelters are the Peirce Smith converter operations. The modern trend is towards installation of secondary gas collection systems which are intended to collect fugitive gases from charging, standy, and skimming operations. These have been installed with varying degrees of success. Often, the major source of emissions from the converter area is leakage of process gas from the primary hoods when the converter is blowing. This gas is typically collected by the secondary system and discharged to atmosphere or scrubbed with an alkaline agent. Those smelters considering installation of secondary hoods or scrubbing of gases from secondary systems should first address the issue of gas leakage from the primary hoods. Improving the primary hood gas collection efficiency will often be more cost effective than installing secondary hooding. In the case of secondary gas scrubbing, it will significantly reduce the reagent consumption and operating cost of the scrubbing system. This paper examines the parameters that determine whether or not a primary hood will effectively collect process gases without leakage and demonstrates, using Computational Fluid Dynamics (CFD), the effects of variations in these parameters on the overall performance of the hood. It also looks at the application of CFD techniques in the design of secondary hoods and presents a summary of measured emissions from converter buildings.

2:25 PM
Boliden's Specialized Technology for Smelter Gas Handling and Effluent Treatment: *Bjorn Lindquist*[1]; [1]Boliden Contech AB, PO Box 745, Gymnasievagen 14, SE-931 27 Skelleftea Sweden

In this paper, Boliden's technologies in the field of gas cleaning and effluent treatment will be described. Special attention will be given to the use of high pressure venturis, the Editube wet electrostatic precipitator, mercury removal processes, and two-stage effluent treatment.

2:50 PM
Optimization of Metallurgical Processes using High Rate Biotechnology: Henk Dijkman[1]; *Johannes Boonstra*[1]; Rick Lawrence[2]; Cees J.N. Buisman[1]; [1]Paques B.V., PO Box 52, 8560 AB, Balk The Netherlands; [2]BioteQ Environmental Technologies, Inc., Ste. 1150, 355 Burrard St., Vancouver, B.C. Canada

Existing metallurgical operations can be optimized by implementing engineered bioreactor systems in the process configuration. For instance, metals can safely and economically be recovered from process and waste streams using biogenic sulfide. The paper focuses upon possible applications of high rate biotechnology in metallurgy. Metal recovery from liquid streams (electrolyte bleed streams, leach water, waste streams), integrated removal of (fugitive) SO2, treatment of acid blowdown and other options will be described. Practical examples will be given. A large scale application of biotechnology at Pasminco's Budel Zink refinery will be described. Here acid plant blowdown is treated together with electrolyte magnesium bleed. Concentrated zinc sulfide (10 tpd) is produced biologically, eliminating the production of gypsum and closing the refinery sulfur cycle. A smaller scale application of biotechnology at Kovohute Primbram in the Czech Republic is described. In this case H2S is produced on-site for metal removal from a waste water stream.

3:15 PM
A Novel Cyclic Process for Producing Elemental Sulfur from Sulfur Dioxide without Generating Secondary Pollutants: *Hong Yong Sohn*[1]; Byung-Su Kim[1]; [1]University of Utah, Dept. of Metallurgical Eng., Rm. 412, 135 S. 1460 E., Salt Lake City, UT 84112 USA

A thermodynamic and experiemental investigation has been performed to develop a new process for converting sulfur dioxide to elemental sulfur by a cyclic process. The most promising new process is a cyclic reaction scheme involving calcium sulfide and calcium sulfate. In this process, the reaction between sulfur dioxide and calcium sulfide produces elemental sulfur vapour and solid calcium sulfate. The latter is reduced by a suitable reductant, such as hydrogen, to regenerate calcium sulfide. Experimental results show that up to 60% of the calcium sulfide powder was converted to calcium sulfate in 10 minutes at 1153 K under a sulfur dioxide partial pressure of 28.5 kPa. More than 95% of the nickel-catalyzed calcium sulfate powder was converted to calcium sulfide in 20 minutes at 1123 K under a hydrogen partial pressure of 86.1 kPa, water vapor being the only gaseous product. Sulfur dioxide-containing streams from certain new sulfide smelting plants contain much higher partial pressures of sulfur dioxide. The rate of the first reaction is expected to be accordingly higher than in the test conditions reported in this paper.

3:40 PM Break

3:55 PM
Recent Improvements in the Acid Plant at Naoshima Smelter: *Shin-ya Yagishita*[1]; *Fumitaka Sakurai*[1]; [1]Mitsubishi Materials Corporation, Naoshima Smelter & Refinery, 4049-1 Naoshima-cho, Kagawa-Gun, Kagawa 761-3110 Japan

The Mitsubishi process in Naoshima has kept steady operation since May 1991. Yet the sulfuric acid plants have been using old equipment for more than 30 years operation. In 1999, the acid plants were modernized by renewing some of main equipment like, drying tower, gas heat exchanger and introducing new technology. At the same time, as an electric energy saving measure, the electric power consumption in sulfuric acid plant was vastly improved by decreasing treated gas volume based on installation of low temperature catalyst in 1st pass of converter, and optimization of main gas blower runner. Furthermore, last year, the oxygen plant was replaced and enlarged, and anode production increased from 240 to 280 KMT/Y. And the sulfuric acid production also increased from 550 to 660 KMT/Y. This paper describes these recent improvement in the acid plant at Naoshima smelter.

4:20 PM
Recovery of Sulphur in Off-Gases from Roasting of Molybdenum Sulphide: *Jens Laursen*[1]; [1]Haldor Topsoe A/S, Nymollevej 55, 2800 Lyngby Denmark

In 1999, Molymex S.A. de C.V., Mexico (a company wholly owned by Molybdenos y Metales S.A. [Molymet], Chile) decided to install the Haldor Topsoe WSA process for cleaning of the 4-5% S02 off-gas from their molybdenum sulphide roaster in Cumpas, Mexico, (WSA-the Wet gas Sulphuric Acid process for converting S02 in off-gases to sulphuric acid). The paper will describe the process layout and the initial experiences from the start up scheduled for end 2001. The installation of the WSA plant was decided, on the basis of successful operation since 1993 of a WSA plant at Molymet's molybdenum sulphide roaster in Santiago, Chile. The paper will describe the operating experience of the Santiago plant and furthermore summarise the application of the WSA process for other roasting plants and smelters within the non-ferrous metals industry (Copper, Lead, and Molybdenum).

4:45 PM
Modern Off-Gas Treatment Secures the Future of Sulfide Smelting: *Karl-Heinz Daum*[1]; *Hans-Jochen Koenig*[1]; Peter Luedtke[1]; [1]Lurgi Metallurgie GmbH, Ludwig-Erhard-Str. 21, D-61440 Oberursel Germany

In order to meet environmental regulations, sulfur capture in smelters has to be further optimized. At the same time, efficiency, reliability, ad economics of smelter operation have to be improved. Thus, off-gas handling systems should give saleable byproducts, not interfere with smelter performance, treat all gases simultaneously, and comply with environmental regulations. The latest developments in sulfur acid plant design will be presented.

5:10 PM Moved to Monday AM
Budel Zink Sets a New Standard for NO_x Reduction in a Sulphuric Acid Plant: *J. van Driel,* B. Giesen, A. Berryman, S. Sampat, S. Enevoldsen, H. Jensen-Holm

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

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

Monday PM — Room: 305
February 18, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: M. J. McNallan, University of Illinois-Chicago, Matls. Sci., Chicago, IL 60607-7023 USA; P. F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

2:00 PM
Breakaway Oxidation of Ferritic Stainless Steels in Water Vapor Atmospheres: *A. Galerie*[1]; S. Henry[1]; Y. Wouters[1]; L. Antoni[2]; [1]Institute National Polytechnique de Grenoble, Laboratoire de Thermodynamique et de Physicochimie Métallurgiques, UMR CNRS/INPG/UJF 5614, BP 75, Saint Martin d'Heres 38402 France; [2]Usinor Recherche et Development, Centre de Recherches d'Ugine, Avenue Paul Girod, Ugine 73000 France

The well-known breakaway oxidation of ferritic stainless steels occurring in water vapor-containing atmospheres appears after an incubation period where a chromia-rich scale grows slowly. It is characterized by nucleation and rapid growth of iron oxide nodules. The mechanisms responsible for this remarkable effect of water vapor are reviewed and discussed under the light of a series of experimental results on 12 to 18%Cr-containing stainless steels. Mechanical features, as microcracking of the chromia scale, are shown to be of importance, but cannot alone explain what is observed. Scale volatilization may occur, but it was calculated to be negligible in oxygen-free water vapor atmospheres. Observations of iron nodule nucleation associated to thermodynamic calculations showed that stress levels are not high enough to induce iron oxide stability at the stainless steel-chromia interface, but that interface microporosity would promote the right driving force. Kinetic considerations, based on the concept of surface acidity promoting H_2O molecule dissociation, help to understand the rapid growth of the iron oxide nodules.

2:30 PM
The Oxidation Behaviour of Ferritic-Martensitic 9% Cr Steels in H2O Containing Environments: *M. Schütze*[1]; [1]Dechema E.V.,

Karl-Winnacker-Inst., Theodor-Heuss-Allee 25, Frankfurt am Main D-60486 Germany

Three commercial 9% Cr steels (P91, E911, Nf616) and 6 laboratory versions of Nf616 with Si contents from 0.04 to 0.78 were investigated at 650°C in dry synthetic air and air enriched with 4 and 10% water vapour, respectively. The aim of these investigations was to measure the time to breakaway for the different steels in these atmospheres and to elucidate the mechanisms responsible for the different behaviour. It was observed that the steels can be classified by 3 groups of behaviour. The first group exhibits breakaway behaviour almost from the beginning of oxidation and oxidizes with high rates. Steels of the second group show protective behaviour at the beginning but after several 100 to 1000 hrs. breakaway oxidation starts. In the third group no breakaway behaviour was observed for at least 10,000 hrs. As a general tendency it was found that increasing the water vapour content in the environment decreases the time to breakaway and shifts steels of groups II and III into group I or II, respectively. Silicon has a beneficial effect and increases the time to breakaway or possibly even suppresses breakaway completely. Interestingly steels which form a very thin protective oxide scale show a mass decrease during oxidation in atmospheres of high water vapour content. As revealed by UV absorption spectroscopy CrH2O4 evaporates from the surface to an extent which makes the mass decrease only detectable for slowly growing scales. The results from microstructural investigations are used for explanation of the behaviour of the different steels.

3:00 PM

Void and Crack Formation in Oxide Scales on Ferritic 9-12%Cr Steels during Steam Oxidation: *W. J. Quadakkers*[1]; J. Ehlers[1]; J. Zurek[1]; L. Singheiser[1]; [1]Forschungszentrum Jülich, Inst. for Matls. & Proc. in Energy Sys., Jülich 52425 Germany

In future, high efficiency fossil fuel fired power plants steam parameters will be increased in the range of 600-650°C and 300 bar. To meet these stringent stress conditions, new ferritic-martensitic 9-11%Cr steels have been developed to replace commonly used steels such as 1CrMoV and 12CrMoV. One of the life time limiting factors of the new high strength steels at the high service temperatures is the surface scale formation occurring during service in steam environments. In this context, not only the growth rates but also the spalling resistance of the scales is an important issue. The spalling characteristics of the magnetite based surface oxides appear to be strongly affected by the formation and growth of in-scale voids. In the present study, the mechanisms of void formation and growth was studied for a number of ferritic steels during oxidation in a simulated steam environment (Ar-50% H2O) at temperatures between 550 and 650°C with exposure times ranging from a few to 5000 hours. The results indicate that void formation is not related to one single mechanisms. In the early stages of oxidation gaps and voids can be formed by formation of a volatile specie, likely Fe-hydroxide. The voids formed by this mechanism heal during prolonged exposure. After longer exposure times formation of tiny voids starts in the inner scale as a result of vacancy condensation. The void nucleation and morphology is related to spinel stringers resulting from oxidation of Cr-rich carbides. In the outer part of the oxide scale formation of large voids starts only after prolonged exposure times. They result from impeded transport of Fe cations through the inner scale. As scale cracking and spalling characteristics during thermal cycling are strongly affected by the location and morphology of voids, knowledge of the void formation mechanisms is of great importance in the estimation of actual materials long term behaviour.

3:30 PM

The Corrosivity of H_2O/O_2 Environments towards Chromia-Forming Austenitic and Ferritic/Martensitic Steels at 500-800°C: *Karin Segerdahl*[1]; Henrik Asteman[1]; Jan-Erik Svensson[1]; Lars-Gunnar Johansson[1]; [1]Chalmers University of Technology, High Temp. Corrosion Ctr., Kemivägen 10, Göteborg 412 75 Sweden

The effect of water vapor on the oxidation of chromia-forming steels with 11-25% Cr between 500 and 800°C is reported. The samples are investigated by thermogravimetry, GI-XRD, SEM/EDX, and SAM. The oxidation Fe/Cr and Fe/Cr/Ni steels in H_2O/O_2 environment is strongly affected by gas velocity because chromium from the oxide is vaporized in the form of $CrO_2(OH)_2$. The oxidation behavior at a certain temperature, gas composition and gas velocity is to a large extent determined by the ability of the metallic substrate to supply the oxide with chromium to compensate for the losses by vaporization. The corrosion resistance of chromia-forming steels is enhanced by high Cr concentration, fast diffusion in the steel bulk (ferrite/martensite rather than austenite), and a high density of steel grain boundaries (small grain size). The corrosivity of the environment increases with the concentration of water vapor and oxygen, with the gas velocity and with temperature.

4:00 PM

Effect of Chromium and Nickel Contents on High Temperature Oxidation of Stainless Steels in Mixed Air and Water Vapor: *R. Peraldi*[1]; J. A. Schenning[1]; B. A. Pint[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., Corrosion Sci. & Tech., PO Box 2008, Oak Ridge, TN 37831-6156 USA

The corrosion resistance of chromia-forming stainless steels depends on their potential to form and maintain a protective Cr-rich oxide scale. Accelerated corrosion attack has been observed for some commercial stainless steels exposed to exhaust gas at 600°-800°C. The rapid growth of an Fe-rich oxide scale significantly reduces the performance of thin-walled components in power generation applications. In order to better understand alloy composition effects, the oxidation behavior of several model alloys with Ni and Cr contents ranging between 0-30wt.% and 10-20wt.%, respectively, have been studied in air + 10vol.% water vapor under various isothermal and cyclic conditions. Performance improvements were observed by increasing either the Ni or Cr contents of the steel. However, corrosion-resistant compositions must maintain good creep properties and meet cost targets. Other factors to be investigated are the effects of alloy grain size, phase composition, surface preparation, and minor alloy additions. The composition and microstructure effects observed in this study also will provide data for life-prediction models and may suggest a mechanistic explanation for the effect of water vapor. Research sponsored by the Microturbines Program, Office of Power Technologies, US Department of Energy (DOE), under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and supported, in part, by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administered jointly by the Oak Ridge Institute For Science and Education and Oak Ridge National Laboratory.

4:30 PM Cancelled

High Temperature Oxidation of Ferritic Cr-Steels for Solid Oxide Fuel Cell Application: *P. B. Friehling*

5:00 PM

Thermochemical Analysis of Oxidation and Corrosion Processes in High Temperature Fuel Cells: *P. Singh*[1]; Gary Yang[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA

This paper examines various oxidation and corrosion processes operating in high temperature fuel cells. Mechanisms for the scale growth in the multi component fuel gas environment containing H_2, H_2O, CO, CO_2, CH_x etc. are proposed and thermochemistry of oxidation processes in the redox gas atmosphere (H_2-H_2O), oxide stability, hydrated oxide formation etc. are analyzed. Role of substrate and oxide stability on the long-term performance of fuel cells are presented and discussed in detail.

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-K^ser-Strasse, Stade 21683 Germany

Following the development of the circulating fluid bed calciners (CFB) at the Nabwerk and L¸nen 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 JosÈ Mendoza*[1]; [1]C.V.G. Bauxilum, Control de Calidad y Proceso, Zona Industrial Matanzas, Puerto Ordaz, BolÌvar 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 ìVolkhov aluminiumî, 20, Kirovsky pr., Volkhov, Leningrad Region 187400 Russia; [3]JSC PC ìGlinozyomî, 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 S^derberg Cell TechnologyñFuture Challenges and Possibilities: *Adolf Karstein Syrdal*[1]; [1]Elkem Aluminium Lista, PO Box 128, Farsund 4551 Norway

In the sixties the S^derberg 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 S^derberg cells. A strong paradigm has been that all S^derberg smelters would be phased out before the new millennium. It is therefore a paradox that in 2002 S^derberg 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 S^derberg cell development and the present distribution of S^derberg lines around the world. S^derberg 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 S^derberg 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 S^derberg 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 ìdryî anode technology in the Krasnoyarsk plant in cooperation with Kaiser Aluminum company at 1990-1998, and ìsemidryî 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 PlantóGuizhou 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

Nor□ur·l, 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 Nor□ur·l 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-ATGís modelling and engineering team developed over the last few years. The pots commissioned during the Nor□ur·l Phase I project are operating well achieving world-class results. Since the Nor□ur·l 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 todayís 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 βî and lath-shape Q phases. Both βî 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 Quebecís H.S. Sˆderberg 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 Alcanís H.S. Sˆderberg plants marks an important step in the Companyís continuing efforts to reduce the Polycyclic Aromatic Hydrocarbons (PAHís) emission of the Sˆderberg technology. Improvements to H.S. Sˆderberg anode formulation and implementation of the low PAH pitch binder has provided a significant reduction in PAHís emission in Alcanís H.S. plants over the last twenty years. A demonstration test was carried out with a low PAH pitch in the Sˆderberg plant before Alcanís 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 PAHís 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 RhodaxÆ 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: *AndrÈ 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 ìgently controlledî 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)ñA 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 Egyptalumís 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 authorís 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 companyís 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 UniversitÈ du QuÈbec ‡ Chicoutimi, DÈpt. des Sciences AppliquÈes, 555 blvd. de lìUniversitÈ Chicoutimi, Chicoutimi, QuÈbec G7H 2B1 Canada; [2]Alcan International, Ltd., Arvida R&D Ctr., 1955 blvd. Mellon, Jonquiere, Quebec G7K 5J7 Canada

TUESDAY AM

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 ìenvelopeî 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 SystemñElectromagnetic 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 ìTop Chargeî 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[a] melt furnace has been developed at Alcanís 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 ceramicís 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 TransformationñA 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 ëAl-spotsí. 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. Yanían 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 doesnít 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 HÂkonsen*[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 substitute 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 $\Sigma(111)$ and Ni $\Sigma5(210)$ 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 Els‰sser*[1]; Rebecca Janisch[1]; Thorsten Ochs[1]; [1]Max-Planck-Institut f¸r Metallforschung, Seestr. 92, C-70174, Stuttgart, Germany

The influence of interstitial impurities B, C, N, O and H on the structure and stability of $\Sigma5$ (310) [001] symmetrical tilt grain boundaries ($\Sigma5$ 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 $\Sigma5$ 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 $\Sigma5$ 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 f¸r 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. ìSnap shotî 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 ìhardî 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

Copperñnickelñsulphide 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 ëbroad brushí computational model of this ingot cooling process, that can predict some ëintegralí 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 ëbroad brushí 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 ëintegralí 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: *FrÈdÈric Pineau*[1]; [1]National Research Council Canada, Indl. Matls. Inst., Proc. Modlg. & Instrm., 75 de Mortagne Blvd., Boucherville, QuÈbec 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 Jouleís 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 predicts 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 ìliquid dendritesî 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 boundary 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 ìconventionalî 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 doesnít 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
February 19, 2002 — Location: Washington State Conv. & Trade Center

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 ULTIMET, 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 SOFCís. 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 ~100∫C, 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, ìsolid acidsî, 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 250∫C. 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 conditions 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 structures). 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 díHeres 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 díHeres 38402 France; [2]INPG, LTPCM, Domaine Universitaire, BP 75, St. Martin díHeres 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 Frˆbel*[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 February 19, 2002 — Room: 601 — 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 Wairegiís 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.ís 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
Electrothermal 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 ìGintsvetmetî, 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 exchange 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]; JosÈ 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 Sneokís 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), permittivity (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 1000∫C: *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 Gursonís 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	Room: 213
February 19, 2002	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 ì70 and ë80ís, 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 (HIPíd 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, Seatle, 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 díHËres 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. ≈gren*[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 f¸r Eisenforschung, Physl. Metall., Max-Planck-Str. 1, D¸sseldorf 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 — Room: 310
February 19, 2002 — 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]UniversitÈ díAix-Marseille III, L2MP, UMR CNRS 6137, FacultÈ de St JÈrÙme, 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 ëForging Suppliers Initiativeí 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 25ñ50° 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 Norandaís 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: *...ric Baril*[1]; Mihriban O. Pekguleryuz[1]; Rubens Verni[1]; [1]Noranda, Inc., Tech., 240, Hymus Blvd., Pointe-Claire, QuÈbec 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 FLUENTÆ 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 FLUENTÆ.

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, Xiían 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 ìmagnetic buoyancyî 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 (ìg-jitterî) 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. Dìaz[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 CAF… 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 FÈdÈrale de Lausanne, Laboratoire de MÈtallurgie Physique, DÈpartement des MatÈriaux, 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 ìGintsvetmetî, 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 ìRyazTsvetmetî (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 ìmetallic phasesî 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 ìmetallic-phaseî 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 ìmetallic phasesî 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]Ris 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. Ung·r and A. BorbÈly, Appl. Phys. Letters, 69, 3173 (1996) [3]T. Ung·r, I. Dragomir, ¡. RÈvÈsz and A. BorbÈly, J. Appl. Cryst. 32, 992 (1999) [4]M. Wilkens, phys. stat. sol. (a) 2, 359 (1970). [5]T. Ung·r, J. Gubicza, G. Rib·rik and A. BorbÈly, 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 samplesí 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 St¸we[1]; [1]Austrian Academy of Sciences, Erich Schmid Inst. of Matl. Sci., Jahnstrafle 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°… under 4.8ßË, in the specimen A120, and its microstructure was nanocomposite type with grain size less than 50 ß¨. An abnormal grain growth was observed and some Al grains grew up over 500 ß¨ in the specimens A300 prepared at 300°…. 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 ßÁ and that of the specimens A300 was 467 ßÁ at room temperature. The strength of the specimens A120 decreased greatly with increasing ductilities at 300-500°…. 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 ìliquid bubblesî 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 ìtrial-and-errorî 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. Ocelìk[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 $a\text{-(Fe-B)}_m$ amorphous phase into paramagnetic $a\text{-(Fe-B)}_p$ 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 Networkñ 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. Outokumpuís 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 Outokumpuís 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]CompaÒÌa 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 didnít 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

Incoís 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 ëregularí and ëstripperí 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 operationís 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. Sch¸tze, 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 ≈ 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. L¸eck[2]; K. Lu[3]; E. J. Lavernia[1]; M. R¸hle[2]; [1]University of California-Irvine, Dept. Chem. & Biochem. Eng. & Matl. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA; [2]Max-Planck-Institut f¸r 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 TiSiî 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

Itís 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: *AndrÈ Lalla*[1]; Rolf Arpe[1]; [1]Aluminium Oxid Stade GmbH, Johann-Rathje-Kˆser-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 LamÈrant*[1]; [1]Aluminium Pechiney, Alumina Tech., BP 54, Gardanne 13541 France

Gardanneís Bayer Plant has been modified to process BokÈ 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 ìhuntsî 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 KolÂs[2]; Trond St□re[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., ≈rdal 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 ìPolitehnicaî 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% Mg_2Si together with excess Si and Cu each up to 0.8 wt%. In this study, the effect of Cu on 0.8 wt%Mg_2Si 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 1960ís 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 multicomponent 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 CedeÒo

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 AlumÌnio Brasileiro S/A, Carbon Plant, Rod. PA 483 Km 21, Vila do MurucupÌ, Barcarena, Par· 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., PolÌgono 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 R¸hle*[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 interfaces 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]Queenís 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[Æ] 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]; AndrÈ 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, ìfoamed surfaceî 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 ìcloud of internal stressesî 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. Bishopís 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 ìHorns of a Dilemmaî: 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 ìeffective diffusivityî 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 ìhornsî 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 ìhornsî 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 ìorderingî 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 ≈gren[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.

3:30 PM
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. ìDowntownî, 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. ìDowntownî, 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.

5:25 PM
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

TUESDAY PM

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 todayís 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.

4:10 PM Break

4:20 PM

Mechanical and Microstructural Differences Between Uniaxial and Multiaxial Creep Loading of Short Fibre Reinforced Aluminium Alloys: *Alejandro Andres Yawny*[1]; Gregor Kaustr‰ter[1]; Birgit Skrotzki[1]; Gunther Eggeler[1]; [1]Ruhr-Universit‰t Bochum, Institut f¸r 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).

4:40 PM
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.

5:00 PM
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 (Alved□re and Niederauflem). 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 componentís 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.

2:30 PM
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.

2:50 PM
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 structure, 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 ($\sim10^9$ 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 instrumented 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 ‹st¸ndag[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 Youngís modulus and Poissonís 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 (MMCís) 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 MMCís 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 MMCís 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 ìLi-ionî 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 dislocations 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 graphiteís 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 (ECís) 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]; *AndrÈ Schneider*[1]; Georg Frommeyer[1]; [1]Max-Planck-Institut f¸r 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[a], 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 powderís 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

TUESDAY PM

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]; Bj^rn Uhrenius[2]; [1]Swedish Institute for Metals Research, Drottning Kristinas v‰g 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 1970ís 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 todayís 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 Engineerís 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 ëstrongest sizeí. 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 ìtonguesî. 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 OfficeñFar 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 interfaceñthe boundary regionñseparating 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 ìdata miningî 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 KovarÆ. 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-BallsñProduction 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 Universit‰t 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 ìroughî 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 ìdarkeningî effect of PbSn-BGA. A ìdarkeningî of the BGA-balls during handling can be observed. This is probably caused by oxidation effects as result of ìmechanical grinding of the surfaceî. This can cause problems by identifying the balls with optical sensors in the pick & place station. The ìdarkening behaviorî was investigated in comparison with PbSn-balls. The results show a significant higher stability against ìdarkeningî 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 heterogenous 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 ìearly failureî 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 multi-ply-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. Youngís 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 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 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

3:55 PM
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 plugís 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.

4:20 PM
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 ìfreeî 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 todayís high-performance structures. Additional advances are being made in NASAís 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 SAFís 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 ìqualityî 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 ìTotal Metal Recoveryî 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 managementís goal was to maximize aluminum unit recovery in order to optimize the facilityís 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 IllÈs[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. GellÈrt tÈr 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, Gyeonggi-do 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]Xiían University of Architecture and Technology, Sch. of Metlgcl. Eng., 13 Yanta Rd., Xiían 710055 China

Only shear and ìequivalentî 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 ìequivalent strainî 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]; Gy^rgy V^r^s[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]E^tv^s University, Dept. of Gen. Phys., 1117 P·zm·ny P. sÈt·ny 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 ìartifactsî and the presence of undesirable interface states. Using a controlled pulsed laser processing, we have produced "artifact" free thin films and composites and measured ìtrueî 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 NanoGramís 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 elementsñseveral hundred microns to millimeters in sizeñposes 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 SWNTís 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 compositeís 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 derivativeís ability to transfer the applied load from the compositeís 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

TUESDAY PM

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 Aveciaís 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 1970ís; 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 AutÛnoma Metropolitana, Dept. Ing. de Procesos e Hidra˙lica, Av. San Rafael Atlixco 186, Col. Vicentina, DF 09340 MÈxico

With todayís 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 ìproductsî 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 SmeltingñThermodynamic 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 M‰kinen[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. Puuruneni[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, Kumeraís 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 accompolished 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.

Advances in Metallic Glasses: Mechanical and Other Physical Properties, and Applications - 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

Wednesday AM   Room: 212
February 20, 2002   Location: Washington State Conv. & Trade Center

Session Chairs: Joseph S. Poon, University of Virginia, Dept. of Phys., McCormick Rd., Charlottesville, VA 22904 USA; A. R. Yavari, Institut National Polytechnique de Grenoble, LTPCM-CNRS umr 5614, BP75, 38402 St.-Martin-d'Heres Campus, France

8:30 AM Invited
Civilian Research on Bulk Metallic Glasses Sponsored by the European Union: *A. R. Yavari*[1]; [1]Institut National Polytechnique de Grenoble, (LTPCM-CNRS umr 5614), BP 75, 38402 St-Martin-díHÈres Campus France

With near 2% elastic strain range (as compared to 0.2% for crystalline materials), BMGs have excellent values of the Ashby performance index σ^2/E (where σ and E are the yield strength and Youngís modulus). High values of this performance index (reversible storing of elastic energy) open applications in sporting goods materials (golf clubs, skis, other high elasticity equipment), cutting, writing, punching and printing tools, springs, gears for micromachines (MEMS parts) and others. In this work we report on the results obtained by the European Union Network on BMGs. This Network brings together 9 research teams in 5 EU and 1 Associated states and the European Synchrotron (ESRF) facilities possessing the fundamental and engineering knowledge-base and equipment required to establish a European pole of excellence for research on BMG.

9:00 AM
Welding Technologies of Bulk Metallic Glasses: *Yoshihito Kawamura*[1]; Yasuhide Ohno[1]; [1]Kumamoto University, Matls. Sci., 2-39-1 Kuro-kami, Kumamoto 860-8555 Japan

The bulk metallic glasses have solved two main problems in metallic glasses, namely, the limitation of product size and the lack of workability. The problem of welding is, however, still unsolved. We have succeeded in welding bulk metallic glasses to bulk metallic glasses or crystalline materials with keeping the amorphous structure by friction, pulse-current and explosion methods. The tensile strength of the welded bulk metallic glasses was the same as that of the parent bulk metallic glasses. The friction welding is a solid state process where we used the superplasticity of the supercooled liquid. The pulse-current and explosion welding methods are liquid state processes where we used the high glass forming ability of the bulk metallic glasses and the high thermal stability of the supercooled liquid. The successful results obtained in this study are expected to push forward the application of bulk metallic glasses.

9:20 AM
Microstructure-Magnetic Properties Relationship in Bulk $Nd_{90-x}Fe_xAl_{10}$ Glassy Alloys: *Nicoleta Lupu*[1]; Horia Chiriac[1]; [1]National Institute of Research and Development for Technical Physics, Magnetic Matls. & Dev., 47 Mangeron Blvd., Iasi 6600 Romania

Rapidly and slowly quenched $Nd_{90-x}Fe_xAl_{10}$ glassy alloys with thicknesses up to 3 mm were investigated comparatively by structural (XRD, neutron scattering and calorimetric measurements) and magnetic AC and DC measurements in the temperature range 5-800K and external fields up to 10 T. These amorphous alloys crystallize through single exothermic peak and exhibit very large values for the reduced crystallization temperature, T_x/T_m, of 0.87 to 0.92. The glass-forming ability decreases with the Fe content increase. The mechanisms that govern the different magnetic behavior in rapidly quenched and respectively slowly quenched $Nd_{90-x}Fe_xAl_{10}$ glassy alloys are related to the cooling rates values assured in those two methods of preparation and will be discussed in detail. The large values of the coercive field obtained for $Nd_{90-x}Fe_xAl_{10}$ glassy alloys are ascribed to the existence of one non-equilibrium or metastable magnetic phase consisting in very small clusters whose sizes approach few nanometers.

9:40 AM
Synchrotron X-Ray Measurements of Lattice Strains during In-Situ Loading of Partially-Crystallized Bulk-Metallic-Glass Composites: Dorian K. Balch[1]; Ersan ‹st¸ndag[2]; *David C. Dunand*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., MLSB 2036, 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]California Institute of Technology, Dept. of Matls., Pasadena, CA 91125 USA

High-intensity synchrotron x-rays were used to study the micromechanics of load transfer upon uniaxial compressive loading of a Bulk-Metallic-Glass (BMG) composite containing 5 vol.% Ta particles. Diffraction rings were recorded for both the Ta particles and crystalline inclusions within the BMG matrix, which were produced by partial crystallization of the BMG and acted as crystalline ìmarkersî in the amorphous matrix. Using elastic constants of each phase, the stresses are then calculated in the matrix and reinforcement of the composite. Load transfer between BMG and Ta is discussed in terms of plastic deformation of the Ta phase, integrity of the BMG/Ta interface and creep relaxation of the BMG matrix after a hold of 1 hour at the maximum uniaxial stress of 1250 MPa.

10:00 AM Break

10:20 AM
Mechanical Response of Zr-Based Metallic Glass: *Alla V. Sergueeva*[1]; Nathan Mara[1]; Amiya K. Mukherjee[1]; [1]University of California, Cheml. Eng. & Matl. Sci., 1220 Bainer Hall, Davis, CA 95616 USA

The new family of Zr-based multicomponent metallic glasses shows a beneficial combination of yield strength value as high as 2 GPa and microplasticity of up to 1% at room temperature and an excellent glass-forming ability in a wide supercooled liquid region. Partial devitrification of glassy alloys upon heating or hot working above the glass transition temperature can lead to formation of nanocrystalline precipitates in the glassy matrix. In this case, the mechanical properties depend on the amount of crystalline precipitates. The nature of the brittle crystalline intermetallic phases is likely to dominate the mechanical behavior with crystallization more than 50%, leading to the observed decrease in ductility for the annealed samples since the deformation is no longer governed by the deformation mechanism of the amorphous phase. A fully crystallized alloy produced by annealing revealed increasing ductility with temperature in the regime where grain growth occurs and this microstructure exhibited the superplastic deformation with high elongation at 650-700°C.

10:40 AM
Superplasticity in a Bulk Amorphous Pd-40Ni-20P Alloy: *Jinn P. Chu*[1]; C. L. Chiang[1]; T. G. Nieh[2]; Y. Kawamura[3]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Lawrence Livermore National Laboratory, L-350, PO Box 808, Livermore, CA 94551 USA; [3]Kumamoto University, Dept. of Mechl. Eng. & Matls. Sci., Kumamoto 860-8555 Japan

Recent advances in metallic glasses that have high glass-forming ability and high thermal stability have launched a number of studies on fundamental science and practical applications. In the present study, compressive deformation behavior of a bulk amorphous Pd-40Ni-20P alloy was characterized in the supercooled liquid region. Significant reductions in sample height (>95%) were achieved after deformation, suggesting the possible superplastic-like behavior in the alloy. Structures of the amorphous material, both before and after deformation, were examined using X-ray diffraction and differential scanning calorimetry. Transmission electron microscopy was also used to examine the deformed samples. The superplastic-like behavior will be presented and discussed in light of the microstructure/crystal structure results.

11:00 AM
Effect of Plasticity on the Elastic Modulus and Density of Bulk Amorphous $Pd_{40}Cu_{30}Ni_{10}P_{20}$: *Ulrich Harms*[1]; Ou Jin[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MS G755, Los Alamos, NM 87545 USA

Although free volume is often used to characterize structural changes in amorphous metals, few density measurements have been reported. We measured the effect of annealing and plastic deformation on the density and modulus of bulk amorphous $Pd_{40}Cu_{30}Ni_{10}P_{20}$. The shear and bulk modulus are measured by a resonant-ultrasound method. The density is measured with a resolution of 1 part in 10,000. Whereas annealing causes the shear modulus and density to increase, the bulk modulus remains approximately constant. Plastic deformation reduces both the density and shear modulus to their values in the as-quenched state. We find a linear relationship between the shear modulus and density changes, suggesting that these parameters could be described by a single type of defect. The relative change in the shear modulus is approximately 30 times larger than the relative change in the density. Models for the ratio of density to modulus change are discussed.

11:20 AM
Mechanical Behavior of In-Situ Formed Bulk Metallic Glass Matrix Composites: *Bjorn Clausen*[1]; Seung-Yub Lee[1]; Ersan ‹st¸ndag[1]; C. P. Kim[1]; Mark A.M. Bourke[2]; Donald W. Brown[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, MST-8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA

Bulk metallic glass (BMG)-matrix composites have recently been developed to increase the ductility of BMGs. One of the most promising composites is the in-situ-formed BMG/ìβ-phaseî composite. Here, the ìβ-phaseî is a fine dendritic structure of a BCC crystalline alloy and precipitates during casting. This phase has been shown to inhibit the formation of macroscopic shear bands that cause catastrophic failure in monolithic BMGs under unconstrained loading. In this study, we have used neutron diffraction to measure the elastic deformation in the crystalline ìβ-phaseî during mechanical loading of BMG/ìβ-phaseî composites and compared this to the deformation of ìβ-phaseî monoliths. We have employed self-consistent deformation models to interpret the diffraction data and to deduce additional parameters such as single-crystal elastic constants. The results suggest that the mechanical properties of the ìβ-phaseî are highly variable and sensitive to processing conditions.

11:40 AM
Acoustic Emission Investigation of Inhomogeneous Plastic Flow of Metallic Glasses: *Vitaly Khonik*[1]; Alexey Vinogradov[2]; [1]State Pedagogical University, Dept. of Gen. Phys., Lenin St. 86, Voronezh 394043 Russia; [2]Osaka City University, Dept. of Intelligent Matls. Eng., Sugimoto 3-3-138, Sumiyoshi-ku, Osaka 558-8585 Japan

A complex statistical and spectral analysis of acoustic emission (AE) during plastic flow of metallic glasses is performed aiming at clarification of mechanisms of their inhomogeneous plastic deformation. The AE power spectra and waveforms of individual signals are analyzed in detail. The onset and kinetics of shear band nucleation have been clearly detected by means of AE. Possible mechanisms of inhomogeneous deformation are discussed in light of AE results.

Alumina and Bauxite: Alumina Industry Trends, Products, Environment

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

Wednesday AM	Room: 609
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: TBA

8:30 AM Invited
The Alumina Industry Technology Roadmap: *Ivan Anich*[1]; [1]Alcoa World Alumina, PO Box 252, Applecross, Western Australia 6153 Australia

In an unprecedented global collaboration, more than 30 international leaders in the alumina refining industry have reached a consensus on the R&D activities needed to address the technical challenges over the next 20 years. The result is the Alumina Industry Technology Roadmap, a strategic plan containing a goal-based R&D agenda that can be pursued by both individual companies and collaborative partnerships between companies, government and other members of the research community. The framework was a set of explicit targets for reducing capital and operating costs; improving energy efficiency; enhancing environmental performance, safety and health; and meeting customersí needs. An alumina techniology committee has been established as a focal point for implementation of the roadmap. The roadmap was coordinated by AMIRA International, and supported by the US Department of Energy; the Australian Department of Industry, Science and Resources; the Aluminium Association; the Australian Aluminium Council; and the Government of Western Australia.

9:00 AM
Extraction of Aluminium Oxide from Bauxite and Redmud: *Vilas D. Tathavadkar*[1]; M. P. Antony[2]; Animesh Jha[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK; [2]Post-Irradiation Studies Section, Fuel Chem. Div., IGCAR, Kalpakkam, TN 603 102 India

Pure alumina (Al2O3) is generally extracted from bauxite via the Bayer process. A complete recovery of alumina is economically not possible via this process. The safe disposal of process residue, red mud, is one of the disadvantages of Bayer process. A novel route for extracting alumina from bauxite has been developed. The process involves roasting of bauxite with an alkali carbonate in air at temperatures above 1050K. The roasted mass is digested in water and filtered. Al(OH)3 is precipitated by adjusting the pH of the solution. Pure alumina is produced by calcining the Al(OH)3 precipitate. The extraction efficiency of alumina is more than 98%. Alkali carbonates can be recovered by evaporating the filtrate after removing Al(OH)3 precipitate. A mechanism based on XRD and SEM of the roasted product is proposed. Alumina from red mud can also be extracted in a similar way. Based on the alkali-roasting technique a zero-waste technology for alumina extraction can be devised.

9:25 AM
Effect of Thermal Pretreatment on Grindability and Upgradation of Bauxite for Refractory Applications: R. Bhima Rao[1]; *M. B. Veeresh*[1]; G. N. Banerjee[1]; [1]Karnataka University, Dept. of Minl. Procg., Regional Rsrch. Lab., (Council of Scientific & Indl. Rsrch.), Bhubaneswar-751013 India

Iron is a detrimental element in bauxite used for refractory applications. A process has been developed in the laboratory on removal of iron and improvement in alumina recovery of bauxite by reduction/calcination roasting followed by magnetic separation. For effective liberation of mineral the ore is to be comminuted to the desired fineness. As this process is energy intensive, a study has been undertaken on determination of overall energy requirement for grinding of ore as well as ore samples were subjected to various pretreatments as reduction roasting with coke/calcination-quenching in water at room temperature for effective liberation of minerals. It has been found that the thermal pretreatment method has several advantages in terms of grinding time, magnetic intensity, alumina recovery, compared to the crude ore.

9:50 AM Break

10:10 AM Cancelled
Boiler Efficiency Improvements in a Bayer Process Plant: *Walters Leighton*

10:35 AM
Future of Guinea Bauxite Mining and Alumina Industry Development: *Siafa Coulibaly*[1]; [1]Ministry of Mines, Geology and Environment, Conakry Guinea

This paper discusses the various aspects of the future alumina-bauxite industry in Guinea. Guinea is endowed with enormous reserves of bauxite estimated to several billion tons of good grade material within 200 kms of the coast, plus a major bauxite province along the northeast slope of the Fouta Djallon mountain. Guinea bauxite reserves development has, over the past forty years, been focused on three specific areas not far from the coast: Fria deposits, Compagnie des Bauxites de GuinÈe, Compagnie des Bauxites de Kindia formerly SBK. Guinea geographic location, particularly in regard to European, North American and African markets is quite favourable. It is also a fact that Guinea has a relatively untapped hydroelectric potential based on the KonkourÈ river which flows into the Atlantic ocean. The larger project, located 40 kms upstream from Fria is Kaleta-Souapiti, estimated at 1000 MW. Guinea coastal and internal infrastructure is reasonable, but needs improvement.

Aluminum Reduction Technology: Cathode Materials –Joint Carbon Technology

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

Wednesday AM Room: 6B
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Siegfried Wilkening, VAW Aluminium-Technologie GmbH, Georg-v-Boeselagerstr. 25, Bonn 53117 Germany

8:30 AM
Anthracite Evaluation for Amorphous Cathodes: *Frank Hiltmann*[1]; Boguslawa Jesionek[2]; Janusz Tomala[2]; Maciej Bojkowski[3]; [1]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, Frankfurt 65933 Germany; [2]ZEW S.A., ul Piastowska 29, Raciborz 47-400 Poland; [3]SGL Carbon S.A., ul. Wegierska, Nowy Sacz 33-300 Poland

One of the key factors for production of high quality cathodes is the selection of proper raw materials. Especially for amorphous cathodes, which usually contain 50-70% of gas or electrically calcined anthracite, the right choice is essential with regard to the higher chemical sensitivity of anthracite vs. graphite. The process of characterizing green anthracites, with emphasis on petrographic analyses, is described and a classification system presented. The lab procedure for estimating the calcination behavior is outlined and the suitability for use in cathode manufacture discussed.

8:55 AM
Porosity Modifications in the Carbon Cathode of Aluminum Reduction Cell II: *Mohamed Othman Ibrahiem*[1]; S. M. El-Raghy[2]; F. M. Ahmed[1]; [1]Aluminum Company of Egypt, R&D Dept., Nag-Hammadi Egypt; [2]Cairo University, R&D Dept., Nag-Hammadi Egypt

In a previous study, reduction of cathode porosity was achieved by impregnation with a high carbon yield resin (furfuryl alcohol), followed by curing and pyrolysis for several cycles. The aim of this study was further characterization and investigation under electrolysis conditions. Thermal conductivity increased from 7.66 W/mK to 10.45 W/mK when the open porosity decreased from 15% to 1% by multiple impregnation/pyrolysis cycles. An increase in ash content was observed as a function of impregnation cycles. The influence of applied current has been studied with a current density of 0.7 A/cm2 in a basic cryolite-alumina melt for durations up to 3 hours. The investigation included measurement of sodium entry, ash content, thermal conductivity, compressive strength, specific electrical resistance, and structure by optical microscopy. Passing electric current increases the sodium level in the cathode and increases its electrical and thermal conductivity. Modified cathodes are less reactive under working conditions.

9:20 AM
A New Ramming Paste for the Aluminum Electrolysis Cell Fitting with Technical and Environmental Issues: Daniel Dumas[1]; Simon Meseguer[1]; Regis Paulus[1]; *Serge Lacroix*[2]; [1]Carbone Savoie, R&D Dept., 30, Rue Louis Jouvet, BP 16, Venissieux, Cedex 69631 France; [2]UCAR SNC, Apply Tech., La LÈchËre, Aigueblanche 73264 France

The ramming paste is known to be a key material in the Aluminum electrolysis cell. The paste must have the relevant properties to avoid poor pot performances. In addition the emission of hazardous compounds must be at the lowest possible level during densification and baking. Coal tar pitch usually used as binder for the ramming pastes contains polycyclic aromatic hydrocarbons, some of which are classified as genotoxic. The present work describes how to decrease significantly the polycyclic aromatic hydrocarbon emissions by using the appropriate binder while maintaining the relevant characteristics of the cold ramming paste regarding pot performances. The data obtained in test pots are included.

9:45 AM
Use of Ceramic Materials for Filling Joints in Cell Cathode Blocks: *Gennadiy Vladimirovich Solonin*[1]; Michail Alexandrovich Fridman[1]; Vladimir Kuzmich Nikitenko[1]; [1]JSC ìZaporozhye Aluminum Worksî (ZALK), 15, Yuzhnoye Shosse, Zaporozhye 69032 Ukraine

The need to improve performance characteristics of cathode lining, in particular to increase cell life, is driven by the high cell repair costs and the disposal of environmentally harmful wastes formed during the reduction cell relining. This paper presents a method of improving the performance characteristics of the cathode blocks in an aluminium reduction cell. A procedure has been developed and the results of the laboratory tests of ceramic materials used in the cathode joints are given. It has been shown that the resistance of joint materials to the melt attack is determined by the porosity of the materials used. The experimental and industrial test results of using a slip casting procedure for filling the cathode blocks joints are given. The results achieved made it possible to eliminate the use of carbon ramming paste from the cathode relining procedure. It has been demonstrated that after the introduction of the new cell relining procedure, there were no premature cell failures even under long electric power outages. Other positive effects of the procedure presented have also been achieved.

10:10 AM Break

10:20 AM
The Role of Silica Rich-Low Permeability Bricks in Multilayer Barrier Design of Aluminum Electrolytic Cells: Don Harris[1]; John Lam[1]; *George Oprea*[2]; [1]Clayburn Refractories, Ltd., 33765 Pine St., Abbotsford, BC V2S 5C1 Canada; [2]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Multilayer barrier designs that employ a combination of different refractories, are standard practices for numerous aluminum smelters. While operating results confirmed the efficiency of such systems, no laboratory work or testing methods had been presented to confirm the effectiveness nor the optimization of the combination of bricks utilized. This paper discusses a test method comparing different refractory brick compositions when exposed to various cryolitic-based environments, simulating the changes in bath chemistry beneath the cathode. The penetration and corrosion results were correlated with the testing parameters, the refractoriesí chemistry and microstructure as well as with the changes in reacted bath chemistry during penetration.

10:45 AM
Aging of Cathode Refractory Materials in Aluminium Reduction Cells: *Rikke Weibel*[1]; Leo Fisher Juhl[1]; Bent Nielsen[1]; Joergen Mikkelsen[1]; Ole-Jacob Siljan[2]; Kjeld Thovsen[2]; Sara Thornblad Mathisen[2]; [1]Skamol, Oestergade 58-60, Nykoebing DK-7900 Denmark; [2]Hydro Aluminium, Rsrch. Ctr. Porsgrunn, PO Box 2560, Porsgrunn N-3901 Norway

Aging of refractory materials during cell life has been studied in respect to changes in thermal conductivity and consequently influence on the heat balances. Autopsies have been performed on electrolytic cells with ages ranging from 500 days to 3200 days. Samples from different positions and layers in the cells, and of three different insulating materials are represented. Thermal conductivity measurements of the small autopsy samples have been performed by the Hot Disk method. During operation of the reduction cells the thermal conductivity of the pot lining material may change substantially (according to position in the cell, which is related to the degree of compression, precipitation of solids in pores, etc.) The physical properties of the virgin material (density, porosity, compressibility, etc.) also influences on the changes in thermal conductivity. The measured general increase in thermal conductivity of ìspentî pot lining materials compared with similar virgin materials reflects the necessity of increasing cell voltage gradually during cell life in order to compensate for increased heat loss. Modelling of heat balances have been based on the thermal conductivity measurements on autopsy samples from three different ways of insulating the cathode construction, comparing two grades of moler bricks (HIPOR and SUPRA) with a combination of calcium silicate (SUPER-1100 E) and moler bricks (SUPRA). Heat balance modelling has showed that the lowest possible thermal conductivity of the insulating material is not the only important factor, also the evolution of the thermal conductivity over time is important (SUPER-1100 E versus HIPOR versus SUPRA).

11:10 AM
Environmental and Economic Aspects of an Effective Diffusion Barrier: *E. Sturm*[1]; J. Prepeneit[1]; [1]Hamburger Aluminium-Werk GmbH, Hamburg 21129 Germany

In pot lining there is a long history of more or less effective diffusion barriers. Some aspects of SPL, the need of reusing materials and further lowering of energy consumption has to be regarded. At HAW a double layer barrier in combination with alumina insulation has been experimented over more than 2000 days. The influence on heat balance has to be taken into account within a total lining concept, that includes cathode type and pot shell changes. The energy saving derives from a decrease in pot voltage of 50 mV. The mass balance of bath additives, impurities and fluorid is changed. Due to bath production there is a demand for low content of Na20 in alumina. Waste dumping of lining materials using alumina as insulation can be reduced. The cost balance of all the previous aspects reveals an economic solution.

11:35 AM
Aluminum-Steel Electrical Transition Joints, Effects of Temperature and Time upon Performance: *John G. Banker*[1]; Antoine Nobili[2]; [1]Dynamic Materials Corporation, Clad Metal Div., 5405 Spine Rd., Boulder, CO 80301 USA; [2]Dynamic Materials Corporation, Nobelclad Div., 1 Allee Alfred Nobel, Rivesaltes, 66600 France

Aluminum-steel electric transition joints (ETJ) are often used in aluminum and magnesium reduction cells for making welds between aluminum buss and steel anodes and cathodes. Depending upon the cell design and operation, ETJís can operate at temperatures ranging between 200°C and 500°C, possibly hotter. Over time, these thermal conditions can have significant deleterious effect upon the ETJ bond strength, resulting in eventual failure. A detailed study was performed measuring ETJ bond strength as a function of time, temperature, and design. Specimens were maintained at temperatures of 300°C to 600°C for up to one year, and then tensile tests were performed. Metallographic and spectrographic analyses were performed to analyze the mechanism of bond degradation. Diffusion and subsequent intermetallic formation were the mechanisms causing bond degradation. Theoretical diffusion equations provide a basis for extrapolation of test data to longer time periods. The addition of a thin interlayer between the aluminum and steel can alter this behavior. Several interlayer materials were evaluated, including chromium and titanium. Titanium provided the most significant improvement in performance. Extrapolations indicate that titanium interlayer ETJís can be expected to operate for over 10 years at 450°C without significant degradation.

Aluminum Sheet and Plate Rolling & Finishing Technology and Applications: Aluminum Sheet and Plate Processing

Sponsored by: Light Metals Division, Aluminum Association, *Program Organizers:* Michael H. Skillingberg, The Aluminum Association, Inc., Washington, DC 20006 USA; John P. Brandimarte, Precision Coil, Inc., Clarksburg, WV 26302-2650 USA; Karl Eminger, Alcoa, Inc., Alcoa Mill Products, Lancaster, PA 17604 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Jeff J. Kadilak, Alcan, Inc., Oswego, NY 13126 USA; Leland R. Lorentzen, Nichols Aluminum, Davenport, IA 52801 USA; Douglas N. McLeod, ARCO Aluminum, Inc., Louisville, KY 40242 USA; Ken Schreckengast, Coastal Aluminum Rolling Mills, Inc., Williamsport, PA 17701 USA

Wednesday AM — Room: 608
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Jeff J. Kadilak, Alcan, Inc., PO Box 28, Oswego, NY 13126 USA

8:30 AM
Strip Casting Technology-A Key to Product Quality: *Frederic Basson*[1]; Pierre-Yves Menet[1]; Klaus Maiwald[1]; Marc Bosch[1]; [1]Pechiney Aluminium Engineering, Continuous Casting Tech., Centrialp, BP 24, Voreppe, Cedex 38341 France

Pechiney has been dedicated to developing twin roll casting of aluminum technology and products for more than 40 years. Lately a new generation of machines, the Jumbo 3CM, has emerged to complete the range of equipment available to produce hot rerolls on an economical basis. The Jumbo 3CM includes the latest technological developments that Pechiney has designed in order to: Reduce Casting gauge; Increase productivity and casting width; Increase quality of existing products and improve consistency of the cast quality; Develop new products from continuous cast sheet. The presentation reviews the numerous technological developments that are now commercially available: liquid metal treatment (Alpur, Pechiney Deep Bed Filter), patented metal feeding into the caster with accurate metal level control and temperature control, patented roll cooling system, improved coating control device (new release-agent spraying technology), cross and longitudinal profile monitoring, casting supervision system. These developments have made possible the casting of alloys for more demanding products such as can stock, thin gauge foil, litho sheet and automotive sheet. Existing casters can also benefit from these developments.

8:55 AM
Aluminium Strip & Foil Rolling-Leading Technologies on the Market: *Axel E. Barten*[1]; [1]Achenbach Buschh¸tten GmbH, Siegener Str. 152, Kreuztal D-57223 Germany

The production of wider and thinner aluminum light gauge strip and foil with best quality and high rolling speeds as well as short change times for a high productivity is the key to the success of a growing number of producers world wide. Advanced machinery, systems, controls and technology provided by Achenbach Buschh¸tten are necessary to reach this excessive goal. The OPTIROLL INTRAPLANTÆ adaptive automation system fulfills all requirements of modern rolling mill technology. Its big advantage lies in its modular design to be installed either in single components or as a complete system using all synergy effects of their combination. All movements and interlocks are realized in two levels in the OPTIROLL INTRAPLANTÆ System: The manual and the automatic control level. The new OPTIROLLÆ flatness measuring roll has been modified completely to optimize the measuring results. The fully digital flatness control system consists of four independent control loops. Roll tilting, work roll bending and variable crown are servo controlled. WIN Spray SÆ is the ultimate cooling system for aluminum rolling mills. The patented OPTIROLL -SGCÆ (Spacer Gauge Control) system with control parameters: roll separating force, adjustment cylinder position, mass flow, strip tensions and rolling speed is described.

9:20 AM
An Approach to Understanding Mill Vibration: *Thomas W.D. Farley*[1]; [1]Alcan International, Ltd., Southam Rd., Banbury, Oxon OX16 2SP UK

Alcan Inc. is a multinational company engaged in all aspects of the aluminium industry. This paper presents an approach adopted by Alcan to understand and solve mill vibration issues experienced on Alcanís cold rolling mills. The different types of mill vibration phenomena are described with reference to our current understanding. Predictions from a computer model of a mill stand are presented and compared to experimental measurements for 3rd and 5th Octave Chatter. Examples from other computer simulations are described that have been used to justify engineering projects to solve vibration problems.

9:45 AM
Roll Cooling Concepts: *Bernard J. Forster*[1]; *David Tucker*[2]; [1]Lechler, Inc., 445 Kautz Rd., Saint Charles, IL 60174 USA; [2]Lechler, Ltd., 1 Fell St., Newhall, Sheffield S9 2TP Great Britain

Properly cooling the rolls in a rolling mill is critical to product quality, product quantity and roll life. To accomplish it effectively, a thorough understanding of current coolant application theories and commonly used practices is necessary. Managing heat in a rolling mill begins with understanding how it is generated. Sources of heat include radiation, deformation, and friction. The role of coolant is to remove heat from the rolls, the product or both. How this is accomplished can affect performance factors such as product shape, roll life, and surface defects. Properly managing heat requires a consideration of several design parameters. The target surfaces, header location, and nozzle characteristics are among the many factors, which must be defined correctly. The purpose of this paper will be to define the current theories on roll cooling applications, compare them with popular application principles and establish a design methodology for effective and economical roll cooling.

10:10 AM Break

10:20 AM Cancelled
Coolant Mist and VOC Control Systems Applied to Hot and Cold Rolling Mills in the Aluminum and Steel Industry: *Richard W. Perryman*

10:45 AM
OPTIPURE-Integrative Filtration Technology for Economical and Ecological Efficiency: *Axel E. Barten*[1]; [1]Achenbach Buschh¸tten GmbH, Siegener Str. 152, Kreuztal D-57223 Germany

For aluminum rolling plants Achenbach Buschh¸tten is providing a complete rolling oil management package under the trade name of OPTIPUREÆ Filtration Technology, following an integrative system philosophy instead of offering ìadditionsî. By this way economical needs are ideally matched with ecological requirements of the rolling process. It gets feasible by the consequent pursuit of the ìclosed loop principleî in our OPTIPUREÆ technology as well as by the extremely high process efficiency of each and every component. Optimum results in rolling as well as highest regeneration rates of the processed rolling oils are achieved. At the same time negative influences on the environment on air, soil and water are minimized. All OPTIPUREÆ

WEDNESDAY AM

systems are equipped with comprehensive visualization packages for online monitoring the automatic filtration process. Essentially, three different types of filtration systems are available: Fume Exhaust Filtration Systems for Rolling Mills: The AIRPUREÆ systems achieve a continuous recovery of evaporated roll coolants from the exhaust air combined with a complete oil treatment process in order to provide ìnew qualityî rolling oil directly back to the rolling process. Micro-Filtration Systems for Roll Coolants: The SUPERSTACKÆII horizontal plate filter systems are the ideal supplement by mechanically filtering all rolling generated particles full-stream out of the mill coolant. Modern filters have a CONDUSENSÆ sensor for on-line measurement of the electrical conductivity of poor conducting liquids. By measuring conductivity, dielectric permittivity and temperature of the rolling oil used in foil rolling, its contamination, the danger of uncontrolled discharges and the inherent fire risk are continuously determined. Rectification Systems for Roll Coolants: The WORÆ systems ìsuperpolishî roll coolants by removing non-mechanical contaminants such as tramp oils, thus closing the complete ìrecycling loopî. These systems can be utilized as stand-alone devices. The strictly modular design allows to optimize each application for any specific mill requirement and to comply with the individual customersí requests. Therefore OPTIPUREÆ systems are not only the ultimate solution for new mills but also for the retrofit of existing ones. Applied in combination as an integrated system the user profits from all synergy effects between the single devices. These effects are: Minimized roll coolant consumption in the rolling process; Consistent high roll coolant quality; Minimized pollution of the environment by reduction of wastes; Optimum energy efficiency.

11:10 AM

Truly Universal Non-Contact Thickness Sensors: *Christopher Burnett*[1]; Gavin Carmichael[1]; Dino Jardina[1]; [1]Thermo-Radiometrie, Sensor & Electl. Dvlp., 9298 Gaither Rd., Gaithersburg, MD 20877 USA

The tight spaces, temperature variations and general environment of the numerous breeds and generations of rolling mills provide inherent physical limits for thickness sensor designs. Compounding these mechanical constraints, thickness sensors must provide higher performance to measure the seemingly infinite variations in alloy chemistries to tighter and tighter tolerances. Striking a balance of high performance and mechanical integrity has been an ongoing effort by gauge manufacturers. As the end market continues to engineer methods of producing more with less material, the gauge manufacturers must design sensors that provide higher performance in a smaller package. To answer the challenge presented by these limits, Thermo Radiometrie has developed a sensor to provide the performance of an x-ray based thickness sensor, capable of measuring from the thinnest foils up to the thicker sheet direct from the hot mill, all in the mill envelope equivalent to an isotope sensor.

11:35 AM

New Annealing Concept for Aluminum Coils: *Dominik Schroeder*[1]; Holger Kehler[1]; [1]Schmitz+Apelt LOI, Clausewitzstrasse 82, D-42389 Wuppertal, Germany

The options of rationalization of roller hearth or continuous strip annealing furnaces for aluminum coils are limited. A line of individual-chamber furnaces, raised above the floor and charged from underneath, renders new possibilities to increase the flexibility and economical operation of the annealing process. Each individual chamber anneals one (1) coil and is equipped with an indirect, natural gas-fired heating system, a high volume flow, variable speed circulation fan, and a cooling unit. Each furnace chamber uses its own PLC, allowing the chambers to operate independently of each other. The furnaces are designed for inert gas operation. The entire annealing process runs without oxygen. In order to save floor space, the chambers are charged from underneath by a mobile, scissors-type lifting table. This new annealing concept complies with all economic and quality requirements, such as: short heating and cooling times, exact temperature control, to prevent superheating at the coilís edges, high temperature accuracy during soak, individual annealing programs, energy-saving operation and minimum maintenance required. A plantís annealing capacity can be increased most easily by adding one or more additional chambers to the line.

Carbon Technology: Cathode Materials –Joint Aluminum Reduction Technology

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

Wednesday AM — Room: 6B
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Siegfried Wilkening, VAW Aluminium-Technologie GmbH, Georg-v-Boeselagerstr. 25, Bonn 53117 Germany

8:30 AM

Anthracite Evaluation for Amorphous Cathodes: *Frank Hiltmann*[1]; Boguslawa Jesionek[2]; Janusz Tomala[2]; Maciej Bojkowski[3]; [1]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, Frankfurt 65933 Germany; [2]ZEW S.A., ul Piastowska 29, Raciborz 47-400 Poland; [3]SGL Carbon S.A., ul. Wegierska, Nowy Sacz 33-300 Poland

One of the key factors for production of high quality cathodes is the selection of proper raw materials. Especially for amorphous cathodes, which usually contain 50-70% of gas or electrically calcined anthracite, the right choice is essential with regard to the higher chemical sensitivity of anthracite vs. graphite. The process of characterizing green anthracites, with emphasis on petrographic analyses, is described and a classification system presented. The lab procedure for estimating the calcination behavior is outlined and the suitability for use in cathode manufacture discussed.

8:55 AM

Porosity Modifications in the Carbon Cathode of Aluminum Reduction Cell II: *Mohamed Othman Ibrahiem*[1]; S. M. El-Raghy[2]; F. M. Ahmed[1]; [1]Aluminum Company of Egypt, R&D Dept., Nag-Hammadi Egypt; [2]Cairo University, R&D Dept., Nag-Hammadi Egypt

In a previous study, reduction of cathode porosity was achieved by impregnation with a high carbon yield resin (furfuryl alcohol), followed by curing and pyrolysis for several cycles. The aim of this study was further characterization and investigation under electrolysis conditions. Thermal conductivity increased from 7.66 W/mK to 10.45 W/mK when the open porosity decreased from 15% to 1% by multiple impregnation/pyrolysis cycles. An increase in ash content was observed as a function of impregnation cycles. The influence of applied current has been studied with a current density of 0.7 A/cm2 in a basic cryolite-alumina melt for durations up to 3 hours. The investigation included measurement of sodium entry, ash content, thermal conductivity, compressive strength, specific electrical resistance, and structure by optical microscopy. Passing electric current increases the sodium level in the cathode and increases its electrical and thermal conductivity. Modified cathodes are less reactive under working conditions.

9:20 AM

A New Ramming Paste for the Aluminum Electrolysis Cell Fitting with Technical and Environmental Issues: Daniel Dumas[1]; Simon Meseguer[1]; Regis Paulus[1]; *Serge Lacroix*[2]; [1]Carbone Savoie, R&D Dept., 30, Rue Louis Jouvet, BP 16, Venissieux, Cedex 69631 France; [2]UCAR SNC, Apply Tech., La LÈchÈre, Aigueblanche 73264 France

The ramming paste is known to be a key material in the Aluminum electrolysis cell. The paste must have the relevant properties to avoid poor pot performances. In addition the emission of hazardous compounds must be at the lowest possible level during densification and baking. Coal tar pitch usually used as binder for the ramming pastes contains polycyclic aromatic hydrocarbons, some of which are classified as genotoxic. The present work describes how to decrease significantly the polycyclic aromatic hydrocarbon emissions by using the appropriate binder while maintaining the relevant characteristics of the cold ramming paste regarding pot performances. The data obtained in test pots are included.

9:45 AM

Use of Ceramic Materials for Filling Joints in Cell Cathode Blocks: *Gennadiy Vladimirovich Solonin*[1]; Michail Alexandrovich Fridman[1]; Vladimir Kuzmich Nikitenko[1]; [1]JSC ìZaporozhye Aluminum Worksî (ZALK), 15, Yuzhnoye Shosse, Zaporozhye 69032 Ukraine

The need to improve performance characteristics of cathode lining, in particular to increase cell life, is driven by the high cell repair costs and the disposal of environmentally harmful wastes formed during the reduction cell relining. This paper presents a method of improving the performance characteristics of the cathode blocks in an aluminium reduction cell. A procedure has been developed and the results of the laboratory tests of ceramic materials used in the cathode joints are given. It has been shown that the resistance of joint materials to the melt attack is determined by the porosity of the materials used. The experimental and industrial test results of using a slip casting procedure for filling the cathode blocks joints are given. The results achieved made it possible to eliminate the use of carbon ramming paste from the cathode relining procedure. It has been demonstrated that after the introduction of the new cell relining procedure, there were no premature cell failures even under long electric power outages. Other positive effects of the procedure presented have also been achieved.

10:10 AM Break

10:20 AM
The Role of Silica Rich-Low Permeability Bricks in Multilayer Barrier Design of Aluminum Electrolytic Cells: Don Harris[1]; John Lam[1]; *George Oprea*[2]; [1]Clayburn Refractories, Ltd., 33765 Pine St., Abbotsford, BC V2S 5C1 Canada; [2]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Multilayer barrier designs that employ a combination of different refractories, are standard practices for numerous aluminum smelters. While operating results confirmed the efficiency of such systems, no laboratory work or testing methods had been presented to confirm the effectiveness nor the optimization of the combination of bricks utilized. This paper discusses a test method comparing different refractory brick compositions when exposed to various cryolitic-based environments, simulating the changes in bath chemistry beneath the cathode. The penetration and corrosion results were correlated with the testing parameters, the refractoriesí chemistry and microstructure as well as with the changes in reacted bath chemistry during penetration.

10:45 AM
Aging of Cathode Refractory Materials in Aluminium Reduction Cells: *Rikke Weibel*[1]; Leo Fisher Juhl[1]; Bent Nielsen[1]; Joergen Mikkelsen[1]; Ole-Jacob Siljan[2]; Kjeld Thovsen[2]; Sara Thornblad Mathisen[2]; [1]Skamol, Oestergade 58-60, Nykoebing DK-7900 Denmark; [2]Hydro Aluminium, Rsrch. Ctr. Porsgrunn, PO Box 2560, Porsgrunn N-3901 Norway

Aging of refractory materials during cell life has been studied in respect to changes in thermal conductivity and consequently influence on the heat balances. Autopsies have been performed on electrolytic cells with ages ranging from 500 days to 3200 days. Samples from different positions and layers in the cells, and of three different insulating materials are represented. Thermal conductivity measurements of the small autopsy samples have been performed by the Hot Disk method. During operation of the reduction cells the thermal conductivity of the pot lining material may change substantially (according to position in the cell, which is related to the degree of compression, precipitation of solids in pores, etc.) The physical properties of the virgin material (density, porosity, compressibility, etc.) also influences on the changes in thermal conductivity. The measured general increase in thermal conductivity of ìspentî pot lining materials compared with similar virgin materials reflects the necessity of increasing cell voltage gradually during cell life in order to compensate for increased heat loss. Modelling of heat balances have been based on the thermal conductivity measurements on autopsy samples from three different ways of insulating the cathode construction, comparing two grades of moler bricks (HIPOR and SUPRA) with a combination of calcium silicate (SUPER-1100 E) and moler bricks (SUPRA). Heat balance modelling has showed that the lowest possible thermal conductivity of the insulating material is not the only important factor, also the evolution of the thermal conductivity over time is important (SUPER-1100 E versus HIPOR versus SUPRA).

11:10 AM
Environmental and Economic Aspects of an Effective Diffusion Barrier: *E. Sturm*[1]; J. Prepeneit[1]; [1]Hamburger Aluminium-Werk GmbH, Hamburg 21129 Germany

In pot lining there is a long history of more or less effective diffusion barriers. Some aspects of SPL, the need of reusing materials and further lowering of energy consumption has to be regarded. At HAW a double layer barrier in combination with alumina insulation has been experimented over more than 2000 days. The influence on heat balance has to be taken into account within a total lining concept, that includes cathode type and pot shell changes. The energy saving derives from a decrease in pot voltage of 50 mV. The mass balance of bath additives, impurities and fluorid is changed. Due to bath production there is a demand for low content of Na20 in alumina. Waste dumping of lining materials using alumina as insulation can be reduced. The cost balance of all the previous aspects reveals an economic solution.

11:35 AM
Aluminum-Steel Electrical Transition Joints, Effects of Temperature and Time upon Performance: *John G. Banker*[1]; Antoine Nobili[2]; [1]Dynamic Materials Corporation, Clad Metal Div., 5405 Spine Rd., Boulder, CO 80301 USA; [2]Dynamic Materials Corporation, Nobelclad Div., 1 Allee Alfred Nobel, Rivesaltes, 66600 France

Aluminum-steel electric transition joints (ETJ) are often used in aluminum and magnesium reduction cells for making welds between aluminum buss and steel anodes and cathodes. Depending upon the cell design and operation, ETJís can operate at temperatures ranging between 200°C and 500°C, possibly hotter. Over time, these thermal conditions can have significant deleterious effect upon the ETJ bond strength, resulting in eventual failure. A detailed study was performed measuring ETJ bond strength as a function of time, temperature, and design. Specimens were maintained at temperatures of 300°C to 600°C for up to one year, and then tensile tests were performed. Metallographic and spectrographic analyses were performed to analyze the mechanism of bond degradation. Diffusion and subsequent intermetallic formation were the mechanisms causing bond degradation. Theoretical diffusion equations provide a basis for extrapolation of test data to longer time periods. The addition of a thin interlayer between the aluminum and steel can alter this behavior. Several interlayer materials were evaluated, including chromium and titanium. Titanium provided the most significant improvement in performance. Extrapolations indicate that titanium interlayer ETJís can be expected to operate for over 10 years at 450°C without significant degradation.

Cast Shop Technology: Metal Treatment

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

Wednesday AM	Room: 6A
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Michael Scherbak, Alcoa Inc., Ingot Tech., 900 S. Gay St., Riverview Tower, Knoxville, TN 37902 USA; Masood Al-Rais, Dubai Aluminium, Techl. Services, PO Box 3627, Dubai UAE

8:30 AM Keynote
A Technical Perspective of Molten Aluminum Processing: *Peter D. Waite*[1]; [1]Alcan International, Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

In todayís context of global competitiveness, all factors related to molten metal treatment which directly or indirectly affect product quality, the environment and processing costs, must be optimized. In this regard, technology and innovation play a decisive role for the development and implementation of the most appropriate molten metal treatment processes and practices. The following discussion will review the most recent significant developments in the field of molten aluminum processing, and outline potential areas for improvement.

9:00 AM
Investigation of the Process Variables Influencing Particulate Generation from Aluminum In-Line Metal Treatment Units: *Kevin A. Kitzman*[1]; [1]Alcoa, Inc., Environml. Sci. & Tech. Dvpt., Alcoa Technical Ctr., 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

Maximum Achievable Control Technology (MACT) standards under the Clean Air Act set tough new limits on particulate and hydrogen chloride emissions from aluminum in-line degassing units that utilize chlorine. It is desirable to comply with these limits through process adjustments and/or redesign rather than through expensive end-of-pipe controls. Emissions sampling of five (5) types of in-line degassing technologies (3 vendor and 2 internal models) was conducted at various Alcoa plants under actual operating conditions to collect data on emission rates and process parameters. Emission results are presented against several operational factors to determine the factors that most significantly impact emissions. The most dramatic reduction in particulate emissions occurs by sealing the headspace above the metal, thereby preventing ambient air from entering. The impact of other significant process parameters on emissions is also discussed.

9:25 AM
Hycast I-60 SIR-A New Generation Inline Melt Refining System: *Geir MÊland*[1]; Erling Myrbostad[1]; Karl VenÂs[2]; [1]Hydro Aluminium, Hycast a.s., Industriveien 49, Sunndals□ra 6600 Norway; [2]S□r-Tr□ndelag University College, Trondheim Norway

A new generation inline melt refining unit has recently been developed by Hydro Aluminium, Hycast a.s. A traditional gas fluxing principle combined with a unique design, gives optimal utilization of the fluxing gas, and eliminates some of the disadvantages of the conventional degassing systems available today. The unit is characterized by very high removal of Hydrogen and inclusions. The operational costs are low. This is due to very low Argon gas consumption, a cheap preheating system, a drainfree reactor and relatively few rotors in operation. A two rotor system has a capacity of more than 65 metric tons pr. hour. The system is fully automated.

9:50 AM
A Radioscopic Technique to Observe Bubbles in Liquid Aluminium: Marc Bertherat[1]; Thierry Odievre[2]; *Michel P. Allibert*[1]; Pierre Le Brun[2]; [1]INP Grenoble, LTPCM, ENSEEG, BP75, St Martin díHeres 38402 France; [2]Pechiney Centre de Recherches de Voreppe, 725 rue Aristide Berges, BP 27, Voreppe, Cedex 38341 France

A real time radioscopic visualization technique was used to observe the shape and motion of argon bubbles in liquid aluminium. The images provided by an X-ray generator on a radiographic screen were recorded and treated to extract the width and velocity of the bubbles. The treatment was validated by observations, at room temperature, of hollow spheres machined in a rotating aluminium block. The size and the velocity of bubbles were measured in a parallelipipedic graphite crucible where the bubbles could flow up without interactions with the crucible walls.

10:15 AM Break

10:30 AM
Hydrogen Removal Efficiency of In-Line Degassing Units: *Pierre Le Brun*[1]; [1]Pechiney Centre de Recherches de Voreppe, 725, rue Aristide Berges, BP27, Voreppe, Cedex 38341 France

The performance of in-line degassing units is often characterized by its hydrogen removal efficiency, defined by (hydrogen in-hydrogen out)/hydrogen in. This efficiency value has gained a wide acceptance as it is easy to define, understand, and measure, and is expected to be constant. Nevertheless, the meaning of the efficiency of a degassing unit is not often discussed. In this paper, we report on industrial experience, lab-scale experiments, and modeling in order to provide a better understanding of the factors that govern the degassing efficiency of an in line unit. We illustrate, among others, the influence of the following parameters: up-stream quality, casting parameters, control of the equipment, and external parameters. The benefits of a numerical hydrodynamic modeling approach are also illustrated.

10:55 AM
Methods for Alkaline Reduction: Either in the Potroom Crucible or in the Casthouse?: *Martin Bradley Taylor*[1]; [1]Stas, 1846 Outarde, Chicoutimi, Quebec G7K IHI Canada

Lithium, sodium, calcium and magnesium may all be reduced efficiently from molten aluminium to very low levels of ppm, in the pot room crucible before transfer to the melter/holder furnace and this within a few minutes; no chlorine is required, only the use of aluminium fluoride salt. Some removal, particularly of sodium, may also be obtained by simply transferring from one crucible to another whilst transporting or delivering molten aluminium, without the introduction of either gas or salt. On the other hand these same elements may also be reduced in the holder furnace in the casthouse, using either salts or gases by various means including manual additions of fluxes; or chlorine lances; or more recently rotary gas/flux injectors. Degassers may also be used as in-line treatment for removing alkalines with the use of argon and chlorine.

11:20 AM
Solubility of Carbon in Aluminium and its Effect upon the Casting Process: *John R□dseth*[1]; Bj□rn Rasch[2]; Ole Lund[3]; Jomar Thonstad[4]; [1]Hydro Aluminium, R&D Matls. Tech., HÂvik N-4250 Norway; [2]Hydro Aluminium, R&D Matls. Tech., Sunndals□ra N-6601 Norway; [3]Hydro Aluminium, H□yanger Metallverk, H□yanger N-5901 Norway; [4]Norwegian University of Science and Technology, Dept. Matls. Tech. & Electrochem., Trondheim N-7491 Norway

Carbon is slightly soluble in liquid aluminium (ppm range), the solid phase being aluminium carbide (Al_4C_3). The solubility of carbon in aluminium was determined experimentally, and the content of carbon in the aluminium was monitored on its way from the production cells to the casting line. Periodically the casthouse is plagued with metal that appears to be sticky and rapidly causes clogging of casting nozzles and filters in the casting line. This kind of metal, having no official name, is often called ìsummer metalî or ìblue metalî. The amount of aluminium carbide is closely connected to the occurrence of ìblue metalî, being particularly critical for continuous casting processes like rod casting and strip casting. The paper focuses on the level of carbon in the electrolysis cells and the formation and removal of aluminium carbide in the casting process.

11:45 AM
Assessment of the Hydrogen Level in 319 Aluminum Alloy Melts using the Thermal Analysis Technique: *M. Djurdjevic*[1]; R. Hasenbusch[2]; J. H. Sokolowski[1]; [1]NSERC/Ford-Nemak/University of Windsor Industrial Research Chair, Mechl., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Nemak Corporation of Canada, Casting Process Dvlp. Ctr., 4655 G. N. Booth Dr., Windsor, Ontario N9C 4G5 Canada

One of the major problems associated with cast aluminum components is the formation of porosity. Porosity is a leading cause in the reduction of mechanical properties, loss of pressure tightness and a degradation of the surface appearance in cast components. Porosity formation is generally attributed to shrinkage coupled with a lack of interdendritic feeding during the mushy zone solidification and evolution of hydrogen bubbles due to a sudden decrease in hydrogen solubility during the solidification process. This paper presents the results of studies aimed at developing a new procedure for on-line assessment of hydrogen levels in 319 aluminum melts using the Aluminum Thermal Analysis System (AlTAS). The results of the statistical correlation between characteristics of the cooling curve, melt temperature and chemistry indicate that the level of dissolved hydrogen (as measured by the AlSCAN unit) can be determined by the AlTAS system with $R2 = 0.82$ predictability.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: New Methods for Study of Segregation and Fracture II

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

Wednesday AM — Room: 307-308
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Hiroo Ohtani, Sumitomo Metal Industries, Corporate Rsrch. Labs., 1-8 Fuso-cho, Amagasaki Japan 660-0891; Christian Els‰sser, Max-Planck-Institut f¸r Metallforschung, Institut f¸r Werkstoffwissenschaft, Seestrasse 92, D-70174 Stuttgart Germany

8:30 AM Invited
The Relationship Between the Onset of Plastic Flow in Nearly Perfect Silicon Samples and the Brittle to Ductile Transition: *David P. Pope*[1]; M. Khantha[1]; Robert H. Folk[1]; Vaclav Vitek[1]; [1]University of Pennsylvania, Matl. Sci. & Eng., lrsm Bldg., 3231 Walnut St., Philadelphia, PA 19104 USA

Nearly perfect single crystalline Si wafers have been extensively used in the past ten years to study dislocation activity at the BDTT because the absence of pre-existing dislocations in these samples means that all dislocations are generated at the transition. However to date, most of the experiments have been conducted on notched or pre-cracked samples because it is widely believed that the BDT results from some type of crack-tip phenomenon. We believe that the BDT occurs when a critical combination of stress and temperature results in the generation of an avalanche of dislocations in the material, permitting general yielding. In this view, a crack-tip is not necessary for the transition but it may serve as the stress concentrator that precipitates the avalanche of dislocations. To test this hypothesis, experiments were performed on a large number of dislocation free, notch free silicon specimens, produced using photolithography. These silicon beams were tested in bending at various elevated temperatures and strain-rates and reveal a strain-rate dependent brittle-to-ductile transition in these defect-free samples. Dislocation etch-pit analysis of the sample surfaces show that massive dislocation activity occurs prior to yielding in the highest stressed portions of the beam. In addition, the scanning electron microscope images reveal a qualitative increase in dislocation activity above the BDTT, in agreement with the observed upper yield point behavior. The Khantha-Pope-Vitek model of the BDT is proposed to address these new observations.

9:05 AM Invited

Effects of Segregation in Cu and Ni3Al upon Impact Fracture: An Ultra-High Vacuum Study with Local Probe Scanning Auger/Scanning Electron Microscopy: *Jeff Th. De Hosson*[1]; D. van Agterveld[1]; G. Palasantzas[1]; [1]University of Groningen, Dept. of Appl. Phys., Nijenborgh 4, Groningen 9747 AG The Netherlands

This contribution deals with applications of UHV-local probe scanning Auger/scanning electron microscopy to the field of segregation. Results will be discussed of two different systems: Cu-S and Ni3Al-B materials. In-situ fracture of copper-alloys containing copper-sulfide precipitates exhibits areas in the form of pits. The wide variety of morphologies depends significantly on the size of the existing precipitate. As a second example B-doped polycrystalline Ni3Al (~0.1-0.5 % at. B) is chosen. In particular this contribution concentrates on phenomena of the electron beam induced oxidation of surfaces of Ni3Al-base alloys. It appears that an electron beam may contribute substantially to the oxidation behavior of polycrystalline and Ni3Al-B surfaces during Auger analysis at room temperature. Therefore special care should be taken in Boron segregation studies onto Ni3Al-B surfaces/interfaces with Auger microscopy because of irreversibly structural changes associated with an intense Ni-oxide formation by the electron beam.

9:40 AM

Segregation in PdO/Pd Alloy Systems: Matthew Augustine[1]; Heng Zhang[1]; *Harris L. Marcus*[1]; [1]University of Connecticut, Inst. of Matls. Sci., Storrs, CT 06269-3136 USA

PdO is a major catalyst for low temperature combustion of natural gas. This paper will describe research efforts on the nature of surface segregation of alloying elements in Pd and PdO and relate them to the catalytic behavior. The high temperature surface analysis was performed in-situ in both a scanning Auger spectrometer and an XPS system.

10:00 AM

Effect of Solutes on Grain Boundary Sliding in Aluminum: *J. S. Vetrano*[1]; C. H. Henager[1]; R. J. Kurtz[1]; R. G. Hoagland[1]; V. Gertsman[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA

We have used mechanical testing, microstructural evaluation and computational studies to elucidate the role of magnesium and tin on grain boundary dislocations (GBD) and grain boundary sliding (GBS) in aluminum. Whereas in some instances solute segregation can be detrimental to properties, enhanced sliding caused by segregation can be beneficial to forming operations such as superplastic deformation. Mechanical testing has revealed that magnesium segregates to grain boundaries during sliding and tin can either enhance or reduce the superplastic elongation. Microstructural studies in the transmission electron microscope have shown that tin resides along grain boundaries and particularly triple points so its effect is localized. Further studies are being carried out to further understand how tin is affecting the deformation. Computational studies on an aluminum symmetric tilt grain boundary performed using embedded atom method techniques showed that sliding resistance increased with increasing concentration of magnesium. Atomistic studies also revealed that magnesium segregation inhibited core spreading of GBDs thereby decreasing their mobility. These studies are working to better elucidate how solutes effect the structure and properties of grain boundaries. Work supported by the Materials Science Division, Office of Basic Energy Sciences, US Department of Energy under contract DE-AC06-76RLO-1830.

10:20 AM

Surface Segregation in an Al-4.2At%Ag Alloy: *Robert W. Hyland*[1]; H. K. Lee[2]; H. I. Aaronson[3]; P. P. Wynblatt[3]; [1]KB Alloys, Inc., R&D/Tech., 220 Old W. Penn Ave., Box 53, Robesonia, PA 19551 USA; [2]International Business Machines Corporation, Gen. Tech. Div., Essex Junction, VT 05452 USA; [3]Carnegie Mellon University, MSE, Pittsburgh, PA 15213 USA

The equilibrium surface composition of a polycrystalline Al-4.2 at%Ag alloy has been measured as a function of temperature using Auger electron spectroscopy. Results indicate that silver segregates to surfaces with an apparent enthalpy of segregation of about -38 kJ/mol. The equilibrium average surface composition of the alloy has been calculated as a function of surface orientation using the Lee-Aaronson discrete lattice plane model. A modified regular solution model originally due to Alexander et al. was used to represent the interactions between Al and Ag. While this method successfully described the bulk thermodynamic properties of Al-Ag, it predicted no surface segregation of Ag. Possible reasons for the disagreement are considered. Finally, the surface segregation of silver measured by AES was compared with that predicted by the Gibbs adsorption isotherm, wherein the latter calculation made use of results of ultra high vacuum measurements of the bulk composition dependence of the surface tension of α Al-Ag alloys. Qualitative agreement was found between the two experimental methods, both indicating enrichment of Ag at the free surfaces.

10:40 AM

The Effect of Impurities on Phase Transformation Kinetics: *Robert C. Pond*[1]; Steven Celotto[1]; [1]University of Liverpool, Dept. of Eng., Matls. Sci. & Eng., Brownlow Hill, Liverpool L69 3BX UK

The principal thrust of our recent work has been to establish a mechanistic model of martensitic transformation that is consistent with the phenomenological theory of martensite crystallography (PTMC). This has been achieved in terms of a mechanism involving transformation dislocations moving along the parent-martensite interface and lattice-invariant deformation (LID) generated in the martensite. Modelling the transformation process in this way gives a deeper insight into the central proposition of the PTMC, i.e. the existence of an invariant plane in the shape deformation. This approach demonstrates that very special crystallographic circumstances must prevail for a transformation process to be diffusionless. For example, diffusional accommodation will be required if an appropriate mode of LID is activated or when the transformation interface interacts with defects in the parent phase. As a consequence, the rate of transformation will be much slower. The aim of the present paper is to discuss the effect of impurities in transformation kinetics through their influence in the activated mode of LID and the motion of transformation dislocations.

11:00 AM

Segregation, Ordering Effects and Relaxations at Surfaces and Interfaces of the Cu-Au System: *Hariton Michael Polatoglou*[1]; Anthoula Maidou[1]; [1]Aristotle University of Thessaloniki, Phys. Dept., Gr-54006 Thessaloniki Greece

An N-body potential semi-empirical tight binding model in the second moment approximation is used to describe the atomic interactions of the Cu-Au system, that allows the study of dynamical properties of the ensemble. The parameters are determined from T=0K structural data and elastic properties of Cu, Au and Cu3Au. Monte Carlo simulations in the constant temperature, pressure and chemical potential difference method are used to determine thermodynamic properties of the Cu-Au system of bulk, surfaces and interfaces of the intermetallic compounds, and for stoichiometries close to the intermetallic compounds of the system, for temperatures below and above the order-disorder transition temperature. After realistically reproducing the thermodynamic properties of the bulk intermetallic compounds, we investigate the surfaces and interfaces studying segregation phenomena and the ordering processes. Findings show different behavior for different geometries, i.e. strains present at the interfaces may produce ordering phenomena even for temperatures above Tc.

11:20 AM

Neutron Irradiation-Induced Non-Equilibrium Intergranular Segregation: Segregation Capability vs. Kinetics: *Jun Kameda*[1]; Yutaka Nishiyama[2]; Tamara E. Bloomer[3]; [1]Iowa State University, Ames Lab., Ames, IA 50011 USA; [2]Japan Atomic Energy Research Institute, Tokai, Ibaraki 319 Japan; [3]US Nuclear Regulatory Commission, Washington, DC 20555 USA

Non-equilibrium intergranular segregation induced by neutron irradiation and post-irradiation annealing (PIA) has been studied in several model ferritic alloys doped with phosphorus, manganese and/or copper. Irradiation-induced phosphorus segregation became more prominent with decreasing bulk phosphorus content. A PIA study has shown that the phosphorus segregation proceeds at lower PIA temperatures via mobile phosphorus-interstitial complexes while phosphorus desegregation dominates due to vacancy/phosphorus exchange during PIA at higher temperatures and/or for prolonged time. Correlation between the changes in segregated phosphorus and hardness, which is related to phosphorus rich precipitates, is presented. The mechanisms of non-equilibrium intergranular segregation during neutron irradiation are discussed in light of coupled fluxes of point defects and phosphorus, and the segregation capacity of grain boundaries to absorb to phosphorus. This work was supported by the USDOE. Office of Basic Energy Sciences, Division of Materials Sciences.

11:40 AM
Temperature and Stoichiometry Effects on Grain Boundary Structure and Cohesion in NiAl: Xuepeng Xie[1]; *Yuri Mishin*[1]; [1]George Mason University, Sch. of Computl. Scis., 4400 University Dr., MSN 5C3, Fairfax, VA 22030 USA

The equilibrium atomic structure of several symmetrical tilt grain boundaries in the B2-ordered intermetallic compound NiAl is studied using Metropolis Monte Carlo simulations and the embedded-atom method. At high temperatures, the grain boundaries tend to be enriched in Ni relative to the stoichiometric or Ni-rich lattice. In most cases the Ni segregation takes place by the substitution of Al atoms by Ni atoms without changing the boundary structure. In some boundaries, however, the Ni segregation alters the initial boundary structure (structural transformation). In Ni-rich alloys, the structure of some boundaries becomes virtually disordered. The impact of these effects on the reversible work of decohesion and cohesive strength of grain boundaries is evaluated.

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

Wednesday AM — Room: 401
February 20, 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: Wednesday 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

Wednesday AM — Room: 619-620
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mark Sammonds, UES Software, Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA

Keynote
Micro-Macro Modeling of Solidification Processes and Phenomena:*Vaughan Voller*[1]; [1]University of Minnesota, 500 Pillsbury Dr., Minneapolis, MN 55455 USA

The wide range of length and time scales found in solidification processes are outlined and discussed. Methods for Direct Microstructure Simulation (DMS) are introduced. Key features in sharp interface and phase field models are presented. Concepts in micro-macro solidification modeling are covered. Basic details of microstructure and segregation models are provided. A description of a recent segregation micro-macro model is presented in detail.

Computational Modeling of Materials, Minerals & Metals Processing: Track A - Heat & Mass Transfer - 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

Wednesday AM — Room: 619
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Thomas Battle, DuPont Company, White Pigment & Minl. Production, Edgemoor Plant, 104 Hay Rd., Edgemoor, DE 19809 USA; Rod Guthrie, McGill Metals Processing Center, 3610 University St., MH Wong Bldg., Rm. 2MO51, Montreal, Quebec, H3A 2B2 Canada

9:45 AM
A Steady State Electrothermic Simulation Analysis of a Carbothermic Reduction Reactor for the Production of Aluminium:*Dimitrios I. Gerogiorgis*[1]; B. Erik Ydstie[1]; Sridhar S. Seetharaman[2]; [1]Carnegie Mellon University, Dept. of Cheml. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Carbothermic reduction is an important process alternative for aluminium production, based on the endothermic chemical reduction reaction occurring between aluminium oxide and carbon. This process has potential for drastic reduction of fixed and operational costs of the investment. Furthermore, it is environmentally benign and in principle significantly more energy-efficient, as the costly electrolytic ionization is effectively avoided by direct chemical reduction. Carbon monoxide production can contribute to the energy integration of the process, as the installation of a conjugate power cycle allows for production of electric energy necessary for heating purposes. The carbothermic production of aluminium can occur in a two-stage high-temperature reactor. Carbon and aluminium oxide pellets are introduced into an electric arc furnace (1st stage) and undergo melting at a temperature of ca. 1950°C, thus forming a high-temperature molten slag. This multicomponent molten slag (carbon, aluminium oxide and carbide, carbon monoxide) is fed to a subsequent 2nd stage, where it reacts at a higher temperature (2050°C) to aluminium. The 2nd stage reaction products (carbon monoxide, molten pure aluminium) are then separated. The present study focuses on a Computational Fluid Dynamics (CFD) simulation of the flowfield within the carbothermic reduction reactor, which is an essential step for the effective conceptual design of pilot and production plants. This CFD simulation of the melt flow encompasses important research challenges, due to the limited knowledge of thermophysical properties for high-temperature multicomponent molten slags and the significant experimental limitations. The computational domain for the CFD simulation is defined as a rectangular sector, while the computational expense of the simulation can be minimized by considering symmetry planes. The CFD model for computation adopts simplifying assumptions for production (Joule effect) and consumption (endothermic reaction) of heat, considering a pseudohomogeneous molten slag. The CFD simulation is performed in a hexahedral structured grid (selecting uniform meshing) using a commercially available CFD code (CFX 4.3-AEA) with some necessary modifications. The main objective of the present CFD simulation study is to extract conclusions regarding the design of a carbothermic reactor and also understand how process parameters affect this system.

10:10 AM
The Limitations of CFD Modelling for Furnace Atmosphere Troubleshooting:*Paul F. Stratton*[1]; Neeraj Saxena[2]; M. Huggahalli[2]; [1]BOC Gases, European Dvlp. Ctr., Rother Valley Way, Holbrook, Sheffield, SY S20 3RP UK; [2]BOC Gases R&D, 100 Mountain Ave., Murray Hill, NJ USA

Furnace temperatures and atmospheres are the most important factors in an industrial heat treating processes. To create an atmosphere in a continuous furnace, several gases, either singly or as mixtures, are used. These include nitrogen, hydrogen, exothermic and endothermic atmospheres, natural gas, propane and cracked methanol. Which gas or gases and how much of each should be used in the furnace is largely based on experience or on expensive trial and error experiments. BOC Gases has developed a simplified, easy-to-use PC based model to facilitate the understanding, troubleshooting and design of atmosphere injection into heat treating furnaces and to minimise wasteful experimentation. The model takes into account the role of oxygen, nitrogen, hydrogen, methane, carbon monoxide, carbon dioxide and water vapour in the heat treating process. Fundamental energy and mass conservation equations are solved to determine atmosphere profiles in a furnace for given compositions of gases injected. The reducing and carburising potentials of the gases, necessary to design any heat treating process, can be deduced using this model. The model can also be used to design and troubleshoot the atmosphere injection practice. Using model simulations, the critical process variables can be identified and the heat treating process optimised using a minimal number of experiments on the production floor. To validate the model industrial examples are examined. The results of this model were found to compare quite well with actual data obtained from production furnaces.

10:35 AM Break

10:50 AM
Simulation of Internal Oxidation:*Henrik Larsson*[1]; Martin Schwind[1]; John ≈gren[1]; [1]Royal Institute of Technology, Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

Internal oxidation is simulated in three dimensions using a combined random walk-cellular automata approach. Nucleation theory is used for the early stages of growth. Diffusion is modelled utilising discrete units, whereby a transformed Fick-Onsager law is solved by a simple stochastic method. Growth and dissolution of precipitates are treated by applying the local equilibrium hypothesis and the Gibbs-Thomson equation. Grain boundaries are included in the model; their effect on diffusion, nucleation and growth are taken into account.

11:15 AM
Dynamic Model for a Vapor Recovery in Carbo-Thermic Aluminum Process: *Vianey Garcia-Osorio*[1]; Tor Lindstad[2]; B. Erik Ydstie[1]; [1]Carnegie Mellon University, Cheml. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]SINTEF, Matls. Tech., Alfred Getz vei 2, Trondheim N-7465 Norway

As a result of the high temperatures present in a Carbothermic Aluminum Process the content of aluminum and aluminum sub oxide in the gases leaving this process is high. These gases must be recovered. For this purpose a Vapor Recovery Unit is introduced in the system. Inside this unit the aluminum compounds react in a series of heterogeneous non-catalytic reactions forming solid, liquid and gas products. The different product phases depend on the temperature present in the system. Therefore the model needs to capture the physics of the system and accurately represents the interaction between the energy and material balances and process thermodynamics. In this work a dynamic model of the Vapor Recovery Unit is presented. Because of the complexity of the system several models are developed and coupled to capture the essential physics of the multiphase reaction problem. A shrinking core model is used to describe the reaction mechanisms. Then material and energy balances are coupled with the thermodynamic properties from the FACT database of physical properties, resulting in a system of nonlinear Partial Differential Equations. In the paper we describe the model development and governing equations for the reaction mechanisms and fluid flow. Finally we review the numerical schemes created for the solution of the model system. The model can be used for control and design purposes.

11:40 AM
Computer Simulation of the Structure-Energetical Transformations at Combustion Synthesis in the Systems NiAl and TiAl: *Mikhail D. Starostenkov*[1]; Gennadiy M. Poletayev[1]; Alexandra S. Starostenkova[1]; [1]Altai State Technical University, Gen. Phys. Dept., 46 Lenina Pr., Barnaul, Altai Region 656099 Russia

It is known, that combustion synthesis (CS) takes place at high velocities. That is why it is difficult to determine the stages of structure-energetical transformations, taking place at CS. The process of CS is modelled by the computer simulation, using molecular dynamics method. The investigating systems are presented in a form of bimetal thin films at the beginning of the process. The interactions between the atoms are given by the sets of emperic pair potentials. The set of critical parameters, regulating the velocity of the reaction of CS is found in the result of computer experiment. The parameters are the following: limited free volume, its connection with the temperature of the process beginning, the velocity of components mixing, the velocity of formation of intermetallic phases. It is established, that the structure transformations of the system take place at the formation of nanocrystal phases in the process of CS. Character of structure-energetical transformation at combustion synthesis in the systems NiAl and TiAl depend from the orientation of contacting plains in bimetal thin films.

12:05 AM
Influence of Surface Pressure and Slag Layer on Bubble Bursting in Degasser Systems: *Julie Cranga*[1]; Pascal Gardin[1]; Didier Huin[1]; Jacques Magnaudet[2]; [1]IRSID, THEMEF, Voie Romaine, BP30320, Maizieres les Metz 57283 France; [2]IMFT, Groupe Interface, Allee du Professeur Camille Soula, Toulouse 31400 France

For the control of steel grade in steelmaking reactors, IRSID and IMFT are building a new modelling of bubble behavior in the vicinity of liquid steel/slag interface. The objective is to identify the main parameters affecting hydrodynamics of the different phases and to supply the mass transfer models commonly used at IRSID with the appropriate fluid mechanic parameters (local velocities, bubble residence time in the melt ...). Special emphasis is given to the study of pressure influence on bubble bursting. The topics of the paper are: *First to describe the numerical VOF method developed to predict the multiphase flow including bubbles, liquid metal and slag layer. *Second to analyze the influence of pressure in RH degasser on bubble bursting. Validation is given by comparison with previous results in literature. The major point concerns the distinct behavior whether the pressure is low or high. *Third to study the influence of slag layer on liquid metal entrainment in the wake of the bubble and on the nature of projection from the bath surface, in relation with the slag layer thickness and physical properties (viscosity and interfacial tension).

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

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

Wednesday AM	Room: 620
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Mark Samonds, UES Software Inc., 175 Admiral Cochran Dr., Annapolis, MD 21401 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

9:45 AM
Computational Studies of the Control of Convection in Diamagnetic Liquids during Solidification with Magnetic Field Gradient: *C. B. Seybert*[1]; J. W. Evans[1]; [1]University of California, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA

Natural convection is a commonplace phenomenon in the solidification of materials. Driven by temperature gradients, concentration gradients or both, it plays a role in the development of macrosegregation and the microstructure of the solid. The paper describes an investigation, both by experiment and by mathematical modeling, into this natural convection and whether it can be controlled by the application of a magnetic body force. This force is not the Lorenz force, that can be used to damp convection in conducting fluids, but the magnetic body force that arises from a gradient in the magnetic field. If that gradient is of the right size and direction it can be made to eliminate buoyancy or even to levitate a fluid. The experimental investigation has entailed the measurement of velocities and solidification front positions in transparent liquids, with PIV used for the former. The measurements have been carried out for water and aqueous solutions of manganese chloride. In the latter case most of the measurements have been made with the solution in the bore of a superconducting magnet at Marshall Space Flight Center and it has been demonstrated that natural convection in this paramagnetic solution can be greatly reduced, perhaps halted, by the magnetic field. Measurements agree reasonably well with computations performed using FLUENT and this CFD package has been used to predict the conditions under which natural convection within a diamagnetic fluid might be halted.

10:10 AM
Methods for Approximating Discontinuous or Rapidly Changing Conductivity in Numerical Calculations: *Vaughan Richard Voller*[1]; [1]University of Minnesota, Civ. Eng., 500 Pillsbury Dr. S.E., Minneapolis, MN 55455 USA

Much is said in the recent literature on the modeling necessity of obtaining appropriate thermo-physical properties; in particular the

WEDNESDAY AM

requirement to account for properties that are ìstrongî functions of the depended variables, e.g., temperature. Of course obtaining appropriate thermo-physical properties serves no purpose unless they can be accurately integrated into a discrete numerical calculation. The purpose of this paper is to examine how temperature dependent conductivities that exhibit discontinuities or rapid changes can be handled in a numerical code. A simple approach based on a local application of a Kirchoff transformation at the discrete level is presented. The application of this transformationñthat does not require any inversionñis demonstrated on solving a range of one and two dimensional test problems.

10:35 AM Break

10:50 AM
Modelling of the De-Waxing of Investment Cast Shells: Jean-Christophe Gebelin[1]; Sam Jones[1]; *Mark R. Jolly*[1]; [1]The University of Birmingham, IRC in Matls., Edgbaston, Birmingham, W. Midlands B15 2TT UK

The de-waxing process in investment casting is truly a black art which is carried out in a black holeñthe autoclave. The process involves exposing wax, coated by a porous ceramic shell, to steam at a pressure of 10 bar and a temperature of about 180°C. The process which then occur are truly multi-media and multi-physics. Steam condenses in the porous ceramic, wax melts and flows, the shells expand and sometimes crack. Why the shells crack is one of the most difficult questions asked in the industry. Modelling and validating the de-waxing process is not simple. The IRC at the University of Birmingham is attempting to model this process using Physica+ a code developed in the UK. This paper will present the latest results in the attempt to model one of the casting industries most intractable problems.

11:15 AM
The Swirling Effect in an Immersion Nozzle on the Flow in a Continuous Casting Mold: *Shinichiro Yokoya*[1]; Sigeo Takagi[1]; Manubu Iguchi[1]; Katsukiyo Marukawa[3]; Shigeta Hara[2]; [1]Nippon Institute of Technology, Mechl. Eng., 4-1 Miyashiro, Minami-saitama, Saitama 345-8501 Japan; [2]Osaka University, Dept. of Matls. Sci. & Proc., Yamadaoka, Suita, Osakafu 565-0000, Japan; [3]Osaka University, Sumitomo Metal Industries, Ltd., Japan

A numerical analysis and water model study of the mold region of a continuous casting apparatus are performed with a novel injection concept using swirling flow in the pouring tube, to control the heat and mass transfer in the continuous casting mold. As a result, the following results were found: (1)By changing swirl strength, it is easy to control the flow pattern as well as the direction of the flow. (2)Uniform velocity distribution can be obtained within a very short distance from the outlet of the nozzle. (3)Heat and mass transfer near the meniscus can be remarkably activated compared with a conventional straight type immersion nozzle without swirl. (4)Swirl helps the superheat in the melt dissipate. (5)Penetration depth of the nozzle outlet flow is decreased remarkably by the application of swirling. Those findings mentioned above are very useful to control the flow pattern in the continuous caster.

11:40 AM
Computational Modelling of Metals Extrusion and Forging Processes: *A. J. Williams*[1]; T. N. Croft[1]; M. Cross[1]; [1]University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., The Old Royal Naval College, 30 Park Row, London SE10 9LS UK

The computational modelling of extrusion and forging processes is now well established. In this work a novel approach is described which utilises finite volume methods on unstructured meshes. This technique can be used to solve simultaneously for fluid flow, heat transfer and non-linear solid mechanics and their interactions. The approach involves the solution of free surface non-Newtonian fluid flow equations in an Eulerian context to track the behaviour of the workpiece and its extrusion/forging, and the solution of the solid mechanics equations in the Lagrangian context to predict the deformation/stress behaviour of the die. Some preliminary examples of this approach will be discussed.

12:05 PM
The Role of Orifice Shape in the Detection of Inclusions in Liquid Metals: *Roderick I.L. Guthrie*[1]; Mei Li[1]; [1]McGill Metals Processing Centre, Canada

A mathematical model was developed to study the role of orifice shape in the detection of inclusions in LiMCA (Liquid Metal Cleanliness Analyzer) system. The fluid flow field within the ESZ was obtained by solving the Navier-Stokes equations. The trajectories of entrained particles were calculated using the equations for motion of particles. A numerical study of the effect of orifice shape on signal shape generated by inclusions passing through orifice showed that the signals in a parabolic orifice are gaussian in shape, while those in a cylindrical orifice are trapezoidal in shape. This signal shape difference needs to be taken into account when performing particle discrimination. Orifice shape influences also pass-through fraction of inclusions. Finally, a study of the orifice shape on critical conditioning currents suggested that as the polynominal coefficient of the parabolic orifice increases, the critical conditioning current, which is also the maximum operating current, decreases dramatically. These critical conditioning currents also increase as the fluid flow velocity within the ESZ increases.

Computational Phase Transformations: Phase Equilibria from First Principles

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

Wednesday AM — Room: 201
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Mark Asta, Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

8:30 AM
First-Principles Quantum Mechanical Predictions of Alloy Ground States and Microstructures: *Alex Zunger*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

There is a lot of talk of computer-aided discoveries/design of new materials, but the simple fact is that even limiting oneself to materials made of just two elements, (e.g., Cu-Au or Si-Ge), and to a substitutional system there can exist as many as 2^N configurations that include compounds, alloys, superlattices, and impurities. Indeed, even for a modest number of sites N, this is an astronomical number. Thus, the conventional energy minimization approach for selecting the most stable crystal structure for A_pB_q (e.g., first-principles pseudopotentials) is hopeless if one considers the full 2^N space configurations of A, B on a lattice of N points. Thus, contemporary energy minimization approaches use instead the method of ìrounding-up the usual suspectsî: selecting the most stable from only a small number of well-known candidate structures. The potential for missing new and important structures is obviously large. Even if we know the stable structure at T=0, we need to further know how it looks at finite-temperatures. This requires modeling short-range-order (SRO), precipitate morphology, etc. Computationally, this could need ìsupercellsî with 10^4-10^6 atoms. This talk outlines the approach to a solution. It addresses the questions of: (i) finding the lowest energy configuration of substitutional systems; (ii) calculating their composition-temperature phase diagram; and, (iii) their finite-temperature microstructure, including precipitate shapes and SRO, using the first-principles local density approximation (LDA). Mapping of the LDA energies of only 10-20 A_pB_q compounds onto an Ising-like ìcluster expansionî enables use of lattice statistical mechanics techniques that elegantly solve the above problems. This extends the utility of the LDA from simple, perfectly-ordered compounds to truly complex structures. I will illustrate the method for semiconductor systems (GaAs-AlAs, GaAs-InAs, InP-GaP) and transition-metal intermetallic systems (Cu-Pd, Cu-Au, Ni-Pt, Ag-Pd, Cu-Zn, Ni-V, Ag-Au, Rh-Pt, $Li_x\square_{1-x}CoO_2$, $Sc_{1-x}\square_xS$), showing how previously unsuspected structures and materials are predicted, and how one can predict precipitate morphology.

9:15 AM
Electronic Structure Calculations of Random Alloys: A Step Towards a Modeling and Understanding of Phase Equilibria: *Igor Anatolievich Abrikosov*[1]; [1]Uppsala University, Phys. Dept., ≈ngstr^mlaboratoriet, Box 530, Uppsala SE-75121 Sweden

A state-of-the-art approach to the theoretical simulations of the phase stabilities includes a combination of first-principles electronic structure calculations with methods of atomic-scale simulations and statistical mechanics. In doing so one can treat extremely complex systems and processes with very high level of accuracy and reliability. In this regards, an ability to describe the electronic structure of random alloys has become an essential step towards a modeling and understanding of phase equilibria. We discuss recent developments of the methodological base in this field. We formulate the electronic structure problem for systems with or without translational symmetry completely in terms of Greenís functions within a Linear Muffin-Tin Orbitals (LMTO) as well as within an Exact Muffin-Tin Orbitals (EMTO) basis sets. The calculations of the total energies, electronic structure, and effective interatomic interactions in different systems will be discussed, including fcc and hcp phases of Cu-Zn, random fcc Al-Zn alloys, two forms of cerium oxide, and magnetic semiconductors. The whole scheme of this multiscale modeling is illustrated by calculations of surface segregations in fcc Ni-Pt alloy.

9:45 AM
Ab Initio Study of One-Dimensional Long-Period Structures in Al3Ti, Cu3Pd, and Au3Zn Compounds: *Catherine Colinet*[1]; Alain Pasturel[2]; [1]LTPCM/ENSEEG, BP 75, Saint Martin díHeres 38402 France; [2]Laboratoire de Physique numerique des SystËmes Complexes, Maison des Magisteres CNRS, BP 166, Grenoble, Cedex 38042 France

In a number of ordered face-centred cubic A3B compounds one-dimensional long-period structures (1D-LPSís) are observed experimentally. These superstructures are lattices based on L12 structure and may be viewed as periodic arrangements of (001) antiphase boundaries. We have performed ab initio calculations of the stability of 1D-LPSís in Al3Ti, Cu3Pd, and Au3Zn employing the Vienna ab initio simulation package (VASP). In each system the energetic effects due to the tetragonal distortion and of the cell-internal displacements of the atoms in the 1D-LPSís are studied. The effects are very important in the case of Al3Ti compound, and very small in the cases of Cu3Pd and Au3Zn compounds. In the Al3Ti, 1D-LPS of the types <21j> and <2j1> have energies which are very near those of D023 and D022. In Cu3Pd, <3>, <4>, and <43> 1D-LPS have similar energies of formation and are the more stable 1D-LPSís at T=0K. In Au3Zn, D023 structure is the ground state at T=0K, however <32j> 1D-LPSís have energies which are very near. The energetic results are discussed in the framework of the axial-next-nearest-neighbor using (ANNNI) model. In each system the antiphase boundary energy is calculated.

10:15 AM Break

10:30 AM
Incorporating Vibrational Entropies in First-Principles Calculations of Phase Equilibria: *Vidvuds Ozolins*[1]; [1]Sandia National Laboratories, Thin Film & Interface Sci., PO Box 969, MS 9161, Livermore, CA 94551-0969 USA

Recently, it has been demonstrated that the accuracy of first-principles calculated phase diagrams can be dramatically improved by including vibrational contributions to the free energies of alloy phases. For instance, vibrational entropy increases the solid solubility of Sc in FCC Al matrix by a factor of 27, bringing the calculated and experimentally measured solvus boundaries in excellent agreement [see V. Ozolins and M. Asta, Phys. Rev. Lett 86, 448 (2001)]. We find similar strikingly large effects in other Al-based alloys (such as Al-Au, Al-Cu, Al-Mg, Al-Ni, Al-Ti, and Al-Zr). These surprising results have added urgency to the development of new methods for incorporating vibrational entropies in first-principles calculations of alloy phase diagrams. We will discuss the prospects for accomplishing this goal in the framework of a generalized cluster expansion in terms of alloy configuration variables and ionic displacements. Using first-principles calculated vibrational spectra and interatomic force constants, we analyze in detail the configuration dependence of interatomic bond lengths and associated bond stiffness constants in the aforementioned Al-based systems. In particular, we stress the importance of including higher-order anharmonic terms in displacement variables. We also present a comparison with existing cluster expansion methods and discuss implications for treating elastic inhomogeneities and strain energies in coherent microstructures.

11:00 AM
First-Principles Thermodynamic Theory of Ordering in Multicomponent Alloys: *Duane D. Johnson*[1]; [1]University of Illnois, Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1302 W. Green St., Urbana, IL 61801 USA

A first-principles thermodynamic theory of atomic (and magnetic) short-range order for multicomponent alloys is presented. The alloyís ordering is based on the underlying electronic-structure of the high-temperature solid solution phase (using coherent-potential approximation and multiple-scattering theory) so its electronic origin can be determined. In this linear-response method, multicomponent alloys cost no additional computational time over binaries and all possible orderings on a given lattice can be determined in one calculation, similar to phonon modes. Examples in binary and ternary alloys (e.g., fcc Ni3V, bcc Ti-Al-Nb and Cu2AuZn) will be given, in some cases with comparison to results of structural inversion method or direct calculations of energetics in partially ordered alloys.

11:30 AM
Automating First-Principles Phase Diagram Calculations: *Axel van de Walle*[1]; Gerd Ceder[2]; Mark Asta[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Rm. 13-5056, 77 Massachusetts Ave., Cambridge, MA 02139 USA

The determination of phase diagrams from first principles is among the most important steps required to build a ìvirtual laboratoryî where materials could be designed from first-principles, without relying on experimental input. The thermodynamic stability (or metastability) of a new material is important to assess, before spending a considerable amount of time to devise a way to synthesize it. Although the formalism that allows the calculation of solid state phase diagrams from first principles is well established, its practical implementation remains a tedious process. To solve this problem, we have created a software package which automates the process of performing such calculations. This talk will first describe the overall organization of the package, emphasizing its modular design. The algorithm underlying the automated construction of the so-called cluster expansion will be briefly discussed. Special attention will then be given to the methods enabling the automatic computation of various thermodynamic quantities through Monte Carlo simulations. Finally, various detailed examples of application will illustrate the packageís ease of use.

Creep Deformation: Fundamentals and Applications: Aluminum Alloys

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

Wednesday AM	Room: 214
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: W. Blum, University of Erlangen-Nuernberg, Inst. f. Werkstoffwissenschaften LS1, Martensstr. 5, 91058 Erlangen Germany

8:30 AM Keynote
Creep and Creep Fracture of Aluminium Alloys for Supersonic Civil Aircraft: *B. Wilshire*[1]; H. Burt[1]; [1]University of Wales Swansea, Dept. of Matls. Eng., Singleton Park, Swansea SA2 8PP UK

The low specific gravity of conventional aluminium alloys (~2.8 Mg m^{-3}) has ensured their widespread selection for airframe applications, but the low melting point of aluminium (Tm ≅ 933K) means that their creep properties must be considered when exposure temperatures approach $0.4T_m$. Such temperatures can be reached not only with parts deliberately heated for de-icing and cabin heating or components near the engines but also for critical areas of supersonic aircraft subject to kinetic heating. Thus, with Concorde, the critical temperatures must not exceed about 400K, limiting the speed to Mach 2.2. Major research programmes have therefore been launched, seeking to identify materials which may prove superior to the current Concorde alloy, Hiduminium RR58, an alloy containing approximately 2.5%Cu-1.5% Mg-0.2% Si-1.0% Fe-1.0% Ni (wt%). In particular, at Swansea, creep and creep fracture studies have been carried out for three conventional alloys having the following nominal compositions: 2124-T851, ie Al-4.4% Cu-1.5% Mg-0.6% Mn, 2419-T851, ie Al-6.3% Cu-0.15% Zn-0.3% Mn, and 7010-T7351, ie Al-6% Zn-2.4% Mg-1.7% Cu-0.3% Zr. In short-term tests at 423K, 2124-T851 displays the greatest resistance to creep and creep fracture. However, comparable properties are

WEDNESDAY AM

exhibited by 8090-T8771 (i.e. Al-1.3% Cu-1% Mg-0.1% Zr-2.5% Li), an interesting result because this aluminium-lithium alloy has a specific gravity (2.53 Mg m^{-3}) which would offer airframe weight savings approaching those expected with fibre-reinforced polymer composites. Yet, while short-term data provides information for performance ranking, the service lives of future high-speed civil transport will be 100,000 hours or more. For this reason, long-term stress-rupture property comparisons have also been undertaken to assess the creep and creep rupture strengths of 2124-T851 and 2419-T851 in relation to the property values reported for Hiduminium RR58 and the equivalent alloy, 2618.

9:00 AM

Creepal Thematic Network: Achievements and Future Challenges: *J. C. Ehrstrˆm*[1]; Y. Girard[2]; [1]Pechiney CRV, BP 27, 38341 Voreppe, Cedex France; [2]EADS CCR, 12 rue Pasteur, BP76, 92152 Suresnes, Cedex France

The CREEPAL EC funded thematic network worked between 1999 and 2001 to assess the present knowledge on the behaviour of aluminium alloys in creep conditions. The applications of aluminium alloys at high temperature include supersonic aircraft, car engines, turbines, conductors. Wrought 2000 and 6000 alloys and casting alloys (Al-Si-Mg-(Cu)) are particularly suited for these applications. The practical aspects of characterising small deformation creep (0.1% in 1000 h) were addressed via a round robin between 7 laboratories. The test conditions were compared thoroughly and the results statistically analysed. Good practices are defined which ensure consistency of the results between labs. The intrinsic scatter of the repeated tests in the same labs and conditions appears to be high compared with a CONCORDE type specification. Beside monotonic constant load creep, the practical components are submitted to complex loading sequences which result in damages due to fatigue and creep as well as thermal fatigue. These aspects are particularly important in car engines for instance. The damage tolerance of future supersonic aircraft structures i.e. resistance to sustained load cracking, fatigue cracking and residual strength, must be addressed taking into account the complex loading. New sheet material for high temperature application (AA2650) have been developed in the last decade. However, a better understanding of the micro-structural features controlling the behaviour of aluminium alloys at high temperature must be achieved in order to improve the performance of materials in real components. Several experts contributed to a state of the art on this subject. Strain induced ageing, glide types, effect of the different type of precipitates and substructure were addressed. Finally, the long term prediction, i.e. the extrapolation of data obtained with tests of limited duration to the real life time of the structures is a major issue in high temperature application. Empirical approaches as well as physically based approached were evaluated. Among metals, aluminium alloys are not often used for high temperature applications. They offer however unique compromises in the range 100∞C-300∞C thanks to their thermal conductivity, strength, forming and damage tolerance. The need to continue the investigations for a better performance of highly technological products appears clearly. The approach must include the knowledge gained in the development of other metals.

9:20 AM

Tertiary Creep in a Precipitation Hardened Al-Alloy Due to Dynamic Transformation of Precipitates: *Philip Eisenlohr*[1]; Wolfgang Blum[1]; Massimo De Sanctis[2]; [1]University of Erlangen-Nuernberg, Inst. f. Werkstoffwissenschaften LS1, Martensstr. 5, Erlangen 91058 Germany; [2]University of Pisa, Dipartimento di Ingegneria Chimica, Chimica Industriale e Scienza dei Materiali, Via Diotisalvi 2, Pisa 56126 Italy

The present work was performed within the European CREEPAL thematic network in order to check the microstructural origin of tertiary creep in Al-alloys hardened by metastable, (semi-) coherent precipitates. An AlSiCuZnMg-alloy was subjected to creep at 150∞C. A minimum of creep rate followed by tertiary (accelerating) creep was found in compression. Transmission electron microscopy confirmed the aging of the precipitate structure by coarsening and transformation to the stable precipitate phase. The aging causes loss of precipitation hardening which is responsible for the onset of tertiary creep in compression. The localization of strain during creep indicates that the dislocation activity during creep enhances the aging process.

9:40 AM

Primary Creep in Precipitation Hardened Aluminium Alloy RR58: J. T. Evans[2]; *J. S. Robinson*[1]; R. L. Cudd[3]; [1]University of Limerick, Dept. of Matls. Sci. & Tech., Ireland; [2]University of Newcastle upon Tyne, Dept. of Mechl., Matls. & Mfg. Eng.; [3]Formerly of HDA Forgings, Ltd., Redditch, Worcestershire

The CEC funded Thematic Network CREEPAL (BRRT-CT98-5101) was established in 1998 to collect and disseminate information on the long-term creep and thermal mechanical cycling behaviour of aluminium alloys. Through CREEPAL, the opportunity has arisen to analyse a large experimental data set for the alloy Hiduminium RR58 (2618A) provided by HDA Forgings Ltd. (UK). Analysis of creep curves resulting from tests performed at 100∞C indicates a substantial primary creep region. A continuum model of inhomogeneous deformation is presented in which the alloy is considered to consist of a matrix containing domains with a lower creep resistance than the matrix. It is shown that the model could account for the primary creep exhibited by the precipitation-hardened aluminium alloy RR58 at 100∞C. A feature of the deformation in the model is the build up of internal stress, which will drive reverse deformation when the material is unloaded.

10:00 AM Invited

Precipitation Strengthening of Heat-Treatable Al(Sc) Alloys at Elevated Temperatures: *Emmanuelle A. Marquis*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., MLSB 2036, 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

To understand the precipitation-strengthening parameters controlling optimal yield strength and creep resistance at elevated temperature, we studied the effects of precipitate size and volume fraction upon the yield and creep strengths of dilute Al(Sc) alloys in the temperature range of 225 to 300∞C. Coherent, spheroidal Al_3Sc precipitates in alloys with 0.1, 0.2 and 0.3 wt.% Sc (corresponding to precipitate volume fractions in the range of 0.23 to 0.68%) were grown to sizes in the range 1.4 to 10 nm through different heat-treatments. Creep threshold stresses were found to be about ten times lower than the yield stresses at 300∞C, indicative of a climb-controlled bypass mechanism, which is modeled according to existing theory. Transmission electron microscopy observations of deformed samples illustrate the interactions between dislocations and precipitates.

10:20 AM Break

10:30 AM Invited

Modelling Creep Ductilities and Lifetimes in a Commercial Aluminium Alloy: *Brian Dyson*[1]; [1]Imperial College of Science Technology & Medicine, Dept. of Matls. Prince Consort Rd., London SW7 2BP England

In a recent paper (1), Wilshire and Burt comprehensively analysed the tensile creep and fracture behaviour of the commercial aluminium alloy 2124-T851. They also showed that the accuracy of predicting service lifetimes using the θ methodology was seriously compromised because of uncertainties over expected creep ductilities, ε_f, at service stress levels. This led them to conclude that ìa predictive knowledge of the factors determining ε_f remains a key requirement for low-ductility materialsî. The present paper attempts to provide a quantitative explanation. There seems little doubt that the creep fracture path in this (and similar aluminium alloys) is intergranular and that the detailed mechanism is dominated by the kinetics of intergranular cavitation. The paper revisits earlier work on creep-constrained cavitation and extends a previous analysis to obtain a numerical method which allows prediction of isothermal creep ductility during the stress range when the constrain t condition is gradually being relaxed. Applying the analysis to the 2124-T851 alloy reveals a simplifying if surprising feature: the rapidly increasing ductility with stress at each of the three temperatures analysed by Wilshire and Burt (1) and giving rise to their extrapolative problemsñis quantitatively consistent with the transition from constrained to unconstrained growth of an array of cavities having a lower-bound ductility of 1.5%, independent of temperature. It thus provides an answer to the problem posed by Wilshire and Burt. Furthermore, it is shown that when this extended ductility model is incorporated within a hyperbolic sine law kinetic model of creep, it predicts the lifetimes and minimum creep rate data reported by Wilshire and Burt and also confirms their view that the very high and variable ìnî values reported in these alloys (from 8-40) is not a consequence of any change of creep mechanism. 1. B. Wilshire and H. Burt, (2001) in Creep & Fracture of Engineering Materials & Structures, edited by J D Parker, The Institute of Materials, London, 261-268.

10:55 AM

Influence of Sub-Structure and Sub-Structure Development on the Creep of Aluminum Alloys: *Donald R. Lesuer*[1]; Rustam Kaibyshev[2]; [1]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA; [2]Institute for Metals Superplasticity Problems, 39 Khalturina St., Ufa Russia

It is well know that subgrains have a pronounced effect on the elevated temperature plastic flow behavior of coarse-grain, polycrystalline aluminum and aluminum alloys. In general, if a sub-structure develops, then the strain rate at a given applied stress varies with subgrain size (l) as l3. However, the development and stability of substructure is a function of microstructure (especially dispersoid content), temperature and stress (subgrain size varies inversely with stress). In this paper, the creep behavior of a 2219 aluminum alloy is examined relative to the development and stability of its substructure. The alloy was processed to develop a microstructure containing nano-scale Al3Cr and Al3Zr dispersoids and the creep response was evaluated in the temperature range from 250-500∞C. In the high stress/low temperature region, the creep data shows a stress exponent of 7 and the formation of weak cell boundaries, whereas in the low stress/high temperature region, a stress exponent of 5 and subgrains are obse rved. The creep behavior and substructure development in this alloy will be compared to the behavior of pure aluminum and other dispersion-containing aluminum alloys. Work performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.

11:15 AM
Deformation Behavior of a Modified 5083 Aluminum Alloy: *Rustam Kaibyshev*[1]; Oleg Sitdikov[1]; Fanil Musin[1]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

The deformation behavior of a 0.2% Zr and 1.6% Mn modified 5083 aluminum alloy was studied in the temperature range 250-570°C over five orders of magnitude of strain rate. It was shown that the modified 5083 Al exhibits a threshold behavior. Analysis in terms of threshold stress showed that there are three different classes of deformation behavior in the power-law creep regime which correspond with three different temperature dependencies of normalized threshold stress, distinguished by value of the energy term, Qo. At the lower values of normalized strain rates, kT/(D1Eb)<10-13, the true stress exponent, n, is close to 3 and the true activation energy, Qc, is about 130±5 kJ/mol. At normalized strain rates, kT/(D1Eb), ranging from 10-13 to 10-8 the n value is 4 and the true activation energy, Qc, is equal to 140±8 kJ/mol. At higher values of kT/(DlEb) (>10-8), the true stress exponent is 6 and the true activation energy is about 120±8 kJ/mol. The values of Qo were found to be about 31, 17 and 94 kJ/mol in the temperature intervals 250-350°C, 350-525°C and 525-570°C, respectively. The disappearance of threshold stress takes place at temperatures higher than 5500°C at strain rates lower than 10-5 s-1. Deformation mechanisms operating in the 5083 Al and their relations with threshold stress are discussed.

11:35 AM
Deformation Behavior of an Al-Li Alloy: *Rustam Kaibyshev*[1]; *Fanil Musin*[1]; *Donald Lesuer*[2]; [1]Institute for Metals Superplasticity Problems of RAS, Khalturina 39, Ufa 450001 Russia; [2]Lawrence Livermore National Laboratory, L-342, PO Box 808, Livermore, CA 94551 USA

The deformation behavior of a 1463 aluminum alloy (Al-2.1%Li-3%Cu) strengthened by coherent Al3Sc dispersoids was studied in the temperature range 250-480°C. It was found that the 1463 alloy exhibits threshold creep behavior. Analysis in terms of threshold stress revealed that at normalized strain rates, kT/(D1Eb) <10-9, the n value is 3. At higher normalized strain rates the true stress exponent is 5. The true activation energy, Qc, tends to grow from 90 kJ/mol at T=250°C to 142 kJ/mol at T=450°C with increasing temperature. Two different temperature dependencies distinguished by value of an energy term, Qo, were found. In the temperature range 250-450°C Qo is equal to 55 kJ/mol, and in the temperature intervals 450-480°C, Qo is about 22 kJ/mol. An effect of Al3Sc dispersoids on deformation mechanisms is discussed.

Deformation and Stresses in Small Volumes: Length and Size Scaling in Deformation

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

Wednesday AM	Room: 303
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Neville R. Moody, Sandia National Laboratories, PO Box 969 MS 9404, Livermore, CA 94551-0969 USA

8:30 AM Invited
A Volume to Surface Area Concept for Thin Film Fracture: *William W. Gerberich*[1]; John Jungk[1]; Alex Volinsky[2]; [1]University of Minnesota, Cheml. Eng. & Matls. Sci., 545 S.E. Washington Ave., Minneapolis, MN 55455 USA; [2]Motorola, Inc., Mesa, AZ USA

Previous indentation studies at extremely small depths less than 100 nm have verified that the surface energy provides a pivotal role in the Indentation Size Effect (ISE). Based on the ratio of volume work to surface work it has been shown that the surface work is substantial for such indentations. This has led to a length scale defined as V/S. With this further defined for indentation into thin films, a connectivity between elastic-plastic fracture mechanics and this length scale provides a crack resistance criterion. A simplifying assumption is that the indentation contact radius is commensurate with an initial crack size nucleating at the film/substrate interface, as confirmed by focused ion beam sections. An R-curve analysis is then derived for incremental crack growth. It is proposed that the resistance, GR, is not only proportional to the thickness and yield strength squared as in nearly all analyses, but that the GR value also scales with the one-half power of the incremental crack growth. Application to the delamination of Cu-thin films from silicon substrates is demonstrated.

9:00 AM
Indentation Size Effect-Limits of Conventional Deformation Behaviour: *Andrew J. Bushby*[1]; Ian Spary[1]; Nigel M. Jennett[2]; [1]Queen Mary, University of London, Dept. of Matls., Mile End Rd., London E1 4NS UK; [2]National Physical Laboratory, Matls. Ctr., Teddington UK

The indentation size effect, in which the hardness increases as the size of the indentation contact reduces, is well known. In those experiments indentation stress-strain curves were generated using spherical indenters with radii ranging from a few micrometres to several hundred mircometres and these were compared to data from conventional compression tests. Indenters of radius greater than 100 micrometres produced a single indentation stress-strain curve independent of indenter radius with a power law hardening coefficient equivalent to that in the compression tests. However, for smaller indenters the indentation stress-strain curves appeared at progressively higher pressures for smaller radius indenters. In this paper we model those experiments using finite element analysis methods. By inputting the compression stress-strain data to the model indentation stress-strain curves for the macro size indenters are reproduced. However, the model shows no length scale dependence for any size of indenter. We show that by offsetting the compression stress-strain curve in the strain axis and inputting this data to the model, the indentation behaviour of the smaller radius indenters can be modelled. This suggests that the indentation size effect is a result of an increase in the initial yield behaviour of the material and so does not conform to conventional deformation behaviour. Relationships between the strain offset, indenter radius and the indentation response are presented.

9:20 AM
The Indentation Size Effect and Hardening Mechanisms in Salt Crystals: *J. Greg Swadener*[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA

For NaCl and LiF single crystals, indentation with a Berkovich indenter exhibits increased hardness for decreasing depth of penetration, which is know as the indentation size effect. The size effect in these crystals is similar to the effect observed in metals and can be accurately described by a dislocation mechanism model. In contrast to a Berkovich indenter, a spherical indenter does not give a size effect with depth, but can be used instead to determine the effect of work hardening. A combination of spherical and pyramidal indenters was used to separately determine the work-hardening behavior and a true material length scale. The increase in hardness due to work hardening and length scale effects was in agreement with a recently developed model based on geometrically necessary dislocations. In addition, specimens that had been hardened by radiation exposure were found to exhibit the same length scale effects as unexposed specimens. The radiation hardening produced a simple incremental increase in hardness, which was similar to the effect of work hardening.

9:40 AM
Localized Slip around Nanoindentations: Slip Steps and Pile Up: *Kevin A. Nibur*[1]; David F. Bahr[1]; [1]Washington State University, Mechl. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA

Localized slip behavior has been examined in a BCC titanium alloy as a function of crystal orientation and hydrogen concentration. Previous models have suggested hydrogen may alter the cross slip behavior of dislocations. This theory has been investigated using a combination of nanoindentation, atomic force microscopy (AFM) and orientation imaging microscopy (OIM). Nanoindentation allowed dislocation motion to be studied without being influenced by macroscopic features such as grain boundaries and precipitates. Slip steps and spacings were measured using AFM and further quantified using OIM to determine the specific slip system of each band and the number of slip planes between steps, ranging between 100 and 300 slip planes per step depending on crystal orientation and hydrogen concentration. The frequency and magnitude of cross slipping was then linked to hydrogen concentration. The versatility of OIM allows these tests to be performed on common engineering materials without being limited to single crystals. Ultimately, the comparison between macroscopic out of plane deformation via pile up and microscopic deformation in terms of slip steps will aid in developing local plasticity models from which macroscopic flow behavior can be extracted from nanoindentation.

10:00 AM Break

10:30 AM
The Effects of Microstructural Length-Scales on the Strength of Metallic Multilayered Thin Films: *Amit Misra*[1]; John P. Hirth[1]; Richard G. Hoagland[1]; Harriet Kung[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

The strengths of metallic multilayers, composed of alternating layers of soft metals, approach theoretical limits when the bilayer periods are on the order of a few nanometers. Nanoindentation experiments have been used to elucidate the effects of microstructural length scales on the strength of sputter deposited metallic multilayers such as Cu-Nb. The length scales considered are: (i) the bilayer period, in the range of 2 nanometers to a few micrometers (for equal Cu and Nb layer thicknesses), (ii) the in-plane grain size, coarsened by annealing for a constant bilayer period, and (iii) the Cu to Nb layer thickness ratio at a constant bilayer period. The dependence of strength on the microstructural length scales is modeled using dislocation theory. At the micrometer to the sub-micrometer length scales, the dislocation pile-up based Hall-Petch model is applicable. At lower length scales, the strength of multilayers is interpreted in terms of the motion of single dislocations as Orowan-type loops that initiate plastic deformation in the softer phase, and then transmit across the Cu/Nb interface overcoming the resistance from the interface dislocation arrays. It is shown that the strength enhancement at nanometer length scales is primarily due to the stress needed for dislocation transmission across the Cu/Nb interfaces. This research is funded by DOE-OBES.

10:50 AM
Hardness Anisotropy and Surface Deformation around Vickers Indents in Single Crystals: Reina Martinez[1]; Mehdi Hakik[1]; *Pedro D. Peralta*[1]; [1]Arizona State University, Dept. of Mechl. Eng., Main Campus, PO Box 876106, Tempe, AZ 85287-6106 USA

Micro- and nanoindentation of polycrystalline materials often results in indents completely contained within a single grain, where the anisotropy related to single crystal deformation can have a strong effect on the material behavior. In this work, single crystals of two semi-brittle materials (pure MoSi2 and Al-doped MoSi2) and a ductile metal (copper) were indented to sub-micron depths on the (001) plane using a Vickers indenter, with the diagonals of the pyramid aligned along either <110> or <100> directions. The surface deformation around these indents was characterized using Atomic Force Microscopy. The results indicate that sink-in and pile-up behavior depend strongly on the in-plane crystallographic orientation of the diagonals of the indent, even for the soft copper single crystal. These results are discussed in terms of the anisotropy of hardening and deformation expected in the single crystals studied and the differences and similarities with respect to isotropic behavior.

11:10 AM
Study on Lamellar PST-TiAl by Nanoindentations and AFM: *Minhua Zhao*[1]; *Scott Mao*[1]; Huiping Xu[2]; J.M.K. Wiezorek[2]; [1]University of Pittsburgh, Dept. of Mechl. Eng., 648 Benedum Hall, 3700 Oíhara St., Pittsburgh, PA 15261 USA; [2]University of Pittsburgh, Dept. of Matls. Sci. & Eng., 3700 O'Hara St., Pittsburgh, PA 15261 USA

PST(Polysynthetically twinned) Titanium Aluminide alloys have been receiving a lot of attention in high temperature applications due to their outstanding density specific properties. Since the lamellar size of PST-TiAl is in the micron or submicron scale, its mechanical properties cannot be accurately predicted by conventional plasticity theory. In this study, in-situ nano indentations were made by Hysitron nanoindentor, while topography of the sample was analyzed by Nanoscope III AFM. Size effect in PST-TiAl was illustrated by the theory of strain gradient plasticity.

Fatigue and Creep of Metal Matrix Composites: Fatigue of Metal Matrix Composites - I

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

Wednesday AM	Room: 206
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Nik Chawla, Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287 USA; John Lewandowski, Case Western Reserve University, Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA

8:30 AM Keynote
Fatigue of Particle and Whisker-Reinforced Metal-Matrix Composites: *Javier LLorca*[1]; [1]Polytechnic University of Madrid, Dept. of Matls. Sci., E. T. S. de Ingenieros de Caminos, Madrid 28040 Spain

The reinforcement of metallic alloys with ceramic particles or whiskers has generated a new family of composite materials. They have matured during the last twenty years, and are currently used in structural components subjected to cyclic loads. This was partially possible thanks to a large research effort aimed at characterizing their behavior in fatigue. The results of this activity constitute a fairly coherent body which relates the micromechanisms of cyclic deformation to the overall fatigue performance. They are presented in this review, which is divided in seven sections. After the introduction, the microstructural changes induced by the dispersion of the ceramic reinforcements are described. This is followed by two sections devoted to an analysis of the micromechanisms of cyclic deformation from the microstructural and mechanical viewpoints. The next two sections are focused on the origins of crack nucleation and the kinetics of crack propagation upon cyclic loads. The overall fatigue performance of these composites is examined in the last part, which emphasizes their advantages and limitations as compared to the unreinforced counterparts. The effects of the processing, thermomechanical treatments, microstructural features, environmental factors and loading conditions are included in each section to provide a comprehensive picture of the fatigue performance of these composites.

9:10 AM Invited
Fatigue Crack Growth in Composites: *Kuntimaddi Sadananda*[1]; Ronald L. Holtz[1]; Asuri K. Vasudevan[2]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375-5000 USA; [2]Office of Naval Research, 800 N. Quincy St., Arlington, VA 22217 USA

Crack growth behavior in composites is analyzed using the Unified Approach to Fatigue Crack Growth developed by the authors. This approach is based on the fact that fatigue requires two load parameters for unambiguous description. Normally the stress intensity range, *K and load Ratio, R are used but the effects of load ratio are accounted for crack closure arising from pre-mature contact of the mating surfaces. Our analysis indicates that crack closure is either absent or its contribution is very small to have an effect. It is further shown that for fatigue crack growth, *K and Kmax are the two governing parameters providing two crack tip driving forces required for crack growth. Hence there are two thresholds that must be overcome for a crack to grow. Using this approach, the crack growth behavior in composites is analyzed examining the physical significance of these two thresholds in the composites.

9:40 AM
Mechanical Behavior of Al2O3 Fiber-Pure Aluminum Matrix Composites: *R. Prakash Kolli*[1]; Chris Hoppel[2]; Jonathan Montgomery[2]; Robert Dooley[2]; [1]United States Military Academy, Dept. of Civil & Mechl. Eng., West Point, NY 10996 USA; [2]US Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5069 USA

The US Army is considering metal matrix composite (MMC) materials for ordnance applications such as lightweight projectiles and gun systems. Nextel 610 alumina fiber-reinforced pure aluminum, made by 3M Company, is a relatively low cost candidate material with a high specific strength. Tensile specimans with three different fiber orientations, 0^o, 0^o/90^o, and +45^o were tested in monotonic tensile loading and in tension-tension (R=0.1) fatigue. The stress-strain response and low cycle fatigue life were determined. The following mechanical and material properties were evaluated: ultimate tensile strength, modulus of elasticity in the longitudinal direction, and strain to failure. Preliminary evaluation of shear modulus, and Poissonís ratio was also conducted. Optical metallography and electron fractography was performed to investigate microstructural failure modes.

10:00 AM
Fracture and Fatigue Studies on Be-Al ìComposite-Likeî Materials: *Joel Larose*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Matls. Sci. & Eng., 10900 Euclid Ave., White 400, Cleveland, OH 44106 USA

Be-Al alloys are composed of two phases: a Be-rich phase, which has high modulus (e.g. 287 GPa) and is semi-brittle, and an Al-rich phase, which has relatively low modulus (e.g. 70 GPa) and is ductile. This presentation will focus on aspects of the fracture and fatigue crack growth behavior of Be-Al alloys processed via different means. The alloys contain approximately 70vol% Be and 30vol% Al and possess a ìCermet-likeî structure. The damage tolerance is investigated by means of fracture toughness tests (K_Q) and fatigue crack growth (da/dN vs. ΔK). Notched and fatigue precracked specimens were used to determine fracture toughness at different temperatures. The effects of changes in stress ratio (i.e. R) and test temperature on the fatigue crack growth behavior were also determined. Fracture surfaces and fracture paths were examined by both optical and scanning electron microscopy in order to document any changes in observed behavior. Research supported by ONR N00014-99-1-0237 with supply of materials by Brush Wellman, Inc.

10:20 AM Break

10:30 AM Invited
Cyclic Plasticity and Fatigue in Metal Matrix Composites: A Modeling Perspective: *Yu-Lin Shen*[1]; [1]University of New Mexico, Dept. of Mechl. Eng., Mechl. Eng. Bldg., Albuquerque, NM 87131 USA

Continuum-based numerical modeling has been extensively used in studying the thermomechanical behavior of composite materials. By endowing the matrix and reinforcement materials with appropriate constitutive properties, the global composite response and local field quantities can be obtained in a straightforward manner. This presentation is devoted to applying this methodology to cyclic deformation in discontinuously reinforced metal matrix composites. The basic hardening laws used for modeling metal plasticity and their effects on cyclic response are addressed. The disturbance of plastic flow path in the metal matrix, due to the presence of hard reinforcement, is illustrated. Non-uniform matrix plasticity gives rise to early composite yielding and the Bauschinger effect, which provides a mechanistic rationale of the experimental findings. The effects of reinforcement geometry and pre-existing damage on the accumulation of cyclic plasticity, which ultimately leads to fatigue failure, will also be presented. Finally, we will discuss the interaction between intermetallic inclusion particles and reinforcement particles, and its influences on the fatigue crack initiation of the composite.

11:00 AM
Effect of Particle Orientation Anisotropy on the Fatigue Behavior of Metal Matrix Composites: *Vasudevan V. Ganesh*[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

Preferred orientation of reinforcement particles is commonly observed in extruded particle reinforced metal matrix composites. Recent modeling work has shown that while the particles have a relatively small aspect ratio, the elastic modulus of the composite is affected by the direction of applied load with respect to the orientation of the particles. In this study, we have studied the effect of fatigue behavior of a 2080/SiC/10p-T6 composite oriented transverse and longitudinal to the extrusion axis. Stress controlled fatigue tests were carried out at a frequency of 40 Hz and an R-ratio of -1. Microstructural analysis of the fatigued specimens through transmission electron microscopy was conducted to obtain an understanding of the evolution of dislocation structures and interactions with precipitates in the Al matrix. Surface replication was also conducted to examine the role of particle orientation on fatigue crack growth in these materials. Research sponsored by the Office of Naval Research under contract# N00014-01-1-0694 (Program Manager: A.K. Vasudevan).

11:20 AM
Strain Evolution in an Aluminum-Alumina Metal Matrix Composite during Cyclic Loading and Fiber Failure: *Jay C. Hanan*[1]; Geoff A. Swift[1]; Ersan ‹st‚ndag[1]; Bjorn Clausen[1]; Mark M. Bourke[2]; [1]Caltech, Matls. Sci., 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., PO Box 1663, MS H805, Los Alamos, NM 87545 USA

The evolution of matrix strains, particularly after fiber failure is of significant interest in metal-matrix composites. A model composite consisting of a single alumina fiber quenched in an aluminum alloy matrix was engineered to fail at the fiber in the center of the gage section. Neutron and synchrotron X-ray diffraction were employed to observe deformations within the composite while statically loaded and unloaded in tension over several cycles. The fiber failed upon loading leaving the matrix intact. Both fiber and matrix strains were monitored during this process at various length scales. The diffraction results were compared to micromechanics models.

Fundamentals of Advanced Materials for Energy Conversion: Hydrogen & Tritium Storage

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

Wednesday AM	Room: 613
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: MichËle Gupta, UniversitÈ Paris-Sud, Institut Sciences des MatÈriaux, UMR8647 B‚t. 415, Orsay 91405 France; Robert H. Hsu, Westinghouse Savannah River Company, Aiken, SC USA; Dhanesh Chandra, University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

8:30 AM Plenary
Overview of Metal Hydride Technology for Applications in Transportation and Space: *Robert C. Bowman*[1]; [1]Jet Propulsion Laboratory, MS 79-24, 4800 Oak Grove Dr., Pasadena, CA 91109 USA

After a brief survey of the various classes of the metal alloys and compounds that are suitable for hydrogen storage and energy conversion applications, a comparison will be made of their relevant critical properties. Subjects to be covered include hydrogen absorption and desorption pressure, total and reversible hydrogen storage capacity, reaction rate kinetics, initial activation requirements, susceptibility to contamination, and durability during long term thermal or electrochemical cycling. Applications to be considered are hydrogen storage as fuel, electrodes in batteries, gas separation and purification, thermal switches, and sorption cryocoolers. Past and future uses of metal hydrides on space flights will be described.

9:10 AM Plenary
Applications of Metal Hydride Technology in Tritium Processing Systems: *Joseph R. Wermer*[1]; [1]Los Alamos National Laboratory, Tritium Sci. & Eng. Grp., MS C348, Los Alamos, NM 87544 USA

Metal hydride technology is in many ways ideally suited for tritium processing plants. Metal hydride-based tritium processing systems have greatly increased the efficiency and safety of tritium processing operations. While older facilities relied on tanks to store tritium gas and mechanical pumps to store and transfer tritium, newer facilities employ metal hydride technology to store and pump tritium. Hydrogen isotope separation systems have also been developed which take advantage of the large isotope effect of certain metal hydrides. The use of metal hydride technology has reduced the size and complexity of many systems, while increasing overall reliability. The use of metal hydrides in tritium processing is still expanding, and examples will be given of new applications. An overview of current uses of the technology, tritium aging characteristics of the materials, and future research areas will be presented.

9:40 AM Plenary
Modelling Multicomponent Alloys for Hydrogen Storage: *Reiner Kirchheim*[1]; [1]Universit‰t Gˆttingen, Institut f¸r Materialphysik, Hospitalstr. 3-7, Gˆttingen D-37073 Germany

Hydrogen storage alloys used today are multicomponent and structurally disordered containing large fractions of dislocations and grain boundaries. The current status of modelling pressure composition isotherms and H-diffusivity for these hydrogen-metal systems will be reviewed. Most of the models are based on a distribution of site energies for hydrogen which arises from variations of the chemical nature of the nearest neighbouring metal atoms as well as from a variation of the interstitial size occupied by hydrogen. Model predictions are compared with experimental findings. Recent results on the interaction between dislocations, grain boundaries and phase boundaries on the one hand and hydrogen on the other hand are presented for model systems. Corresponding experimental results are obtained by small angle neutron scattering and by tomographic atom probe analysis.

10:10 AM Break

10:20 AM Invited
Amorphization of $GdFe_2$ by Hydrogen Absorption: *Dhanesh Chandra*[1]; Ricardo B. Schwarz[2]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557-0136 USA; [2]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MST Div., MS G755, Los Alamos, NM 87545 USA

We have studied the reaction of hydrogen with $GdFe_2$, a Laves phase alloy. At room temperature and for a pressure of 1.3 MPa, the alloy absorbs reversibly 1.6 atoms of hydrogen per metal atom, forming $GdFe_2H_{4.8}$. At this temperature, the slow kinetics makes it difficult to remove all the hydrogen from the alloy within reasonable time and, for pressures below 10^3 Pa, the desorption isotherm deviates from the absorption isotherm. At intermediate temperatures and pressures, the reaction leads to the formation of a single-phase amorphous $GdFe_2H_x$ phase. This reaction is quite unusual in that, depending on the temperature and pressure of the transformation, the crystal-to-amorphous transformation is accompanied by either a gain or a loss of hydrogen. At temperatures above 525 K, hydrogen absorption causes the disproportionation of the $GdFe_2$ crystal into a two-phase mixture of GdH_x (x ª 2) and bcc-Fe.

10:40 AM Invited
Electronic Structure of Hydrogen Storage Intermetallic Compounds: *MichËle Gupta*[1]; [1]UniversitÈ Paris-Sud, Inst. Scis. des MatÈriaux, UMR8647, B‚t. 415, Orsay 91405 France

We review our recent work on the electronic structure of hydrogen storage intermetallic compounds of AB5, AB2 and AB type, where A and B are transition elements. Using the results of ab-initio band structure calculations, we discuss the features of the electronic properties relevant to storage applications such as the factors controlling the stability, the maximum hydrogen absorption capacity, the H site occupancy; we also analyze the modification of some fundamental properties of the matrix such as superconductivity and magnetism on hydrogen absorption. The following examples will be presented: i) Effect of mono and polysubstitutions of Ni in LaNi5 by 3d and s-p elements (Mn,Fe,Co,Cu,Al,Sn). ii) Role of chemical bonding on the stability and hydrogen site occupancy of AB hydrides (A=Zr, B=Co,Ni). iii) Hydrogen induced change of the superconductivity of the C-15 Laves phases AB2 (A=Zr, Hf, B=V) and modification of the magnetic properties of YB2 (B=Mn,Fe). We review our recent work on the electronic structure of hydrogen storage intermetallic compounds of AB5, AB2 and AB type, where A and B are transition elements. Using the results of ab-initio band structure calculations, we discuss the features of the electronic properties relevant to storage applications such as the factors controlling the stability, the maximum hydrogen absorption capacity, the H site occupancy; we also analyze the modification of some fundamental properties of the matrix such as superconductivity and magnetism on hydrogen absorption. The following examples will be presented: i) Effect of mono and polysubstitutions of Ni in LaNi5 by 3d and s-p elements (Mn,Fe,Co,Cu,Al,Sn). ii) Role of chemical bonding on the stability and hydrogen site occupancy of AB hydrides (A=Zr, B=Co,Ni). iii) Hydrogen induced change of the superconductivity of the C-15 Laves phases AB2 (A=Zr, Hf, B=V) and modification of the magnetic properties of YB2 (B=Mn,Fe). We review our recent work on the electronic structure of hydrogen storage intermetallic compounds of AB5, AB2 and AB type, where A and B are transition elements. Using the results of ab-initio band structure calculations, we discuss the features of the electronic properties relevant to storage applications such as the factors controlling the stability, the maximum hydrogen absorption capacity, the H site occupancy; we also analyze the modification of some fundamental properties of the matrix such as superconductivity and magnetism on hydrogen absorption. The following examples will be presented: i) Effect of mono and polysubstitutions of Ni in LaNi5 by 3d and s-p elements (Mn,Fe,Co,Cu,Al,Sn). ii) Role of chemical bonding on the stability and hydrogen site occupancy of AB hydrides (A=Zr, B=Co,Ni). iii) Hydrogen induced change of the superconductivity of the C-15 Laves phases AB2 (A=Zr, Hf, B=V) and modification of the magnetic properties of YB2 (B=Mn,Fe).

11:10 AM Invited
Palladium-Coated Kielselguhr for Simultaneous Separation and Storage of Hydrogen: *Robert H. Hsu*[1]; J. E. Klein[1]; [1]Westinghouse Savannah River Company, Aiken, SC USA

Palladium finely dispersed in a substrate of kielselguhr, Pd/k for short, has been successfully used for hydrogen storage, separation and pumping for a number of years at the Savannah River Site (SRS). Recently SRS has developed a new pd/k flow through bed (FTB) prototype for separating hydrogen from other gases and simultaneously storing the hydrogen on palladium. The FTB uses single-pass liquid nitrogen cooling during hydrogen absorption/separation/storage and electrical heating for desorption/unloading of hydrogen. More than ten (10) absorption-desorption or loading/unloading cycles using hydrogen have been conducted with the new FTB prototype. Test results show that all design performance objectives have been successfully achieved: recover >95% of hydrogen gas from feed gas, produce 99.9% hydrogen product gas and < 5% hydrogen in discharge gas. This presentation will discuss characteristics of the palladium-coated kielseguhr or diatomaceous earth, design and operation of the FTB, and results of performance tests such as separation efficiency, hydrogen storage capacity and system heat transfer characteristics.

11:40 AM Invited
Hydrogen Sorption of Mg Based Mixtures Elaborated by Reactive Mechanical Grinding: *J.-L. Bobet*[1]; B. Chevalier[1]; M. Y. Song[2]; B. Darriet[1]; [1]Institut de Chimie de la MatiËre CondensÈe de Bordeaux, ICMCB, CNRS [UPR 9048], UniversitÈ Bordeaux 1, 87 Ave. du Dr A. Schweitzer, 33608 Pessac, Cedex France; [2]Chonbuk National University, Div. of Adv. Matls. Eng., 664-14 1ga Deogjindong Deogjingu Chonju 561-756, S. Korea

Due to their high hydrogen storage capacity, lightweight, low cost and abundance in the earthís crust, magnesium is one of the most promising candidates for hydrogen storage materials. However, the hydriding-dehydriding reaction takes place at high temperature (i.e. 287∞C) and is relatively slow. One of the ways to kinetically improve magnesium based hydrogen storage materials is by the (i) addition of metals and/or intermetallics and or (ii) application of high energy ball milling. The aim of this study was to show the influence of mechanical alloying under hydrogen (Reactive Mechanical Grinding) on the chemical properties (crystallographic and phase composition) and on the hydrogen storage properties of different mixtures Mg + additive (= metal, intermetallics or oxides). The use of Reactive Mechanical Grinding (MG under H2) on magnesium powder improves the hydrogen sorption properties. The hydrogenation of Mg starts in situ during the milling process that allows suppressing the activation procedure generally requested for Mg. The addition of metals (Co, Ni), which acts as a catalyst for the reaction of dissociation of H2, also leads to an improvement of the hydrogen sorption properties (but a strong dependence upon the milling time is reported). The addition of intermetallics such as YNi and YNi2 also leads to an improvement of the hydrogen absorption kinetic but the mechanism is different. In this case, the additive acts as a hydrogen ìpumpî and then enables the diffusion of hydrogen in the Mg matrix. It is also shown that the addition of oxides such as Cr2O3 also improve the kinetic on the contrary the addition of Al2O3 (same structure) or CeO2 do not improve it. The hydriding is generally determined to be a two-step process: nucleation and diffusion. A direct relationship exists between the nucleation duration and the specific surface. Increasing the milling time up to 5 hours allows to increase the specific surface. The diffusion is controlled by the number of crystallites per particle. A critical milling time exists below which the diffusion process is improved and beyond which no more improvement is observed (the maximum internal stress in the powder is also reached at this critical time). We also try to explain why Cr2O3 addition lead to an improvement of the kinetic as CeO2 and Al2O3 does not improve it.

Fundamentals of Structural Intermetallics: Strengthening and Microstructure of TiAl

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

Wednesday AM	Room: 615-616
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Young-Won Kim, UES, Inc., Matls. & Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; Marc Thomas, ONERA-DMMP, 29 ave. de la Div. Leclerc, Chatillon, Cedex 92322 France

8:30 AM Invited

Elevated Temperature Tensile Properties of Gamma TiAl: Marcus Zupan[2]; *Kevin J. Hemker*[1]; [1]Johns Hopkins University, Dept. Mechl. Eng., Baltimore, MD 21218 USA; [2]Cambridge University, Micromech. Ctr., Trumpington St., Cambridge CB2 1PZ UK

Deformation in two-phase TiAl alloys occurs most readily in the gamma-TiAl phase, and measurements of the elevated tensile properties of gamma-TiAl are needed to provide a solid foundation for modeling the mechanical performance of these alloys. Microsample specimens of single crystalline gamma-Ti 55.5at%Al oriented near the [001], [010] and [-110] crystallographic axes have been tested in both tension and compression at temperatures ranging from 500∞ to 1000∞C. Measurements of the of the Youngís modulus, coefficient of thermal expansion, and flow strength have been made as a function of temperature, crystal orientation and sense of the applied load. The Youngís modulus and coefficient of thermal expansion measurements were found to be comparable to literature values, obtained at lower temperatures, and can be used to expand the range over which these properties are known. Anomalous dependence of the flow strength, measurable violations of Schmidís law and significant tension/compression asymmetries have been recorded at all orientations. This unusual tensile behavior has been related to the dissociation and cross-slip locking of superdislocations, and the details of this locking mechanism will be used to characterize the tension/compression asymmetry and the elevated temperature deformation behavior of this alloy.

9:00 AM

High Temperature Deformation Behavior of Gamma TiAl Alloy-Microstructural Evolution and Mechanisms: J. H. Kim[1]; T. K. Ha[1]; Y. W. Chang[1]; *C. S. Lee*[1]; [1]Center for Advanced Aerospace Materials, Dept. of Matls. Sci. & Eng., Pohang Univ. of Sci. & Tech., San 31, Hyoja-dong, Pohang 790-784 Korea

Enhancement of ductility and hot workability is an important issue in the gamma-TiAl alloys. To achieve such a goal, it is necessary to clarify the governing deformation mechanisms with the variation of temperature and microstructure. Present investigation was carried out to enlighten the high temperature deformation mechanism in view of the inelastic deformation theory. A series of load-relaxation tests, tensile tests and compression tests were conducted on two-phase gamma TiAl alloy at 800C to 1020C. Results of load relaxation test (e = 0.04) showed that deformation was well described by sum of grain matrix deformation and dislocation climb process. When the alloy was deformed up to e = 0.8, grain refinement was observed to occur due to dynamic recrystallization process. Correspondingly, the flow curve changed its shape significantly resulting the broad concave portion in the intermediate strain rate region, which indicates the operation of GBS at this large strain level.

9:20 AM

Work Hardening Stability and its Implication on Component Strength: *Jonathan Paul*[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strasse, Geb45, WFP, Geesthacht, D-21502 D-21502 Germany

This paper studies the origin and thermal stability of the defects that give rise to work hardening during room temperature deformation. The investigations involve mechanical testing, electron microscope observations of the defect structure, and recovery experiments. The investigations indicate that work hardening is derived from long-range elastic interactions between dislocations on parallel and oblique slip planes. Another source of work hardening arises from dislocation dipoles and debris defects, which were trailed and terminated at jogs in screw dislocations. Removal of these defects through annealing gives rise to a significant recovery of the work hardening. Thus, most of the room temperature work hardening is lost after just a few hours exposure to operating temperatures (700-750∞C). A novel heat-treatment/processing procedure that has the potential to increase high temperature strength has also been investigated. The method involves introducing work hardening through deformation and then performing a heat-treatment to stabilize deformation induced defect structure.

9:40 AM Invited

Effect of Y-Addition on the Mechanical Properties and Oxidation Resistance of TiAl-Mn-Mo: *Sun-Keun Hwang*[1]; Ying Wu[1]; [1]Inha University, Sch. of Matls. Sci. & Eng., 253 Yonghyun-Dong, Nam-Gu, Incheon 402-751 Korea

TiAl-based alloys with the nominal chemical compositions of Ti-46.6Al-1.4Mn-2Mo-0.3C-xY (x=0.1, 0.33 and 0.6), were developed with Elemental Powder Metallurgy method. The alloys with yttrium addition had higher ultimate tensile strength, elongation and oxidation resistance than the Y-free alloy. Combined solid solution strengthening of Mn, Mo, C and Y, and the microstructural refining effect of C and Y were responsible for the increased strength at room temperature as well as at high temperature up to 800∞C. Besides, Y2O3 and carbide precipitation also enhanced the strength. The room temperature ductility was improved by the microstructural refinement and by the reduction of interstitial oxygen in the a2 and g matrix. The improved ductility was evidenced by dislocations with ordinary perfect Burgers vectors in deformed g phase. The improvement of oxidation resistance by yttrium addition was attributed to the reduction of the concentration of oxygen vacancies in the TiO2 scale.

10:10 AM

Interstitial Elements in Ternary Ti-Al-Cr Alloys: Microstructural Effects: *MÈlanie Lamirand*[1]; Jean-Louis Bonnentien[1]; Michel Cornet[1]; *Jean-Pierre Chevalier*[1]; [1]CECM-CNRS, 15, rue Georges Urbain, Vitry, Cedex 94407 France

Microstructural modifications in Ti-Al-Cr as a function of controlled additions of O, C and N will be presented. Previous work on binary Ti48Al has shown that these interstitial elements affect significantly the proportion of alpha 2 and gamma phases in fully lamellar alloys. The alpha 2 volume fraction increases with increasing interstitial content, yielding a smaller interlamellar spacing, which results in a higher yield stress. Primary creep rate also showed a marked reduction as a function of increasing O, C and N content. This approach has been applied to the ternary Ti48AlCr2 alloy, using both ultra high purity (60 wt ppm oxygen) and commercial purity Ti associated with clean processing. Variable amounts of O, C and N have been added in a controlled manner, and the microstructure has been analysed using a field emission gun scanning electron microscope, for given heat treatments and cooling rates. The aim of this work is to ascertain whether the effects previously observed can be transposed to such ternary alloys. The study will also be extended to the duplex (gamma plus lamellar) microstructure. This work also explains, at least partially, the substantial scatter in properties observed in industrial grade alloys, where both the initial interstitial content and pick up of at least O during heat treatment is not necessarily systematically assessed.

10:30 AM

Characterization of Solute Partitioning in a TiAl-Based Alloy by the Quenching during Directional Solidification Technique: *Matthias Charpentier*[1]; Marjolaine Grange[2]; Dominique Daloz[1]; Elisabeth Gautier[1]; Alain Hazotte[1]; GÈrard Lesoult[1]; [1]ENSMN-LSG2M, Parc de

Saurupt, Nancy, Cedex 54042 France; [2]SNECMA Moteurs, DT/DMP, Site de Villaroche, Moissy-Cramayel 77550 France

One major hindrance to effective application of cast gamma TiAl-based intermetallic alloys in aircraft engines lies in the variability of their mechanical properties resulting from chemical thus microstructural heterogeneities. In the present work, the build-up of microsegregation in a cast Ti-48Al-2Cr-2Nb alloy is investigated through experiments of quenching during directional solidification (QDS). The first solidification steps as well as the alloying elements partitioning between the solid and the liquid phases are clarified. The results are discussed with regard to the range of chemical heterogeneity determined within Ti-48Al-2Cr-2Nb investment cast parts. Regarding subsequent solid phase transformations, the study also tends to explain the formation of minor phases stabilised by the ternary additions.

10:50 AM
Phase Transformations in Multicomponent Gamma Titanium Aluminides: Zhihong Zhang[1]; Dennis M. Dimiduk[2]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Air Force Research Laboratory, Matls. Direct., WPAFB, Dayton, OH 45433 USA

Phase transformations and microstructure evolution in Ti-(45-47)Al-2Cr-2Nb-(0-1)Mo-(0-0.2)B and Ti-45Al-2Mn-2Nb-0.3 wt.%B XD alloys have been studied by electrical resistivity measurements, x-ray diffraction, scanning electron microscopy with back-scattered electron imaging and analytical transmission electron microscopy. Phase boundaries and transformation temperatures during heating and cooling were determined. The transformations of the high-temperature beta, alpha or both phases during continuous cooling, as well as during step-quench+isothermal holding were studied, and the competition in temperature and time space between the various transformation modes, namely, lamellar, cellular, feathery and massive gamma were ascertained. The structure and chemistry of phases present in alloys with and without boron were determined, and these were useful for establishing isothermal phase diagrams displaying the change in phase equilibria with alloying and temperature. Continuous cooling transformation diagrams were also established and detailed microscopy was performed to unravel transformation mechanisms associated with the various non-lamellar transformation modes (especially the cellular and feathery modes) and the influence of chemistry. Finally, experiments aimed at elucidating the effects of boron on transformations and microstructural evolution were performed. Based on the results obtained, explanations for the effects of the alloying additions and thermal history on microstructure evolution, transformation modes, mechanisms, kinetics and phase equilibria are provided. Support for this research from NSF Grant# DMR-9731349, Dr. Bruce MacDonald, Program Monitor and AFOSR Grant# F49620-95-1-0116, Dr. Craig Hartley, Program Monitor, is deeply appreciated.

11:20 AM
Influence of Alloying Elements on the Kinetics of Massive Transformation in Gamma Titanium Aluminides: Uttara Prasad[1]; *Mahesh C. Chaturvedi*[1]; [1]University of Manitoba, Dept. of Mechl. & Indl. Eng., 356 Engineering Bldg., 15 Gillson St., Winnipeg, Manitoba R3T 5V6 Canada

The effect of alloying elements, Nb and Mn on massive transformation in Ti-45Al based alloys was studied by heat treatment of the alloy samples at 1350∞C for 30 minutes followed by cooling to room temperature at the rate of ~700∞C/sec and subsequent microstructural characterization. The amount of massively transformed gamma was observed to increase from zero for 0 percent Mn to ~70 percent for 2 percent Mn. Nb had minimal influence on the rate of transformation. This effect of Mn and Nb has been rationalized on the basis of the site occupancy of the alloying elements and their influence on the phase boundaries and grain size. TEM analysis has also been conducted to understand the mechanism of massive transformation in these alloys. Based on the results obtained, the effect of Mn and Nb on the kinetics of massive transformation will be discussed and CCT diagrams for different alloys will be proposed.

11:40 AM
Influence of Oxygen on Phase Transformations in a Ti-48 at. % Al Alloy: *Williams Lefebvre*[1]; Annick Loiseau[2]; Alain Menand[1]; [1]Groupe de Physique des MatÈriaux, UMR CNRS 6634, FacultÈ des Sciences, 1 rue Thomas Beckett, Mont Saint Aignan 76 821 France; [2]Laboratoire díEtude des Microstructures, UMR CNRS-Onera, Onera, BP 72, 29 av. de la Division Leclerc, Chatillon 92322 France

Near equiatomic Ti-Al alloys, especially the Ti-48 at. % Al alloy, have been intensively investigated during the last decade. It is now well established that Ti-48 at. % Al alloys can exhibit various microstructures, depending on the cooling rate from high temperature (i.e. 1670 K). Many studies have contributed to get a better understanding of phase transformations involved in this alloy. However, the influence of interstitial impurities (e.g. C, N, O) on phase transformations has not been sufficiently investigated. This kind of study is often difficult to make, as most analytical techniques cannot quantitatively analyse light elements. In this study, alloys with a Ti- 48 at. % Al base composition and various oxygen contents have been investigated by APFIM (Atom Probe Field Ion Microscopy) and TEM (Transmission Electron Microscopy). Unlike other analytical techniques, AP can detect all elements including light atoms. Heat treatments have been applied to obtain either lamellar (slow cooling rate) or massive structures (high cooling rate). Beyond the well-known oxygen scavenging effect of the $\alpha 2$ phase, it will be demonstrated that oxygen has an essential influence during both lamellar and massive transformations.

General Topics in Waste Treatment and Minimization: Environmental Thermal Treatments and Processes

Sponsored by: Extraction & Processing Division, Process Mineralogy Committee, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Cheryl Dahlin, US Department of Energy, Albany Research Center, Albany, OR 97321-2152 USA; Jasper Kwong, US Department of Energy, Albany Research Center, Albany, OR 97321-2198 USA

Wednesday AM	Room: 604
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Jasper Kwong, US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA; Cheryl L. Dahlin, US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

8:30 AM
Effect of Slag Viscosity on the Separation of PGMís during Electric Scrap Melting: *Jeong-Whan Han*[1]; Jae-Hong Zee[1]; Sin-Suk Han[1]; *Jae-Chun Lee*[2]; [1]Inha University, Sch. of Matls. Sci. & Eng., 253 Yonghyun-dong Namku, Inchon 402-751 Korea; [2]KIGAM, Recycling Rsrch. Ctr., 30 Gajeong-dong Yuseong-ku, Daejeon 305-350 Korea

A recovery of PGMs from spent printed circuit board(PCB) scraps has been investigated by using pyrometallurgical process. For this purpose vertical electrical resistance furnace and high frequency induction furnace were used for clacination and melting the scraps, respectively. In order to enhance PGMs separation from the scrap, various slag system were attempted to be used in the process and copper was used for collector metal to collect PGMs from the scrap. Main variables used in the experiments were the slag properties such as composition and temperature of the slags. High temperature viscometer was used to measure the viscosity of slag systems. It was found that viscosity of slag greatly influences on the separation of PGMs from the melted scrap.

8:50 AM
The Environmental Benefits from Computer Modeling EAF Slag Chemistry: *Kyei-Sing Kwong*[1]; James P. Bennett[1]; [1]US Department of Education, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

The recycling of MgO-C refractories as a slag conditioner in EAF (electrical arc furnace) foaming slags has been evaluated by ALRC. The amount of MgO in the slags is critical for the foaming quality. Good foaming slags provide the benefits of high energy efficiency and low refractory consumption and require saturation with MgO. A computer model predicting the chemistry of MgO saturated foaming slags was designed to predict suitable amount of MgO additives from MgO-C spent refractories. This model can also help to reduce the amount of slags and to improve the economics of producing steel. The implementation of this recycling program is underway at steel plants and the experience of recycling spent refractories will be discussed.

9:10 AM
Zinc Recovery from Eaf Dust through Thermal Reduction: *D. Mishra*[1]; N. Kanari[1]; I. Gaballah[1]; B. DuprÈ[2]; [1]Laboratorie Environmental et Mineralurgie, Minl. Proc. & Environml. Team, Rue du Doyen Marcel Roubault, BP 40, 54501, Vandoeuvre, Cedex France; [2]Laboratorie de Chimie du Solid Mineral, UHP Nancy 1 UMR 7555, BP 239, 54506, Vandoeuvre, Cedex, France

Electric arc furnace dust (EAFD) generated during the recycling of steel-scrap is classified as a hazardous waste for landfilling and dumping. This is because it contains leachable heavy metals such as zinc, lead? Recovery of these metals could result in both resources conservation and resolving waste disposal problems. A study of the thermal reduction of a sample of EAFD was carried out in an inert gas atmosphere using coke as a reducing agent. Preferential volatilization of zinc and lead was achieved during the reduction process at about 800∞C. More than 96% of both zinc and lead were removed from the dust and were recovered in the condensates. The treatment residue was essentially composed of iron oxides and almost free from heavy metals.

9:30 AM
Behavior of Ceria as an Actinide Surrogate in Electro-Slag Remelting and Refining Slags: *Uday B. Pal*[1]; Timothy Keenan[1]; Ajay Krishnan[1]; William C. Chernicoff[1]; Christopher J. MacDonald[1]; Edward Chiang[1]; James Van Den Avyle[2]; M. A. Molecke[2]; D. Melgaard[2]; [1]Boston University, Mfg. Eng., 15 St. Maryís St., Boston, MA 02446 USA; [2]Sandia National Laboratory, Liquid Metals Proc. Lab., Albuquerque, NM 87185 USA

Downsizing and decommissioning of nuclear facility operations is increasing the stockpile of various different grades of Radioactive Scrap Iron (RSI). It is desirable to be able to decontaminate the RSI to a very low level that can be recycled or used for fabrication of containers for RSI disposal. Electro-Slag Remelting and Refining (ESR) is often used for decontaminating radioactive contaminated sources of scrap iron such as stainless steel. Non-radioactive oxides of cerium are used to simulate the radioactive oxide contaminants of Uranium and Plutonium. The success of the ESR process is strongly dependent on having the right thermo-physical-and-chemical slag properties. We measured the following relevant slag properties: capacity to incorporate the radioactive contaminant (simulant), volatilization rate and volatile species, electrical conductivity, viscosity, surface tension, slag-metal partition coefficient, and ceria dissolution kinetics as a function of temperature. The impact of these properties on the ESR decontamination process will be discussed.

9:50 AM Break

10:10 AM
A Plasma Assisted Cyclone Reactor for Vitrification: *Patrick Rodger Taylor*[1]; [1]University of Tennessee, Matls. Sci. & Eng., 435 Dougherty Engineering Bldg., Col. of Eng., Knoxville, TN 97996-2200 USA

A non-transferred arc thermal plasma assisted cyclone reactor has been designed, built and operated to evaluate its use in vitrification of wastes. Simulated waste (liquid, slurry or fine particulate) is injected near the plasma flame, where nearly instantaneous dehydration and calcination reactions happen. A non-transferred arc plasma torch was attached tangentially to the cyclone and is utilized as both an enthalpy source and to provide the gas velocities required to generate the cyclonic gas flow patterns. The closed top cyclone flow sends the solid particulates to the wall, where they are captured by a thin film of molten glass that is injected as a powder at a position lower in the reactor. The molten glass is then collected in a crucible. The feasibility of simulant injection directly into the flame zone and its effect on electrode lifetimes has been studied. Several candidate refractory systems have been evaluated for their suitability for extended use under the demanding conditions present in the cyclone reactor.

10:30 AM
Properties of Iron Phosphate Waste Forms: *Patrick Rodger Taylor*[1]; Wanqing Huang[2]; [1]University of Tennessee, Matls. Sci. & Eng., 435 Dougherty Engineering Bldg., Col. of Eng., Knoxville, TN 97996-2200 USA; [2]University of Idaho, Moscow, ID 83844-3024 USA

Iron phosphate glasses are promising host matrices for vitrifying actinide rich nuclear waste materials because of their high solubility of actinide oxides, high chemical durability and relatively low melting temperature. Viscosity measurements were made on iron phosphate over the temperature range of 1000 to 1200C. Partitioning behavior was studied for Ce and Sr. The results show that the viscosity behavior is very sensitive to the composition and that both Ce and Sr have high solubilities in the glass under the conditions studied.

10:50 AM
Understanding of Hazardous Waste Incineration through CFD Simulation: *Yongxiang Yang*[1]; Markus A. Reuter[1]; Jack Voncken[1]; Joep Verwoerd[2]; [1]Delft University of Technology, Dept. of Appl. Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands; [2]AVR-Chemie, Prof. Gerbrandyweg 10, Rotterdam-Botlek 3197 KK The Netherlands

Rotary kiln incinerators are widely used in the incineration of various types of hazardous wastes. However, the complex transport processes within the incinerator are not well understood. Due to large variations in waste types and difficulties in feed characterization, the incineration process meets great challenges in a smooth operation, and expects uncertainties in the process chemistry and emission control. In order to get better understanding of the incineration process, process simulation was made on the kiln behavior by using Computational Fluid-dynamics (CFD) to characterize gas flow, temperature distribution and waste combustion in the incinerator. Temperature measurement was conducted in an operating incinerator to validate the CFD model. This paper addresses the environmental issues related to the hazardous incineration process, and reports the recent simulation results on gas flow and mixing, combustion heat transfer, and potentials to reduce emissions (CO and dioxins).

11:10 AM
Environmentally Sound Technology for Processing of Domestic Solid Waste and Other Toxic Waste in Sparged Molten Slag: *A. V. Grechko*[1]; V. F. Denisov[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals ìGintsvetmetî, 13, Acad. Korolyov St., 129515 Moscow Russia

An environmentally clean and virtually waste-less technology for treatment of toxic chloride-containing waste materials has been developed and proven on a full scale by the Gintsvetmet Institute in cooperation with other Russian organizations. The proposed process resolves the acute and globally significant problem of dioxin formation. The purpose of the proposed technology is to ensure a destruction of the structural lattice that would guarantee irreversible transformation of the resultant substances to prevent formation of dioxins. The preconditions that are required to achieve this purpose are a temperature >1250C; an oxidant excess coefficient >1.05; a retention time of the products under these conditions (>2 s); and a thermal instantaneousness of the process involving a transition with no intermediate phase. These conditions were achieved with the experimental development of the Vanyukov furnace design commonly used for sparged smelting processes at non-ferrous metallurgical plants in Russia and Kazakhstan.

High Performance Metallic Materials for Cost Sensitive Applications: Intermetallics

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

Wednesday AM — Room: 213
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Deliang Zhang, University of Waikato, Hamilton New Zealand; Sunil N. Patankar, University of Idaho, Moscow, ID 83844 USA

8:30 AM
Thermal Stability of Alumina Mold against Molten Ti-Al Alloys: *Myoung-Gyun Kim*[1]; Si-Young Sung[1]; Hee-Kook Kim[1]; *Young-Jig Kim*[1]; [1]Sungkyunkwan University, Sch. of Metlgcl. & Matl. Eng., 300, Chunchun-dong, Changan-gu, Suwon, Gyounggi-do 440-746 Korea

Investment casting of titanium aluminides has received a considerable attention in the cost-saving process because of their poor workability and ductility. Especially, the selection of mold material is essential for investment casting of titanium aluminides due to their high reactivity in the molten state. Alumina is regarded as a promising investment mold material in terms of formability, cost and thermal stability from the previous study. In order to investigate the thermal stability of alumina mold against molten Ti-Al alloys, the Ti-(10°≠55)at% Al alloy buttons were prepared by melting and casting in a plasma arc melting furnace under argon atmosphere. The selections were based on the Ti-Al binary phase diagram. The cast microstructures of interface between Ti alloy and alumina mold have been studied as a function of composition using optical microscope, electron probe X-ray microanalyzer and hardness measurement.

8:50 AM
Grain Size Control in Ti-48Al-2Cr-2Nb with Yttrium Additions: *Pankaj Trivedi*[1]; E. G. Baburaj[1]; S. N. Patankar[1]; F. H. (Sam) Froes[1]; A. Genc[2]; L. Ovecoglu[2]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Istanbul Technical University, Istanbul, Turkey

Gas atomized powder of the Ti-48Al-2Cr-2Nb intermetallic with additions of 1.6 wt. % Y were mechanically alloyed (MAíd) and hot isostatically pressed (HIPíd) to produce fully dense nanocrystalline material. MA of the as blended powder for 16 hrs resulted in the formation of a disordered fcc phase. HIPíing of the alloy powder produced a single-phase nanocrystalline TiAl intermetallic, containing a distribution of 5-10 nm sized Al2Y4O9 particles. The formation of oxide particles occurred by the chemical combination of Al and Y with oxygen, the latter element entering as a contamination during milling. Oxide particles increased the strength of the intermetallic compound and minimized grain growth even at 0.8Tm, where Tm is the melting point of the compound.

9:10 AM
Synthesis and Consolidation of Nanocrystalline Titanium Aluminides: E. G. Baburaj[1]; C. Draney[1]; R. Fielding[1]; F. H. (Sam) Froes[1]; S. Thevuthasan[2]; V. Shutthanandan[2]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]Pacific Northwest National Laboratory, Environmental Molecular Scis. Lab., Richland, WA 99352 USA

Nanocrystalline TiAl based intermetallics have been produced by mechanical alloying (MAíing) followed by reaction during hot isostatic pressing (HIPíing). In this process titanium hydride and aluminum powders are milled to an ultrafine size followed by HIPíing to form the hydrided Ti-Al. The advantages of the process are the ease of alloy formation and auto-hydrogenation of the ultrafine powders to passivate the powders against oxidation during consolidation. Studies on dehydrogenation of consolidated samples using resonant Nuclear Reaction Analysis (NRA) revealed a concentration gradient of hydrogen in TiAl samples as a function of depth from the surface and complete removal of hydrogen from the TiAl lattice by vacuum annealing. The effect of hydrogen in TiAl as a temporary alloying element will be discussed in detail.

9:30 AM
Production of γ-TiAl Sheets with Improved Superplastic Properties by Pack Rolling: *M. R. Shagiev*[1]; G. A. Salishchev[1]; F. H. (Sam) Froes[2]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin Str., Ufa-450001 Russia; [2]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The sheets of γ-TiAl based alloys with improved superplastic properties are of a great interest for aerospace applications. Such properties can be achieved through the grain refinement to micron and submicron level. Grain refinement may also lead to considerable decrease in the temperature intervals of brittle-to-ductile transition and, consequently, of pack rolling. Therefore, the smaller the grain size in rolling preform and the lower the rolling temperature, the higher superplastic properties in the sheet. Besides the microstructure improvement in γ sheets, decrease in rolling temperature will decrease their cost because of energy savings and use of the cheap can materials. The temperature intervals of pack rolling of several γ alloys were defined based on tensile properties at strain rates corresponding to rolling ones. Optimization of texture and rolling regimes were performed in order to eliminate anisotropy of superplastic properties in the sheet. Pack rolling temperature of Ti-50Al alloy with homogeneous microcrystalline structure was found to be 950∞C while that of Ti-45Al-2Cr-2Nb-0.45B alloy was 1100∞C. At 1000∞C and strain rate of 10-3 s-1, both sheets exhibited elongations of about 200% and steady state flow stresses of 140 MPa.

9:50 AM Break

10:10 AM
Chemical Reaction Behaviour of Titanium Aluminides and α-Al2O3 with NaOH Aqueous Solution: *G. Adam*[1]; Deliang Zhang[1]; [1]University of Waikato, Dept. of Matl. & Proc. Eng., PB 3105, Hamilton, New Zealand

Titanium metal and titanium alloys are materials of high value, because of their high specific strength, high corrosion and oxidation resistance and good biological compatibility with human tissues. Although they are mainly used in aerospace and chemical processing industries, titanium metal and alloys are particularly attractive to the automotive industry. However, they have not been widely used in manufacturing automobiles, because of their high cost. Development of the ways in which the cost of producing titanium metal and alloys has a significant implication in the pursuit of making lighter and lighter vehicles. As part of a study which aims at developing techniques of extracting titanium alloys or titanium based intermetallic compounds, we investigated the reactions of Ti3Al, Al3Ti and Al2O3 fine particles with caustic solutions under different conditions. Reactions of alpha alumina (α-Al2O3) with caustic solutions have been previously studied, but there has not been much research work on the reactions between titanium aluminide powders and caustic solutions. This paper is to present and discuss the results of this investigation and the implications of the results in developing an alternative process of extracting titanium metal from titanium containing minerals.

10:30 AM
Processing and Properties of Titanium Aluminide-Ceramic Composite Materials: *Deliang L. Zhang*[1]; Z. H. Cai[1]; J. Liang[1]; G. Adam[1]; [1]University of Waikato, Dept. of Matls. & Proc. Eng., PB 3105, Hamilton New Zealand

Titanium aluminides (Al3Ti, TiAl and Ti3Al)-ceramic composite materials are important materials due to their high specific modulus, high specific strength, high creep resistance and good oxidation resistance. Depending on the matrix phase and the desired ceramic reinforcement features, This type of materials can be synthesized by either reacting TiO2 with Al or mechanical alloying Ti and Al elemental powders and ceramic powders. The former technique is used for producing low cost and moderate performance composite materials which are potential materials for manufacturing automotive parts, while the latter technique is used to produce high cost but high performance nanocomposites with potential applications in aerospace industry. It has been established that a combination of high energy mechanical milling, thermal treatment is very effective in achieving the desired microstructure and properties. This paper is to provide an overview of the results of a comprehensive study undertaken at Waikato University on the inter-relationships among composition, processing condition, microstructure and properties of the titanium aluminide-ceramic composite materials synthesized using these techniques. Work published in literature on this type of materials will also reviewed. Ways of improving the fracture toughness of the composite materials and their potential applications will be discussed.

10:50 AM
Production of Ti3Al Based Composite Materials using Mechanical Alloying: *Jing Liang*[1]; D. L. Zhang[1]; W. Gao[3]; Z. W. Li[2]; [1]The University of Waikato, Dept. of Matls. & Proc. Eng., PB 3105, Hamilton New Zealand; [2]The University of Auckland, Dept. of Cheml. & Matls. Eng., Auckland, New Zealand; [3]The University of Auckland, Dept. of Cheml. & Matls. Eng., Auckland, New Zealand

Ti3Al based composite powders containing SiC, TiC and other ceramic particulate were produced by using high-energy mechanical milling. The morphology and microstructure of the as-milled powder particles were investigated using X-ray diffraction (XRD) and scanning electron diffraction. Composite structured powder with the ceramic particles incorporated in the metal matrix formed after milling the elemental powder mixture for 2 hours. With increasing the milling time up to 16 hours, the ceramic particle sizes were reduced down to about 200 nm in average. The composite materials were then produced via cold isostatic pressing and vacuum sintering of the as-milled powder. The phases and structures of the composite materials were characterized using XRD, SEM, TEM and compositional analysis using energy dispersive X-ray spectrometer (EDX). It was found that the ceramic phase heavily reacted with the Ti3Al matrix under certain sintering conditions, so the effects of the ceramic type, size and sintering temperature and time on the reactions were studied. In order to lower the sintering temperature required to produce fully dense composite materials, hot isostatic pressing was used in consolidating the as-milled powders. High temperature oxidation resistance of the materials has also been studied. This paper is to present and discuss the results of this study, with the focus on the understanding of the relationships between processing conditions, microstructures and oxidation resistance of the Ti3Al based composite materials.

11:10 AM
Investigation on Fabrication of TiNiCu Shape Memory Alloy from Elemental Powders: Zheng Qifei[1]; Xie Shuisheng[1]; Yuan Guanshen[1]; [1]Beijing General Research Institute for Non-Ferrous Metals, Beijing China

TiNiCu alloy is one of shape memory alloy, which have excellent property and have been really used. It is difficult to get chemical homogeneity for TiNiCu alloys in usual melt-cast production process. The fabrication of TiNiCu shape memory alloy directly from elemental powders was studded. Powders of Ti, Ni and Cu element were mixed and cold pressed to ingot. The ingot was vacuum-sealed in a glass tube and heated in furnace for 0.5-2 hours at the temperature of 900-1050°, then pressed by a pseudo-HIPíing method at the press of 500-1000MPa and the time of 5-30 second. X-ray analysis of the product show that by this process the powders of Ti, Ni and Cu elements have became to TiNiCu alloy. Effects of parameters on the fabrication of TiNiCu alloy via above method, such as sintering temperature, sintering time, pressure and cold compact density, were studied.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Advanced Materials

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

Wednesday AM	Room: 617
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Ilya Ovidíko, Institute of Problems of Mechanical Engineering, Russian Academy of Sciences, Lab. for Theory of Defects in Matls., Bolshoj 61, Vas. Ostrov, St. Petersburg 199178 Russia; Donald U. Gubser, Naval Research Laboratory, Matls. Sci. & Tech., Code 6300, Washington, DC 20375 USA

8:30 AM Invited

Interfaces and Stresses in Nanostructured and High-Tc Superconducting Materials: *Ilya Ovidíko*[1]; [1]Institute of Problems of Mechanical Engineering, Russian Acad. of Scis., Lab. for Theory of Defects in Matls., Bolshoj 61, Vas. Ostrov, St. Petersburg 199178 Russia

This presentation briefly reviews theoretical models that describe the specific structural and behavioral peculiarities of interfaces (intergrain and interphase boundaries) in nanostructured and high-Tc superconducting materials. The special attention is paid to the role of interfaces as sources of stresses and their influence on the properties of nanostructured and high-Tc superconducting materials. In doing so, theoretical models are considered which focus on the following: (i) Interphase boundaries and stress distributions in nanocrystalline films and nano-scale multilayer coatings. Their effects on the protection properties of nanostructured films and coatings. (ii) Grain boundaries and grain boundary defects in nanostructured bulk materials. Their effects on the mechanical behavior of nanostructured materials. (iii) The structure and stress fields of grain boundaries in polycrystalline high-Tc superconductors. The effects of dilatation stresses of interfaces on high-Tc superconductivity in thin-film and bulk cuprates. This work was supported by the Office of US Naval Research, INTAS and Volkswagen Foundation.

8:55 AM Invited

Grain Boundary Effects in High and Low Tc Superconductors: *Chandra Shekhar Pande*[1]; Robert A. Masumura[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div, Code 6325, 3555 Overlook Ave. S.W., Washington, DC 20375 USA

A high critical current capacity is one of the most important properties needed in superconductors used in large scale applications such as electric motors, The role of grain boundaries is the most significant among the many factors that can effect the critical current in these materials. Interestingly, the behavior of grain boundaries is very different in low and high Tc superconductors. We discuss why grain boundaries enhance Jc in low Tc superconductors but it is the elimination of most of the large angle grain boundaries that is most critical for high Tc Superconductors. Specifically, it is the c-axis texture and a-b grain alignment that is important. These results are verified by texture measurements on BSCCO tapes. Current transport properties of these tapes were also measured and correlated with texture parameters. Mathematical models connecting Jc with grain boundaries in both low and high Tc materials are also discussed.

9:20 AM Invited

Concerning Doping, Segregation, Charge, and Stress at the Grain Boundaries of Cuprate High-Temperature Superconductors: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Matls. & Cheml. Scis. Div., ES&T Dept., Bldg. 480, PO Box 5000, Upton, NY 11973-5000 USA

Grain boundaries play a very significant role in the science and technology of high-temperature superconductors. Their effects can be desirable or undesirable, according to the required properties. On the one hand, Josephson junction devices can be fabricated using grain boundaries to form the junctions; on the other hand, great efforts must be made to reduce the deleterious effects of grain boundaries on the attainable critical current density in conductors for electric power applications. The short superconducting coherence lengths, the low carrier density with its sensitivity to stress and disorder, as well as the sensitivity of the metal-insulator transition to these variables, the complex crystal structure and the resulting anisotropy of superconducting properties are all important factors in the structure and the effects of grain boundaries in cuprate superconductors. In this talk I will discuss the thermodynamics and kinetics of these effects, with reference to the available data from transmission electron microscopy and with emphasis on the need for the use of open-system thermodynamics and kinetics, and for consideration of space charges and the effects of low carrier densities and the anisotropy of the layered crystal structure on its screening. I will also discuss the prospects for the use of suitable doping to alleviate the undesirable effects of grain boundaries in cuprates. This research was performed under the auspices of the US Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

9:45 AM Invited

Substituted Yttrium and Rare Earth Iron Garnet Films for Magneto-Opt Devices: *Pragati Mukhopadhyay*[1]; T. Chandra[2]; [1]Advanced Centre for Research in Electronics, Indian Inst. of Tech., Bombay, 4000076 India; [2]University of Wollongong, Dept. of Matls. Eng., Wollongong, NSW 2522 Australia

Garnet films have emerged as promising magneto-optic (MON, materials with good oxidation resistance compared to the more conventional but corrosion/oxidation prone Re-Tm alloy films Substitution of bismuth ion and rare earth ions like Gd, Ce, Pr, Nd, Tb, Lu, Tin, etc are known to enhance the MO activity in rare earth garnets. We report here the magnetic, magneto-optic, (MO) and crystalline properties of Bi- substituted Lutetium, Thulium, Cerium Iron Garnet films (of the type Bi:LuIG, Bi:TmIG, Ce:YIG) epitaxially grown on single crystal Gadolinium Gallium Garnet (GGG) and compare with other substituted rare earth garnet films.

10:10 AM Invited

Melting of Nano-Embedded Particle: The Role of Interface Structure: *Ramasis Goswami*[1]; *David Welch*[2]; *Kamanio Chattopadhyay*[3]; [1]SUNY at Stony Brook, Matls. Sci. & Eng., Stony Brook, NY 11794 USA; [2]Brookhaven National Laboratory, Energy Scis. & Tech. Dept., Upton, NY USA; [3]Indian Institute of Science, Dept. of Metall., Bangalore, Karnataka 560012 India

Melting of nanosized embedded particles have drawn considerable attention in recent time. A considerable amount of superheating is reported in nanoparticles embedded in a matrix. A large superheating of approx. 470K was observed for nanocrystals of Ar embedded in an Al matrix, where the amplitude of vibration of atoms is reported to be depressed at the solid-solid interface. The superheating of nanocrystals of Pb embedded in Zn is attributed to the change in the morphology of the particle which modifies the particle-matrix interface. The presence of epitaxy of closed-packed planes and directions can promote the condition for superheating. The present work reports on the existence of steps at the interface on melting of the nanosized-embedded Bi and Pd particles in different matrices. Experiments have also been carried out to study the influence of the defects like dislocations, introduced at the particle-matrix interface by deformation, on melting. Acknowledgements : We acknowledge the Alexander von Humboldt Foundation and the US Department of Energy, under contact No. DE-AC02-98CH 10 886 of the Energy Sciences and Technology Department, Brookhaven National Laboratory, Upton, NY.

10:35 AM Invited

Interfaces in Soft Materials: *R. Shashidhar*[1]; [1]Naval Research Laboratory, Code 6900, Washington, DC 20375 USA

Soft condensed matter materials like liquid crystals and polymers exhibit unique properties in their thin film state. These properties depend very strongly on the interaction of the materials with surfaces. An example if such interfacial interaction between liquid crystal and solid surface is the ability to orient microscopic liquid crystals using an extremely thin and tailored interface. This talk presents the development of a self-assembling monolayer which, when fabricated on a solid surface, dictates the orientation of liquid crystal molecules, the relation between the interfacial energy, the chemical anisotropy and the properties of liquid crystals. This talk also discusses the prospects of using liquid crystals for imaging the structure of soft interfaces, particularly bio-interfaces.

11:00 AM Invited
Interface Studies in Quasicrystalline AlñCuñFe and AlñPdñMn Alloys: R. Divakar[1]; V. S. Raghunathan[1]; *S. Ranganathan*[2]; [1]Materials Characterisation Group, Indira Gandhi Ctr. for Atomic Rsrch., Kalpakkam 603102 India; [2]Centre for Advanced Study, Dept. of Metall., Indian Inst. of Sci., Bangalore 560012 India

The structural characterization of interfaces in quasicrystalline systems has been of interest for some time now. Characterization of interfaces in these materials is of particular interest with respect to the known structural relations between the quasicrystalline phases and the coexisting crystalline phases. High-resolution transmission electron microscopy (HREM) has been a technique of choice for these studies. In the present paper, the status of experimental results on the structure of interfaces in quasicrystalline systems is reviewed. In addition, our results of the experimental investigation of interfaces in AlñCuñFe and AlñPdñMn are systems are reported. In both systems, coexisting crystalline phases are found that are structurally related to the icosahedral quasicrystals. Thus, in addition to the variability of interfaces with respect to structure, composition and orientation of the grains, translational order also change across an interface. Two classes of interfaces have been studied in these systems. The first is the interface between icosahedral phase grains. In Al-Cu-Fe, quasicrystalñquasicrystal interfaces show a high degree of curvature with frequent, irregularly spaced ledges that are atomically flat with little disorder. For the second class of interfaces, similar interfacial structures have been observed at interfaces between the icosahedral phase and the related crystalline phase where the interfacial plane corresponds to the common symmetry axis shared by the two phases, in both AlñCuñFe and AlñPdñMn systems. The observed special features of grain boundaries and ledge interfaces are discussed in terms of common structural motifs and the resulting orientation relation between the quasicrystalline and related crystalline phases.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Mechanical Properties, Fatigue and Creep

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

Wednesday AM	Room: 612
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Sungho Jin, Bell Labs, Agere, 600 Mountain Ave., Murray Hill, NJ 07974 USA; Carol Handwerker, NIST, Metall., Gaithersburg, MD USA

8:30 AM Invited
A Constitutive Model for Predicting Thermal Mechanical Fatigue in Low-Temperature, In-Sn Solder Interconnects: *Paul T. Vianco*[1]; Jerome Rejent[1]; Arlo Fossum[1]; Michael Neilsen[1]; [1]Sandia National Laboratories, PO Box 5800, MS 0889, Albuquerque, NM 87185-0889 USA

The interest in low-temperature, In-based solders has stemmed satellite/space applications as well as cryogenic service. A study was performed to develop the constitutive model for the Pb-free solder, 52In-48Sn (wt.%). Compression stress-strain tests determined the yield stress and static elastic modulus values at -25∞C, 25∞C, 75∞C, and 100∞C. Yield stress values ranged from 17 MPa at -25∞C to 1.32 MPa at 100∞C. The static elastic modulus ranged from 1.6 GPa (-25∞C) to 0.3 GPa (100∞C). Compression creep behavior exhibited an apparent activation energy of 55 kJ/mol and stress term exponent of 1.7. Sandia is a multiprogram laboratory operated by Sandia Corporation, A Lockheed Martin Company, for the US Dept. of Energy under Contract DE-AC04-94AL85000.

8:55 AM
Low Cycle Fatigue Life of Lead-Free CSP/PWB Interconnects: *Yoshiharu Kariya*[1]; Tomoo Morihata[1]; Yasunori Tanaka[2]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Matls. Sci. & Eng., 3-9-14 Shibaura, Minato-Ku, Tokyo 1088548 Japan; [2]NEC Corporation, Mobile Terminal Div., Ikebe-cho, Tsuzuki-Ku, Yokohama, Kanagawa 224 Japan

With increasing of the production volume of fine pitch BGA type packages such as CSP, a demand on the establishment of the reliability evaluation for BGA/PWB (Printed Wiring Board) joints is increasing. Especially, the shear fatigue properties of the joints made up using lead-free solders are great interest. However, the shear fatigue data of actual BGA joints very limited, as the fatigue test requires fine displacement or load control ability of the test machine. In this study, the straddle board fatigue test using a conventional servo-valve-controlled electro-hydraulic testing machine has been performed to look for strain-life relationship of real lead-free CSP/PWB interconnections. The fatigue life was correlated to inelastic displacement divided from hysteresis loop by a pseudo Coffin-Mansonís law. The difference in the fatigue lives of 0.8mm pitch and 0.5mm pitch CSP will be presented. The comparison between the bulk data and the joint data also will be presented.

9:15 AM
Modeling Thermomechanical Fatigue Behavior of Sn-Ag Solder Joints: *J. Lee*[1]; H. Rhee[1]; K. N. Subramanian[1]; T. R. Bieler[1]; J. P. Lucas[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing 48824-1226 USA

Stresses that develop due to CTE mismatches between the solder/substrate/components contribute to the TMF of solder joints. However, the relative importance of the several processes that contribute to the observed damage accumulation and their role in affecting the reliability of the solder joint are far from being understood. Processes such as aging, creep/stress relaxation, and stress reversals are some of the important processes. These factors are affected by service conditions such as the temperature extremes experienced, rates of heating/cooling, dwell times at the extreme temperatures, etc. These preliminary efforts to model TMF in Sn-Ag solder joints will focus on the findings of the role of each of these parameters and their inter-relationships. Acknowledgment: This project is funded by National Science Foundation under grant NSF-DMR-0081796.

9:35 AM
The Vibration Fracture Characteristics of Sn-Ag Lead-Free Solders: *Chiang Ming Chuang*[1]; [1]National Cheng Kung University, Matls. Sci. & Eng., Tainan 701 Taiwan

The research has two aspects; one is the different solidification rate, putting Sn-3.5wt%Ag alloy in different cooling-rate mold. And other is variation of Ag content from 3.5 to 6.0wt%. The experimental results show that the vibration-fracture resistance of lower cooling-rate and higher Ag content specimens is better than higher cooling-rate and lower Ag content specimens. Comparing to near eutectic Sn-Pb solder, Sn-Ag samples have better vibration properties for they possesses a smaller initial deflection amplitude under the same vibration push force and higher crack propagation resistance under the same initial deflection amplitude. Differing vibration-fracture resistance could be correlated with differing crack-propagation behavior.

9:55 AM
High Temperature Mechanical Properties of the Lead-Free Sn-3.5Ag-xCu Alloys: *S. W. Shin*[1]; D. K. Joo[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls., 373-1 Kusong-dong, Yusong-gu, Daejon 305-701 Korea

In the electronics industry worldwide, using lead-free solders is becoming more mandatory recently, and Sn-3.5Ag based ternary alloys with Cu are strong candidate materials. On that regard, substantial amounts of work on various properties of Sn-3.5Ag based alloys are under way. However, high temperature mechanical creep data of the Sn-3.5Ag alloys, including the effects of the solder microstructure, cooling rate and ball size, are rather rare in literature. In the present work, Sn-3.5Ag-xCu alloys with five different levels of Cu (0, 0.5, 0.75, 1.0, 1.5wt%) and a Sn-0.7Cu alloy were prepared. Conventional creep tests using rolled and stabilized bulk specimens were conducted in order to understand the creep mechanism of the alloys under the stable microstructure, while lap shear tests with reflowed solder balls of varying size were conducted to simulate the creep deformation of solders under the real package situation. The latter usually involves the effects of the intermetallic formation at the solder/die pad interface. Since the creep deformation depends on the microstructure of the reflowed solder which varies with the ball size, as-cast bulk specimens with the same microstructure as the reflowed solder balls of varying size were prepared by controlling the cooling rate, and their creep behaviors were compared with those of the lap shear and the as-stabilized bulk specimens. Result show that the creep strength were best with 0.75~1wt% Cu and the creep exponent was ~4 with the nucleation and growth of creep voids at the grain interior. Results also indicate substantial differences among creep behaviors of the as-stabilized bulk, reflowed solder, and as-cast bulk with the microstructure of the reflowed solder specimens.

10:15 AM Break

10:30 AM Invited

Damage Accumulation under Repeated Reverse Stressing of Sn-Ag Solder Joints: *K. C. Chen*[1]; K. N. Subramanian[2]; [1]California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA; [2]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

Sn-Ag solder joints were subjected to repeated reversed stress under constant strain amplitude conditions. Effects of strain amplitude, strain rate, hold times at the stress extremes, solder joint thickness, and temperature were investigated. The role of constraints present in the joint on the Bauschinger effect and on damage accumulation was also evaluated. Microscopic studies indicate significant amount of damage arising from grain boundary sliding and grain separation (decohesion) as a result of repeated stress. Implications of the findings from this study to electronic solder joints will be presented. Acknowledgment: The authors thank the National Science Foundation for providing financial support for this project as additional funding to NSF grant NSF-DMR-0081796.

10:55 AM

A High Creep Resistance Tin-Based Alloy for Soldering Applications: *Rodney J. McCabe*[1]; Morris E. Fine[2]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA; [2]Northwestern University, Matl. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Soldering applications such as optical interconnects require dimensionally stable solders because any creep deformation may result in device degradation or failure. Using comparatively simple processing steps, tin-based alloys strengthened by the precipitate SbSn were found to possess excellent creep resistance below 100∞C compared to other soldering alloys. The creep properties of these alloys were found to be relatively unaffected by aging at temperatures up to 100∞C for as long as 8 weeks. These alloys owe their strength to an advantageous array of high aspect ratio SbSn whiskers crisscrossing in five crystallographic directions. This configuration results in a composite strengthening effect that is a function of particle size, spacing, and volume fraction.

11:15 AM

Effect of Cooling Rate on the Tensile Behavior of Pb-Free Solders: *Felipe Ochoa*[1]; George Piotrowski[1]; Jason Williams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85284 USA

The mechanical behavior and microstructure of bulk, pure solders as a function of cooling rate was studied. Scanning and transmission electron microscopy were used to characterize the effect of cooling rate on solder microstructure. Thermal aging studies on solder joints, also as a function of cooling rate, were conducted and the evolution of intermetallic growth was characterized. Solder fracture surfaces were charaterized to elucidate the synergistic relationship between mechanical behavior and microstructure. Research sponsored by the National Science Foundation under contract# DMR-0092530 (Dr. K. L. Murty, Program Manager).

11:35 AM

Creep Behavior of Sintered Solder Joints: *Mark A. Palmer*[1]; Nicole S. Erdman[1]; [1]Kettering University, IMEB Dept., 1700 W. Third Ave., Flint, MI 48504-4989 USA

Low-flux lead-free composite solder joints have been shown to have mechanical properties comparable to reflowed eutectic tin-lead solder joints. However, before such materials can be fully considered as a low-temperature processing alternative the creep behavior of such materials must be understood. The creep behavior of composite Sn-Ag-Bi and Sn-Ag-Cu will be evaluated at high fractions of the measured yield stress and high fractions of the sintering temperature. This work is funded by the National Science Foundationís Environmentally Benign Manufacturing Program.

11:55 AM

Creep and Microstructure of Lead-Free Solders: *Vladimir Igoshev*[1]; Jacob Kleiman[2]; [1]Research in Motion, 295 Phillip St., Waterloo, ON N2L 3W8 Canada; [2]Integrity Testing Laboratory, 80 Esna Park Dr., Units 7-9, Markham L3R 2H7 Canada

A critical review of data on activation energy of creep for three lead-free solders is presented. The review indicates that a large scatter exists in the data. In order to explain the discrepancy, an assumption was made that such fracture processes as nucleation, accommodation and propagation of pores/cracks take place in an earlier stage of creep. These processes will play a significant role during creep deformation of lead-free solders and should be taken into consideration to calculate the value of true, not apparent activation energy of creep. Creep experiments conducted on bulk Sn-3.5%Ag solder alloy and solder coupons (Cu coupons soldered with the alloy) confirmed that nucleation of cracks in the solder occurs after a testing time of $t = 0.2t_c$, where t_c is the specimen lifetime.

Magnesium Technology 2002: Magnesium Wrought Products–A Session in Memory of William A. Barnes

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

Wednesday AM	Room: 606
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Darryl Albright, Hydro Magnesium, Magnesium Mkt. Dvlp., 39209 Six Mile Rd., Ste. 200, Livonia, MI 48152 USA; Henry J. Rack, Clemson University, Sch. of Cheml. & Matls. Sci., 208 Rhodes Hall, Clemson, SC 29634-0921 USA

8:30 AM Opening Remarks
William A. BarnesñA Dedication

8:40 AM Invited

Magnesium Wrought and Fabricated ProductsñYesterday, Today, and Tomorrow: *Robert Brown*[1]; [1]Magnesium Monthly Review

The developers of magnesium metal production were always interested in methods of producing alloys and products. Casting was the first major development, but it was closely followed by rolling, forging and extrusion. This paper will review the tremendous historical developments in wrought products during the 1930ís in Germany and during the 1940ís and after in the US and Great Britain. At the peak of wartime production, thousands of tons of magnesium sheet were produced each month and used in aircraft in many areas. Aircraft of all magnesium, extrusions and sheet and castings were made by the Germans (Arado 196) and the US, XP56 and F-80. The US produced B-36 had 19,000 pounds on each aircraft, including a large amount of sheet for the pressurized fuselage. Most of the magnesium sheet were produced on rolling mills developed for other metals until Dow built the large magnesium fabrication facility at Madison, Illinois. (Owned and operated by Spectrulite). In the 1950ís, Brooks and Perkins began to develop magnesium rolling and eventually had two rolling mills. They also were instrumental in building the primary magnesium reduction plant in Selma, Alabama to reduce their dependence on Dow for magnesium. The rapid expansion and development of rolling and forging will be discussed and contrasted to the present day experiments and testing. Included will be a discussion of the twin roll casting operations proposed by Magwerkes in 1988-89.

9:10 AM
Relation between Microstructure and Mechanical Properties in Magnesium Wrought Alloy AZ31: *Dietmar Letzig*[1]; Jan Bohlen[1]; Heinz-G¸nther Brokmeier[2]; Andrzej Styczynski[3]; Christian Hartig[3]; Karl-Ulrich Kainer[1]; [1]GKSS Forschungszentrum GmbH, Zentrum f¸r Magnesiumtechnologie, Max Planck Str., Geesthacht 21502 Germany; [2]TU Clausthal, GKSS-Hochschulgruppe, Max-Planck-Str., Geesthacht 21502 Germany; [3]TU Hamburg Harburg, Werkstoffphysik/Technologie, Eiflendorfer Str. 42, Hamburg 21073 Germany

Magnesium wrought alloys as light weight materials offer a high potential for industrial structural applications. However, due to the basic hexagonal lattice structure magnesium alloys show poor mechanical properties and formability. In order to improve the mechanical properties the influence of the microstructure on the deformation behaviour is of fundamental interest. The processing of wrought materials itself influences the microstructure and thus the mechanical properties. The relation between process parameters and microstructure is therefore of interest for the development and improvement of industrial applicable magnesium wrought alloys. Using alloy AZ31 the process conditions of extruded bars and rolled sheets are characterized and compared in order to determine the effect of the microstructure on deformation mechanisms. Microstructural evaluations, texture measurements and mechanical properties are presented.

9:35 AM
Plastic Anisotropy in Magnesium Alloy AZ31B Sheet: *Sean R. Agnew*[1]; [1]University of Virginia, En-Matl. Sci./Eng. Dept., PO Box 400745, Thornton Hall, B120, Charlottesville, VA 22904-4745 USA

It is well established that crystallographic texture induced plastic anisotropy in sheet metal can give rise to a variety of formability problems. The earing behavior of deep drawn aluminum beverage cans is a classical example. Interest in the application of lightweight magnesium alloys has been growing over the past decade. One problem facing the application of wrought magnesium alloys, in particular, is their poor cold forming properties. Historically, this has been linked with an inadequate number of independent slip systems within magnesiumís hexagonal crystal lattice. An issue that has received less attention is the impact of the strong crystallographic texture induced during primary forming operations, such as rolling, upon subsequent forming steps, such as sheet stamping. Measurements of the R-value for 0, 45, and 90∞ oriented tensile samples have highlighted a strong in-plane anisotropy, R(0∞)~1 and R(90∞)~4. Simulations using the viscoplastic self-consistent model have been used to link crystallite level plastic anisotropy with the macroscopic properties. This information may be used to explain the poor sheet forming behavior of magnesium alloys and offer insights for potential solutions to the problem.

10:00 AM
Elevated Temperature Behavior of Sheet Magnesium Alloys: *Paul E. Krajewski*[1]; [1]General Motors Corporation, R&D Ctr., 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA

The mechanical behavior of three commercially available magnesium sheet materials was investigated at temperatures between 300∫C and 500∫C. The three materials, AZ31B-O, AZ31B-H24, and AZ61A-O, all showed significant improvements in total elongation at strain rates between 0.001/sec and 0.01/sec. AZ31B-H24 exhibited a total elongation of over 400% at temperatures above 450∫C. The dominant failure mechanism underwent a transition from cavitation controlled fracture, to necking controlled fracture in this temperature/strain rate regime. The transition from cavitation to necking dominated failure was evaluated microstructurally and the results were related to total elongation.

10:25 AM Break

10:45 AM
Grain Refinement under Warm Deformation of Magnesium Wrought Alloy ZK60: R. Kaibyshev[1]; *A. Galiyev*[1]; G. Gottstein[2]; [1]Institute for Metals Superplasticity Problems, Khalturina 39, Ufa 450001 Russia; [2]Institut fur Metallkunde und Metallphysik, RWTH Aachen, Aachen D-52056 Germany

The possibility of producing an ultrafine-grained structure in magnesium alloy was considered. The coarse-grained Mg-5.8%Zn-0.65%Zr alloy was examined. Compression tests were carried out in air at a temperature of 423K and at a fixed strain rate of $2.8x10^{-3}$ s^{-1}. It was shown that formation of new grains took place during warm deformation. Almost full-recrystallized structure with an average grain size of 0.8μm was evolved after large cumulative strain. The main feature of recrystallized structure was highly non-equilibrium grain boundaries, which were sources of internal long-range stress fields. It was suggested that the structural changes were connected with operation of a specific type of dynamic recrystallization defined as low temperature dynamic recrystallization (LTDRX). The mechanism of ultrafine-grained structure formation under warm deformation was discussed in terms of LTDRX.

11:10 AM
Direct and Indirect Extrusion of AZ 31: *Klaus B. M¸ller*[1]; [1]Technische Universit‰t Berlin, Forschungszentrum Strangpressen, Gustav-Meyer-Allee 25, Sekr.TIB 4/1-2, Berlin D-13355 Germany

The paper describes the results of direct and indirect extrusion of the magnesium alloy AZ 31. The trials were carried out on the 8 MN horizontal extrusion press of the r&d center of extrusion. Both extrusion procedures, direct and indirect, are possible. The press unit is equipped with load cells to record the total force F(T), friction force F(F) and die force F(D), whereby friction force F(F) only occurs during direct extrusion. Programmable logical controllers offer a broad margin for development. By means of a computer-aided measuring system, forces can be determined in relation to the ram displacement. The important advantage of indirect extrusion is the absence of friction between billet and container. This ensures a largely homogenous flow of the material and an important decrease of required total extrusion force. Trials were done by different temperatures and extrusion speeds.

11:35 AM
The Hot Working Flow Stress and Microstructural Evolution of Wrought Magnesium Alloy AZ31: *Aiden G. Beer*[1]; Matthew R. Barnett[1]; [1]CRC for Cast Metals Manufacturing (CAST), Deakin University, Sch. of Eng. & Tech., Pigdons Rd., Geelong, Victoria 3217 Australia

The evolution of hot working flow stress and microstructure for wrought magnesium alloy AZ31 was characterised. Torsion and compression testing, at temperatures between room temperature and 450°C and strain rates between and 0.01s-1 to 1s-1, was employed to determine the influence that these variables have on flow stress and microstructural development. Constitutive equations were developed for the peak and steady-state flow stresses. Optical observations revealed twinning and dynamically recrystallised (DRX) grains. The latter were observed at test temperatures of 200°C and above. The influence of strain rate and temperature on the dynamically recrystallised grain size was quantified for samples that displayed more than 25% dynamic recrystallisation. The flow stress at low strains was found to be strongly dependent on texture and deformation mode. This can be described in terms of the influence of the deformation accommodating processes of prismatic slip, twinning and DRX. At higher strains, the flow stress was relatively insensitive to deformation mode and initial texture.

Materials Processing Fundamentals - III

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

Wednesday AM Room: 614
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Ramana G. Reddy, University of Alabama, Metall. & Matls. Eng., A129 Bevil Bldg., 126 7th St., Tuscaloosa, AL 35487-0202 USA; Patrick R. Taylor, University of Tennessee, Metlgcl. Eng., 1010 W. Cayuse Dr., Knoxville, TN 37996 USA

8:30 AM
Computer Modelling of Rotary Furnaces: A Lumped Parameter Approach: *Ian Masters*[1]; [1]University of Wales-Swansea, Mechl. Eng., Singleton Park, Swansea SA2 8PP UK

Rotary furnaces are widely used throughout the metals industry. These furnaces vary in size and geometry, but are fundamentally the same from a thermodynamic viewpoint. A generic method is proposed that can be used to model all furnaces of this type, provided that the correct material and process parameters are known. Previous work has been based on a continuum (FEA or CFD) approach. However, the complex fluid/solid interactions present in a rotary vessel are not easily solved using these methods, which work best where the material boundaries are well defined. In this paper a lumped parameter model is proposed. Variations within a section of the model are ignored, but to ensure thermodynamic correctness, the heat transfer between sections is carefully considered. For example, it is reasonable to assume that the whole of the melt pool is at the same temperature, due to rotary mixing. This may then be modelled with a single set of parameters, and the detail is contained in obtaining the correct heat transfer mechanisms from the rest of the furnace.

9:00 AM
Investigation of the Al Ball Plug/319 Al Casting Hole Press-Fit System: W. Kasprzak[1]; J. H. Sokolowski[1]; *D. Szablewski*[1]; [1]NSERC/Ford-Nemak/University of Windsor, Mech., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, ON N9B 3P4 Canada

Press-fit systems are widely used to seal oil lines in automotive engine components. In the case studied a ball plug made from an Al alloy was inserted into a 319 Al alloy cast component. The oil lines were subjected to the required component service pressure. This paper addresses the analysis of the ball plug/casting hole press-fit performance. The tools used to investigate the plug system included: SEM/EDS technique for the assessment of the plug and casting hole wall surface integrity, Image Analysis methods for the characterization of the ball plug/casting hole interface, matrix microhardness measurements of the plug and casting components, laboratory ball plug insertion and pressure tightness tests, as well as identification of the leaking mechanisms present in the system. Laboratory tests indicated that optimization of the aluminum matrix hardness in the 319 Al alloy cast component resulted in considerable improvement of the sealing properties.

9:30 AM
Evaluation of Field Activated Sintering of Silicon: *Martin Kraemer*[1]; Joanna R. Groza[1]; [1]University of California, Chem. Eng./Matl. Sci. Dept., One Shields Ave., Davis, CA 95616 USA

A kinetic study on sintering pure, fine grained silicon powder by field activated sintering using the Sumitomo SPS sintering system has been carried out. Densities better than 97% could be achieved by tailoring temperature and pressure profiles. Densification kinetics as a function of different heating rates and uniaxial pressures have been determined to derive experimental values for activation energies. The results are discussed in terms of kinetics and the technical capabilities and potential of the sintering system addressing both problems of temperature distribution throughout the specimen and furnace setup which have been studied by finite element modeling and experiments.

10:00 AM Break

10:30 AM
Rotating Disk Studies of Iron Reactions in Water under Abiotic and Biotic Conditions: *Batric Pesic*[1]; [1]University of Idaho, Matls. Sci. & Eng., COMER-McClure, Moscow, ID 83844-3024 USA

The iron reactions with water were examined under abiotic and biotic conditions by using pure iron in a rotating disk technique. The biotic conditions were produced by utilizing harvested cells of Thiobacillus ferrooxidans. The parameters studied were: pH, rotation speed, bacterial cells concentration, and concentration of oxygen. The reactions of iron were studied by measuring the rate of iron dissolution and by electrochemical techniques. It was found that iron reactions with water were mostly independent of all parameters studied except of the concentration of oxygen. In addition, the iron dissolution data and the results from electrochemical experimentation will also be correlated to the morphology of surface reaction products, which was done by atomic force microscopy.

11:00 AM
Magnetic Properties of Undercooled Co-Based Alloys Measured by Means of a Faraday-Balance: *Sven Reutzel*[1]; Dieter M. Herlach[1]; [1]German Aerospace Center, Inst. of Space Simulation, Linder Hoehe, Cologne 51170 Germany

Investigations show that Co-based melts embedded by glass flux in a crucible can be undercooled nearly as strong as samples processed containerlessly by electromagnetic levitation. This allows to measure magnetic susceptibility on undercooled melts while combining the faraday method with undercooling technique. The magnetic susceptibility of Co and Co-based alloys (such as Co-Cu, Co-Pd, Co-Au) is analysed as a function of temperature from superheated to undercooled state. This is done by measuring the change of magnetic force FZ on a sample in a constant magnetic field B0 and an additional gradient field by means of a faraday-balance. When liquid Co-based alloys are undercooled the magnetisation steeply rises if temperature approaches the Curie-temperature. The magnetisation is measured for some Co-based alloys in liquid state as function of alloy system and its concentration. The results show that the magnetic susceptibility of the liquid undercooled samples follows a Curie-Weiss behaviour, from which Curie-temperature and magnetic moments are inferred.

11:30 AM
Potentiostatic Leaching Kinetics of Pyrite/Marcasite in Sterile Acid Ferric Sulfate Media: *Sylvie C. Bouffard*[1]; David G. Dixon[1]; [1]The University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Abstract unavailable

12:00 PM
The Effect of Tap Water and Selected Anions on the Corrosion of Copper: *M. A. Llavona*[1]; B. Diaz[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 Engineering, Reinerio Garcia s/n, 33600, Mieres Spain

For several decades, numerous researchers have tried to artificially prepare Patinas in different metals, using diverse procedures. In some of these, immersion methods were proposed, controlling the type of solution, concentration, pH, temperature and time. In other procedures, different types of paints or varnishes were applied. The development of an artificial process to create copper Patina supposes an important success on the market as it is a product which is being demanded by the promoters because of both the added value and the aesthetic outlook of the buildings.

Materials & Processes for Submicron Technologies - II: Materials & Processes for Microelectronic Devices

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

Wednesday AM — Room: 209
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mahesh K. Sanganeria, Novellus Systems; Sailesh M. Merchant, Agere Systems, VLSI Process Dvlp.; Nuggehalli M. Ravindra, New Jersey Institute of Technology, Dept. of Phys., 161 Warren St., Newark, NJ 07102-1982 USA

8:30 AM Invited
Recent Developments in Rapid Thermal Processing: *Anthony T. Fiory*[1]; [1]New Jersey Institute of Technology, Dept. of Phys., Newark, NJ 07012 USA

Complementary metal-oxide semiconductor Si processing uses rapid thermal annealing with a short dwell time at maximum temperature to reduce the diffusion of implanted dopants in the formation of shallow junctions and for high electrical activation of dopants in polycrystalline Si gate electrodes. Advanced methods use ìspike anneals,î wherein high temperature-ramp rates are used for both heating and cooling, while also minimizing the dwell time at peak temperature to nominally zero. The fast thermal cycles are required to reduce the undesirable effects of transient enhanced diffusion and thermal deactivation of the dopants. Original methods for single-wafer heating by incandescent or electric arc lamps, or by a steady heat source with rapid wafer transfer, have been refined for mass production of integrated circuits. Since junction profiles are sensitive to annealing temperature, the challenge in spike annealing is to maintain temperature uniformity across the wafer and repeatability from wafer to wafer. Multiple lamp systems use arrayed temperature sensors for individual control zones. Other methods rely on process chambers that are designed for uniform wafer heating. Generally, sophisticated techniques for accurate temperature measurement and control by emissivity-compensated infrared pyrometry are required, since processed Si wafers exhibit appreciable variation in emissivity.

9:00 AM
Integration Issues in the Development of Poly-Metal(W/WN/Poly Silicon) Gate using 0.15μm DRAM Technology: *Jun Ho Choy*[1]; Byung Hak Lee[1]; Chang Ki Kwan[1]; Jae Hyung Kim[1]; Tae Keun Hwang[1]; Yeong Cheol Kim[2]; Joo Chul Park[1]; [1]Hynix Semiconductor, Inc., R&D Div., 1 Hyangjeong-dong, Hungduk-gu, Cheongju, Chungbuk

361-725 Korea; [2]Korea University of Technology and Education, Dept. of Matls. Eng., Chonan, Chungnam Korea

Poly-metal(W/WN/poly silicon) gate process has been successfully developed and integrated using 0.15μm technology for the first time, in an effort to reduce RC delay for giga bit DRAMs. This paper summarizes the problems in the process incompatibility with respect to the conventional polycide(WSix/poly silicon) gate process, device and circuit performances, and the product performance of the fully working 256Mbit DRAM samples employing poly-metal gate. In the integration, the main focus was on the search of a new cleaning chemical for post gate etch process, which is directly related to tungsten contamination. The AC characterization of the circuit revealed the anomalous decay of an electrical signal as it proceeds through the inverter delays. The phenomenon may be explained by the presence of parasitic capacitance in the gate electrode. A solution to this problem was found by experiments employing several different deposition schemes for tungstgen and poly-silicon. The physical characterization on this problem is also addressed. Finally, the examination on the data retention characteristics shows a result quite comparable to that obtained from the samples using conventional polycide gate.

9:20 AM Invited
An Overview of Interdiffusion Phenomena in Ternary Systemsñ Unusual Diffusion Paths and Diffusion Structures: *M. A. Dayananda*[1]; [1]Purdue University, Sch. of Matls. Eng., W. Lafayette, IN 47907 USA

Interdiffusion in multicomponent systems is reviewed with emphasis on the determination of interdiffusion fluxes and assessment of diffusional and thermodynamic interactions among the diffusants. Selected diffusion phenomena such as uphill diffusion and development of zero-flux planes for individual components are illustrated with specific examples in selected ternary metallic and intermetallic phases and silicide systems. Unusual diffusion structures and diffusion paths experimentally observed for single phase and multiphase diffusion couples in Cu-based and Fe-based ternary systems as well as in selected ternary aluminides and silicides are presented and discussed in the light of uphill diffusion, zero-flux planes and flux reversals, demixing of phases, and variation of diffusion coefficients with composition.

9:50 AM
Transient Enhanced Diffusion in Shallow Junction Formation: *Anthony T. Fiory*[1]; Samrat Chawda[1]; Sridhar Madishetty[1]; Vishal Mehta[1]; N. M. Ravindra[1]; [1]New Jersey Institute of Technology, Dept. of Phys., Newark, NJ 07102 USA

Shallow junctions are formed in silicon for CMOS devices by low-energy ion implantation of B, P, or As species and spike thermal annealing. Boron enhanced transient diffusion, which occurs at high surface concentrations of B, is associated with an excess population of Si interstitials. Rapid thermal annealing with ramp rates of 100 C/s or greater, and nominally zero dwell time at the maximum temperature, denoted as spike annealing, is shown to improve electrical activation while minimizing the diffusion. A similar transient diffusion also occurs in the case of P implantation, where modeling by coupled P-Si diffusion indicates an excess Si-interstitial population of 6 to 10% of the implanted P dose. The electrically-activated fraction of implanted dopant depends mainly on temperature for B species, while for P species it depends on both temperature and P dose. The relatively small diffusion associated with As implants is favorable for shallow junction formation with spike annealing.

10:10 AM Break

10:30 AM
Copper/Low-k Metallization for Semiconductor Devices: *Sailesh M. Merchant*[1]; Seung H. Kang[1]; Mahesh Sanganeria[2]; Bart van Schravendijk[2]; Tom Mountsierb[2]; [1]Agere Systems, VLSI Proc. Dvlp., 9333 S. John Young Parkway, Orlando, FL 32819 USA; [2]Novellus Systems, 3970 N. First St., San Jose, CA 95134 USA

Copper/low-k dielectric materials have been rapidly replacing conventional Al-alloy/SiO2-based interconnects for the fabrication of todayís semiconductor devices. This paper reviews the advantages of transitioning to Cu/low-k interconnects. Materials and process challenges during the fabrication of devices with Cu/low-k interconnects are discussed. Reliability concerns associated with such devices are highlighted.

10:50 AM Invited
Low-k Materials Integration Challenges in High Performance Interconnects: *Andrew J. McKerrow*[1]; [1]Interconnect Process, Equipment & Materials, Silicon Tech. Rsrch., Texas Instruments Inc., 13570 N. Central Expressway, MS 3700, Dallas, TX 75243 USA

Abstract unavailable

11:20 AM
Computer Controlled Reactive Ion Plasma Dry Etching of MRAM Stacks: *Todd Scott*[1]; R. B. Young[1]; Keith Prisbrey[1]; [1]University of Idaho, Matls. & Metlgcl. Eng., Moscow, ID 83844-3024 USA

MRAM technology is the next step in computer chip manufacturing. Effectively etching the material without degrading the magnetic properties is a complicated process. 2-micron diameter holes were dry etched in a grid pattern in a Fe/Co/Cu thin film deposited on a silicon wafer. The vertically and undercutting of the holes were measured under SEM, AFM and optical microscopes. The stackís magnetic properties are then characterized. Computer control was necessary to alter the operating parameters online during etching as the reaction progressed through different layers. Spectrometers, electrical and magnetic field measurements, langmur probe, and mass flow controllers allowed continuous online computer control. The control system is based on multiphysics models using partial differential equations. This allows the etching to progress under optimal conditions for each layer.

11:40 AM Invited
Si Solar Cell Processing for Minimizing the Influence of Impurities and Defects: *Bhushan Sopori*[1]; [1]National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA

Commercial silicon solar cells are fabricated on low-cost substrates that contain high concentrations of impurities and defects. To achieve high device efficiency, it is necessary to getter the impurities and to passivate the residual impurities and defects through hydrogenation. However, to maintain the cost-effectiveness, these processes must be included as a part of a typical cell fabrication sequence without increasing the number of process steps. This paper will review various approaches used to optimize impurity gettering and defect passivation in photovoltaic device fabrication. We will discuss the influence of each device process step on the changes in the impurity concentrations and the minority-carrier lifetime (or diffusion length), and discuss how defect passivation can be made a part of an efficient device fabrication schedule.

Modeling of High Temperature Alloy Processing - I

Sponsored by: Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Matthew Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907 USA; Suhas P. Vaze, Concurrent Technologies Corporation, Johnstown, PA 15904 USA

Wednesday AM — Room: 310
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Suhas P. Vaze, Concurrent Technologies Corporation, 1450 Scalp Ave., Johnstown, PA 15904 USA; Matthew John M. Krane, Purdue University, Dept. of Matls. Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA

8:30 AM
The Use of 2D and 3D Finite Element Analysis to Simulate Evolution of Nickel Base Superalloy Billet Structure and Inspectability: *Bruce Fergus Antolovich*[1]; Bruce Andrew Lindsley[1]; Michael William Hill[1]; Howard William Sizek[2]; [1]Special Metals Corporation, 4317 Middle Settlement Rd., New Hartford, NY 13413 USA; [2]Special Metals Corporation, 100 Willowbrook Ave., Dunkirk, NY 14048 USA

Billets of nickel base superalloy intended for eventual use in rotating parts of modern turbine jets are subject to intense inspectability, grain size and mechanical property requirements. The conversion process from ingot structure to billet structure plays a crucial role in achieving these requirements. Cogging consists primarily of drawing operations in which the material is repeatedly deformed in a direction perpendicular to its longitudinal axis. Recent advances in numerical analysis capabilities have enabled the switch from 2 dimensional to 3 dimensional analyses for cogging. Direct prediction of grain size evolution during cogging is now possible and has been demonstrated. 3D finite element analysis has been used to better understand the role cogging plays in ultrasonic inspectability. Previous efforts in 2D have been very misleading due to previously unrecognized, but significant, interaction effects in the third dimension. Efforts using 3D modeling have allowed the better understanding of the development of billet structure and ultrasonic inspectability. This better understanding has in turn allowed for novel processing routes for improved structure and inspectability. The impact of forging route and use of 3D FEA upon ultrasonic inspectability of nickel base superalloy billets is discussed.

9:00 AM
Simulation of Directional Annealing: *I. Baker*[1]; J. Li[1]; A. Badmos[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA

A front-tracking, capillarity-driven, grain-growth model has been modified to simulate microstructural development during directional annealing. Preliminary simulations have been conducted for an infinite temperature gradient with a hot zone of uniform temperature moving along the length of the sample. The effects of the initial microstructure, hot zone velocity, hot zone width and grain boundary mobility have been investigated. A columnar grain structure is shown to form more easily with a finer initial grain structure and within a certain hot zone velocity range which depends on the grain boundary mobility, i.e. the hot zone peak temperature. The width of the hot zone is also shown to be critical in the development and continued propagation of a columnar grain structure. The effects of changing the temperature gradient ahead of the hot zone have also been investigated. Research supported by AFOSR grant F49620-00-1-0076 and NSF grant DMI9976509.

9:30 AM
A Software Tool to Design Preform and Die Shapes for Radial-Axial Profile Ring Rolling: *Anil Chaudhury*[2]; Chandrasekhar Reddy[1]; Urban De Souza[1]; Suhas Vaze[1]; Ken Phillips[3]; [1]Concurrent Technologies Corporation; [2]Applied Optimization, Fairborn, OH USA; [3]Firth-Rixson Viking, Reno, NV USA

On a radial-axial ring rolling mill, typical profile rings may be produced from a starting ring preform by using up to three mandrels and two king rolls. The design of dies and preforms has mostly relied on shop-floor experience and multiple ìcut-and-tryî methods. The process risks are high. For example, a ring part with the required profile but wrong diameter is considered 100% scrap and cannot be salvaged. Under these circumstances, computer simulation and design tools, which reduce process risk, offer tremendous potential to address cost reduction and process improvement for aerospace ring forged parts. Recently MSC.Software modified their 3D-finite element tool (MSC.MARC) to efficiently simulate the complex interaction between the rolls and the ring. To rapidly generate and evaluate preform and die shapes for the detailed analysis in MSC.MARC, a design tool (preForma-ring) was developed which will be described in this paper. This tool is capable of generating smooth intermediate shapes between preform and final part geometry in matter of minutes. These shapes are then evaluated using a number of geometric rules, which help to select the optimal preform shape for subsequent FEA evaluation and/or shop trials.

10:00 AM
Application of the Upper Bound Elemental Technique (UBET) to the Simulation of Profile Ring Rolling of High Temperature Alloys: *Jay Gunasekera*[2]; Vipul Ranatunga[2]; Urban De Souza[1]; Suhas Vaze[1]; [1]Concurrent Technologies Corporation, Johnstown, PA 15904 USA; [2]Ohio University, Dept. of Mechl. Eng., OH USA

Currently, simulation of profile ring rolling requires application of time-consuming 3-D finite element techniques. There is a need for rapid software tools to aid designers in developing ring-rolling process schedules thereby helping in reducing the design cycle time. The upper bound elemental technique is a rapid load estimating technique that has potential to meet this need and therefore its application to the unique 3-D flow situation experienced in profile ring rolling is being explored. This paper describes a UBET-based analytical method developed for the modeling of profile ring rolling of high temperature aerospace materials. The plastic flow of the material during rolling is described by dividing the deformation region between the two rolls (bite section) into rectangular and triangular prismatic elements and deriving kinematically admissible velocity fields for the 3-D material flow in each element. The currently developed method is capable of predicting ring diameter growth, die loads, material flow, ring strains and temperatures as a function of time. Numerical results have been compared with data available for high temperature alloys such as IN718 and Ti-6Al-4V.

10:30 AM Cancelled
Process Modeling of Thixotropic Forming Process of Steel Alloys: *Dierk C. Hartmann*

Modeling of Multi-Scale Phenomena in Materials Processing: Grain Boundary Phenomena

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

Wednesday AM — Room: 304
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Anthony D. Rollett, Carnegie Mellon University, Matls. Sci. & Eng., Wean Hall 3327, 5000 Forbes Ave., Pittsburgh, PA 15213-2890 USA

8:30 AM
Providing a Length Scale by Capturing the Influence of Grain Boundaries in Polycrystal Plasticity Modeling: *Thomas E. Buchheit*[1]; Corbett C. Battaile[2]; Gerald W. Wellman[3]; Elizabeth A. Holm[2]; [1]Sandia National Laboratories, Dept. 1835, MS 0889, PO Box 5800, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 1834, MS 1411, PO Box 5800, Albuquerque, NM 87185 USA; [3]Sandia National Laboratories, Dept. 9123, MS 0847, PO Box 5800, Albuqueque, NM 87185 USA

A material model which uses a crystal plasticity framework to simulate deformation response of polycrystalline metals has been implemented into JAS-3D, a quasistatic finite element code developed at SNL. The resultant microstructure-based deformation model has successfully performed 3-D simulations of realistic polycrystalline microstructures composed of 200 grains with approximately 200 elements per grain. Within this framework, a phenomenology has been devised that contains a gradient in hardening near grain boundaries. Simulations which include this phenomenology accurately capture the Hall-Petch effect and provide a length scale within the polycrystalline material response. Results will be presented. In addition, model results contain crystal orientation and hardening state variable information within each finite element of the model which can be compared with experimental data obtained by methods such as EBSD and nanoindentation. These type of microstructure level comparisons, useful in guiding development of this deterministic modeling approach, will also be included in the presentation. 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.

8:55 AM
Simulation Studies of Grain Boundary Diffusion in NiAl: *Diana Farkas*[1]; Benjamin Soule de Bas[1]; [1]Virginia Tech, Dept. of Matls. Sci., Blacksburg, VA 24060 USA

We present the results of large scale molecular dynamics simulations of grain boundary diffusion in B2 NiAl. The studies use EAM interatomic potentials to simulate the diffusion process in a direct manner, starting with vacancies located in different sites in the grain boundary region. The simulations show a variety of diffusion jumps, all introducing disorder in the grain boundary. This is in contrast to diffusion in bulk NiAl, where cyclic mechanisms, restore order. Several series of correlated jumps were identified in the grain boundary region. The most common of these result in the vacancy remaining in the same structural unit. The mechanisms of the vacancy diffusing to a neighboring structural unit were also observed and are reported in detail.

9:20 AM
Subgrains Formation in Uranium as an Experimental Tool for Dislocation Plasticity: *Gabriel Abraham Frank*[1]; Roni Zvi Shneck[1]; Alex Landau[2]; Arie Venkert[2]; [1]Ben-Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer Sheva 84105 Israel; [2]NRCN, PO Box 9001, Beer Sheva Israel

Texture formation and dislocation patterning into walls and cells are long-standing questions in the theory of plasticity that may benefit from the study of the deformation of metals having low symmetry. A central difficulty in analysis of the deformation of most metals is the multiplicity of slip systems, which gives rise to ambiguities in the selection of the active systems. We study the subgrain formation in uranium during β-α phase transformation and illustrate its association with predictable plastic deformation. These subgrains were suggested to be creep cells formed by the plastic deformation accommodating the transformation strains. The low ambiguity allows determining a unique combination of the active slip systems. Misorientations across subgrains in α-uranium were determined by SAD/TEM and shown to be accommodated by the dislocations arising from the active slip systems. Frank-Bilby equation was used to determine the relative densities of the dislocations in each boundary.

9:45 AM Break

10:00 AM
Experimental Characterization of the Three-Dimensional Dislocation Substructure Evolution in Copper during Changing Strain Paths for Input and Validation of the Dislocation Dynamics Models: *Rodney J. McCabe*[1]; Amit Misra[1]; Terence E. Mitchell[1]; Michael G. Stout[1]; George C. Kaschner[1]; Kathleen B. Alexander[1]; [1]Los Alamos National Laboratory, MST-8 Struct./Prop. Relations Grp., MS-G755, Los Alamos, NM 87545 USA

Innovative modeling techniques are being developed at all length scales that incorporate three-dimensionality of the relevant microstructures. However, the body of experimental work available for 3D model input and validation is limited. We have performed a detailed, quantitative TEM study of the 3D dislocation substructure evolution in polycrystalline copper loaded monotonically as well as under changing strain paths. Stereo-TEM is used to reveal the three-dimensional dislocation configurations that can be used as input for dislocation dynamics models or as verification of the predicted equilibrium dislocation structures. In situ straining TEM, in conjunction with post mortem TEM, are useful for tracking the evolution of individual dislocations and dislocation substructures. Through this quantitative TEM study, we interpret the macroscopic work hardening/softening observed under changing strain paths and provide experimental support for the 3D dislocation dynamics simulations. This research is funded by the US Department of Energy, Office of Basic Energy Sciences.

10:25 AM
Investigation of Temperature Influence on Film Structure using the Level Set Method: *Anthony Tongen*[1]; David L. Chopp[1]; [1]Northwestern University, Eng. Scis. & Appl. Math., 2145 Sheridan Rd., Evanston, IL 60208 USA

We investigate two dimensional grain growth by means of the level set method. This study examines the relationship between film growth temperatures and the preferred orientation of surface crystals. Some materials, such as TiN, can have disparate textures due to different annealing temperatures. For instance, at 800° the (001) orientation is observed, while at room temperature the (111) face dominates. The predominant orientation of the film controls the material properties of the overall film. The level set method is a powerful method for moving fronts which can handle sharp corners and cusps in the interface and topological changes naturally. Anisotropic surface diffusion, a fourth order nonlinear operator, is often the rate limiting step in numerical simulations. We have employed a new technique coupling the level set method with a marker particle method to speed up the computational time. We simulate deposition and annealing to demonstrate agreement with experimental results.

10:50 AM
The Impact of Anisotropic Grain Boundary Properties on Texture Development: *Anthony D. Rollett*[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Wean Hall 4315, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Since grain boundary properties are generally highly anisotropic, their impact on other material properties is of great interest. For example, the strong variation in grain boundary mobility observed for the transition from low angle to high angle boundaries can lead to highly abnormal grain growth. This effect of grain boundary properties has been explored computationally with a Monte Carlo model that incorporates texture. When coarsening (grain growth) is simulated in a single crystal that possesses a finite (mosaic) spread in orientation, abnormal grain growth occurs for a particular critical dispersion. This behavior represents one mechanism for the appearance of recrystallization nuclei. The grain boundary properties used in the simulations represent a distillation of experimental and theoretical information. The latter have largely been obtained through Monte Carlo and molecular dynamics simulations of the structure and kinetics of boundaries. As an example of their importance, the growth of the {001}<100> component in fcc metals has been found to be highly dependent on the grain boundary properties.

11:15 AM
Process Design to Achieve Targetted Microstructures: The Spectral Method: *Surya R. Kalidindi*[1]; Joshua Houskamp[1]; Brent L. Adams[2]; Hamid Garmestani[3]; [1]Drexel University, Matls. Eng., Philadelphia, PA 19104 USA; [2]Brigham Young University, 435 CTB, Provo, UT 84602 USA; [3]FAMU-FSU College of Engineering, Tallahassee, FL 32310 USA

The authors have recently developed and reported a new paradigm to facilitate materials by design by using spectral representations of microstructure and the associated properties. In an accompanying paper, the tremendous advantages of using the spectral method in describing the universe of all relevant microstructures for the design problem at hand (denoted as the material hull) and the space of all physically realizable property combinations (denoted as the property closure) are being described in detail. In this paper, this paradigm will be extended to include considerations of process design that is predicted to result in pre-selected microstructures. It will be shown that there are significant advantages to describing microstructure evolution in the Fourier space. Specifically, the concept of streamlines has been used to mathematically represent both the microstructure evolution in the material hull and the concomitant property evolution in the property closure. The newly developed methodologies will be demonstrated in the context of the design example.

11:40 AM
Incorporation of Deformation Twinning in Modeling the Mechanical Behavior of A-Titanium: *Ayman A. Salem*[1]; Surya R. Kalidindi[1]; Roger D. Doherty[1]; [1]Drexel University, Dept. of Matls. Eng., Philadelphia, PA 19104 USA

In this work we have modeled the stress-strain response and the texture evolution of a-Titanium using a polycrystal plasticity model. The model incorporated both crystallographic slip and deformation twinning as the modes of plastic deformation. The validity of the model was verified through comparisons of predicted textures and stress-strain curves with the direct measurements. The experiments included simple compression, plane strain compression, and simple shear tests. Texture measurement and twin volume fraction were evaluated by orientation imaging microscopy. Deformation twinning was found to play a vital role in the strain hardening behavior of a-Titanium. The onset of deformation twinning was associated with a marked increase in the strain hardening rate. Deformation twins are believed to reduce the effective slip distance and increase the strain hardening rate via Hall-Petch hardening mechanism. When slip, as a competing deformation mechanism, became dominant, the effect of twinning on strain hardening rate was found to saturate.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - II

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

Wednesday AM — Room: 211
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: 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; S. W. Chen, National Tsing-Hua University, Dept. of Chem. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu 30043 Taiwan

8:30 AM Invited
The Constitution of the In2O3-SnO2 System: Implications for Transparent Conducting Oxide Applications: *Douglas J. Swenson*[1]; William J. Heward[1]; [1]Michigan Tech University, Matl. Sci. & Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

SnO2-doped In2O3, also referred to as indium-tin-oxide or ITO, is a leading commercial transparent conducting material. Although widely utilized in commercial products, basic information available about the phase stability of ITO is lacking, a situation that hampers effective optimization of its processing-properties-structure relationships. In this work, phase equilibria in the In2O3-SnO2 system have been studied over the entire composition range between 1000 and 1650°C, using electron probe microanalysis and x-ray diffraction analysis. Two ternary phases, In4Sn3O12 and In2SnO5, were found to exist in this system. Both phases are stable only at high temperatures, decomposing eutectoidally at 1325 and 1600°C, respectively. The maximum solubility of SnO2 in In2O3 was found to increase dramatically with increasing temperature, ranging from 1.3 mol% at 1000°C to 13.1 mol.% at 1650°C. These results suggest that most commercially produced ITO thin films are thermodynamically metastable, being substantially supersaturated with SnO2. Other implications of the study are also discussed.

8:55 AM
Preferential Dissolution and Inter-Metallic Compound Formation with Multi-Component Base Metal and Solder Alloys: *Kenneth L. Erickson*[1]; Polly L. Hopkins[1]; Paul F. Hlava[1]; Paul T. Vianco[1]; Jerome A. Rejent[1]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

An understanding of the physical and chemical mechanisms controlling reaction between base metal and molten solder is important for developing soldering processes, particularly for multi-component base metal and solder alloys. Depending on materials and process conditions, solid-solid and liquid-solid reactions coupled with solid-state and liquid-phase diffusion can cause preferential dissolution of base metal constituents and non-equilibrium inter-metallic compound formation between base metal and solder. For example, recent experiments with 76Au-21Pt-3Pd (wt%) alloy sheet and 63Sn-37Pb solder showed that preferential dissolution of Au by molten 63Sn-37Pb solder causes formation of Pt-rich inter-metallic compounds between alloy sheet and molten solder. During subsequent solid-state aging the Pt-rich compounds transform to Au-rich inter-metallic compounds consistent with the initial base metal composition. This paper summarizes results from experiments with 76Au-21Pt-3Pd (wt%) alloy sheet and 63Sn-37Pb solder and discusses the computational models developed to examine the competing effects of diffusion and reactive phase formation and dissolution.

9:15 AM Invited
Diffusion and Interfacial Reactions between Thin-Film Ti/Ni/Ag Metallization and a Lead-Free Solder: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The interfacial reactions involving thin-film Ti/Ni/Ag metallizations on two semiconductor devices, diode and MOSFET, a Sn-3Ag-0.7Cu solder, and a Au-layer on the substrate are studied. Interfacial reactions are investigated during reflow process and during accelerated aging at 125°C. To understand the interdiffusion processes, the interfacial reaction products are characterized using SEM, TEM and AEM. Two intermetallics, Ni_3Sn_4 and Cu_6Sn_5, are found to form during reflow process. The primary microstructural dynamics during solid state aging are the coarsening of intermetallic particles and reactions involving the Ni- and Ti-layer with Au and Sn. While the reaction with the Ni-layer yields only Ni_3Sn_4, reaction involving the Ti-layer suggests the formation of Ti-Sn and Au-Sn-Ti intermetallics. These results along with the relevant phase stability and the diffusion mechanism will be discussed in detail.

9:40 AM Invited
Flux-Free Direct Chip Attachment of Flip Chip with Lead-Free Solder Bump: *Choon-Sik Kang*[1]; Soon-Min Hong[1]; Jae-Pil Jung[2]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., San 56-1, Shillim-dong, Kwanak-ku, ENG445_F, Seoul 151-744 Korea; [2]University of Seoul, Dept. of Matls. Sci. & Eng., 90 Jeonnong-dong, Dongdaemun-ku, Seoul 130-743 Korea

The flux in soldering removes contaminants and dissolves surface oxides to improve solderability. The application of flux, however, could lead to the corrosion of the circuit and deterioration of long-time reliability due to the flux residues in the joint. Especially in fine pitch flip chip package, the residual flux may exist in the region where removal of the residue is almost impossible. Besides, using flux causes environmental concerns, so manufacturer needs to find an effective replacement of the flux. Therefore, fluxless soldering is becoming an active research area. In this work, fluxless bonding of flip chip with Sn-Pb and Sn-Ag solder bump to Glass substrate was studied with the purpose of optoelectronics application. Instead of flux, plasma treatment was used to remove the oxides and other contaminants. The degree of oxide removal on solder surface was examined by AES. The effect of flip chip process parameters were evaluated and discussed.

10:05 AM Break

10:25 AM Invited
Reliability Issues of Pb-Free Soldering Caused by Metallurgical Reactions: *Kejun Zeng*[1]; King-Ning Tu[1]; [1]UCLA, Dept. of Matls. Sci. & Eng., 6532 Boelter Hall, Los Angeles, CA 90095-1595 USA

Some reliability issues of Pb-free solder processing in electronic packaging technology, that are caused by metallurgical reactions between solder and under-bump-metallizations (UBM), are discussed. These include the formation of the interfacial intermetallic compounds, interfacial segregation, contamination of Pb-free solder joints, and compatibility of UBM and surface finishing with Pb-free solders. Thermodynamic calculations and kinetic considerations are applied to the above cases. Attention has been paid to the influences of processing or experimental conditions on the microstructural stability of the interfacial areas in solder joints and the joint reliability.

10:50 AM Invited
Solder Joint Strength and Interfacial Reaction between Cu/X (X=Ni, Ag or Ni/Ag) Substrate and Sn-36Pb-2Ag Solder Alloy: Seung Wook Yoon[1]; *Jong Hoon Kim*[2]; Chang Joon Yang[2]; Hyuck Mo Lee[2]; [1]Hynix Semiconductor Corporation, Pkg./Module R&D, San 136-1, Ami-ri, Bubal-eub, Ichon-si, Ichon, Kyoungki-do 467-701 Korea; [2]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Kusung-Dong 373-1, Yusung-Gu, Taejon 305-701 Korea

Interfacial reaction between electroplated Ni, Ag or Ag/Ni on Cu alloy substrate and Sn-36Pb-2Ag solder ball was investigated and their bonding strengths were measured with Dage2000 ball shear tester. Ag3Sn IMC was found at the interface between 2mm-Ag/solder ball and Ni3Sn4 was for 2mm-Ni/solder ball. In case of 2mm-Ag/2mm-Ni electroplated Cu substrate, there were Cu/Ni/Ni3Sn4/Ag3Sn/solder layers formed at interface and each IMCs were confirmed by EDX analysis. Ag layer was completely consumed by formation of Ag3Sn, but most Ni layer was remained. After various environmental tests, Ag/Ni plated Cu substrate had superior solder joint reliability than other Ni or Ag single plated Cu substrate. Two different type of reflow profile were investigated and the specimens prepared by higher soldering temperature showed superior solder joint strength. The solder joint strength and the microstructural change were observed with number of reflow cycle in considering the real board mounting. There was drastical evolution of microstructure and these could explain the solder joint strength change with reflow cycles.

11:15 AM Invited
Interface Reactions between 50In-50Pb and Copper Substrates Due to Solid-State, Acclerated Aging: *Paul Thomas Vianco*[1]; Jerome Rejent[1]; Alice Kilgo[1]; Paul Hlava[1]; [1]Sandia National Laboratories, Dept. 1835, PO Box 5800, MS 1411, Albuquerque, NM 87185-1411 USA

A study examined the interface reaction between 50In-50Pb (wt.%) solder and Cu substrates. Substrates were hot dipped in the molten In-Pb solder and aged at 55, 70, 100, 135, and 170°C for time periods of 1 to 350 days. The reaction layer composition was determined by electron microprobe analysis. The growth kinetics were calculated from layer thickness data. Ring-and-plug shear test samples were fabricated and aged for 10, 50, and 100 days under 55, 100, and 170°C. Aging caused as strength increase for a gap of 0.008 in; however, the strength decreased with aging when the gap was enlarged to 0.015 in. The fracture morphology as well as the impact of the reaction layer on shear strength will be discussed. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy under contract DE-AC04-94AL85000.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Processing and Service Properties - Session V

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

Wednesday AM Room: 205
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Tom H. Sanders, Georgia Institute of Technology, Dept. of Matls. Sci. & Eng., Atlanta, GA 30332 USA; T. Dennis Claar, Fraunhofer USA, 501 Wyoming Rd., Newark, DE 19716 USA

8:30 AM
Compressive Properties of Maraging Steel Honeycomb under Quasi-Static and Dynamic Loading: *Alethea M. Hayes*[1]; David L. McDowell[2]; Joe K. Cochran[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. N.W., Atlanta, GA 30332-0405 USA

Two-dimensional linear cellular solids or honeycombs are known for their high-strength to weight ratios compared to other low-density architectures. Georgia Tech has developed a process that produces honeycomb from maraging steel compositions including the 18% Ni series, which has high yield and ultimate strength after heat treatment. This steel incorporated into a honeycomb structure with a relative density of 20-25% produced very high compressive strength under both quasi-static and dynamic loading conditions. Both quasi-static and dynamic experiments were conducted for the out-of-plane direction of the maraging steel honeycombs. The dynamic data was generated by the split Hopkinson pressure bar and produced strain-rates on the order of 103 sec-1. Peak compressive strengths, collapse modes, and strain-rate sensitivity were examined in this study. The experimental peak quasi-static results were compared to the models developed by Thomas Wierzbicki and Lorna Gibson and Michael Ashby. All of the models under-predicted the collapse strength for this material, despite the presence of honeycomb defects and cell wall porosity inherent in this developing process. In addition, the strain-rate sensitivity for the maraging steel was very minimal.

8:55 AM
Processing and Properties of Steel Foam Materials and Structures: *T. Dennis Claar*[1]; Virgil Irick[1]; Kenneth Kremer[1]; Jim Adkins[1]; [1]Fraunhofer USA, Ctr. for Mfg. & Adv. Matls., 501 Wyoming Rd., Newark, DE 19716 USA

The powder metallurgy-based foaming process is capable of producing foams from a variety of metals, including aluminum, tin, lead, copper, and iron. This paper will focus on the processing and properties of steel foams for weight reduction of engineering structures. Steel foam precursors are produced by mixing iron powders with alloying elements and a foaming agent, and then consolidating the powder mixtures into dense compacts via hot isostatic pressing, hot pressing, extrusion, or hot rolling processes. The precursor material is then foamed by heating into the semi-solid state, during which evolved gas forms the closed-cell foam structure. The effects of precursor consolidation method, as well as various alloy and foaming agent additions, on steel foam microstructure will be discussed. The relationship between steel foam compressive strength and porosity level will be shown and compared with aluminum and titanium metal foams. The fabrication and characterization of steel foam sandwich panels and foam-filled tubes will be described. Potential uses of steel foams in ship structures, automotive, and other applications will be presented.

9:20 AM
Fabrication and Creep Studies on Open Cell Nickel Structures: *R. K. Oruganti*[1]; A. K. Ghosh[1]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward, Ann Arbor, MI 48109 USA

Open cell Ni-base alloy porous structures have potential for light-to-medium load bearing applications at elevated temperatures while they may also be actively cooled by fluid flow. In this study, fabrication of open-cell Ni structures was made by using two different processes. A process to create honeycomb cells was developed that relied on diffusion bonding of thin-walled tubes wherein sintering forces were used to convert circular core wall geometry into hexagonal ones. Linear cellular structures with very low density (6% dense) were prepared by this method. The other process utilized woven wire mat to diffusion bond a layered stack to create denser structure (56% dense). Compressive creep behavior of these structures was characterized as a function of the cell size and orientation and the results compared with those of a solid material. Inhomogeneity of deformation and large springback response differentiate the creep behavior of such cellular structures from that of solid materials. Among other features, a thermoelastic after-effect in cooling from test temperature and a mode of shear instability in progressive collapse of the cells during large deformation are seen.

9:45 AM Break

10:05 AM Cancelled
The Stiffness and Weldability of an Ultra-Light Steel Sandwich Sheet Material with a Fibrous Metal Core: *Bill Clyne*

10:05 AM
Fabrication, Mechanical Properties, and In-Situ Diffraction Strain Measurements in Aluminum-Mullite Microsphere Syntactic Foams: *Dorian K. Balch*[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., MLSB 2036, 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

High-strength aluminum syntactic foams are fabricated using a liquid metal infiltration technique. Unalloyed and 7075 aluminum are infiltrated into a packed bed of hollow ceramic spheres. The spheres consist of a combination of crystalline mullite (3 Al2O3-2 SiO2) and amorphous silica (SiO2), and have diameters of 15-75 microns and wall thicknesses of 5-10 microns. Melting of the aluminum under vacuum followed by pressurization of the infiltrator with inert gas to 3.5 MPa forces the liquid metal between the packed spheres, producing foams with relative densities between 0.45 and 0.60 (1.3-1.6 g/cm3). High compressive strengths have been observed, 100-250 MPa depending on alloy and heat treatment, with high densification strains and exceptional energy absorption. In-situ measurements of lattice strains within the mullite phase during compressive loading are performed using high-energy synchrotron x-rays, permitting investigation of sphere stresses and foam damage evolution during loading.

10:30 AM
Vibration Properties of Closed-Cell Aluminum Foam: H. F. von Bremen[1]; V. S. Sokolinsky[1]; S. Rajaram[1]; H. Shen[1]; Z. Song[1]; Y. Zhou[1]; *Steven R. Nutt*[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA

Aluminum foam may be useful as a core for sandwich structures. Many applications of sandwich structures experience dynamic excitations. Therefore, it is important to assess the vibration properties of aluminum foam. A simple experimental layout consisting of a shaker and an accelerometer is used to investigate the natural vibration modes of a cantilever aluminum foam beam. The natural vibration frequencies of the beam were measured over 20-5000 Hz. The experimentally established natural frequencies are compared with theoretical predictions. The results of experimental and theoretical studies are discussed and conclusions are drawn. Experiments show that damping properties of the foam core are manifest most noticeably in the case of symmetric vibration modes, whereas the influence on anti-symmetric modes is insignificant. The experimental measurements are in good agreement with the analytical predictions of the higher-order sandwich theory. Supported by Honda R&D Americas.

10:55 AM
Development of Vibration Damping Techniques in Metallic-Intermetallic Laminate (MIL) Composites: *Aashish Rohatgi*[1]; Kenneth S. Vecchio[1]; John B. Kosmatka[2]; [1]University of California, Mech. & Aeros. Eng., 9500 Gilman Dr., EBU-2, Rm. #262, La Jolla, CA 92093-0411 USA; [2]University of California, Struct. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0085 USA

Lightweight Ti-Al3Ti metal-intermetallic laminate (MIL) composites with Lodengraf damping have been developed. Lodengraf damping was achieved by creating cylindrical cavities in the composites and filling them with low density glass spheres. For a given Ti-Al3Ti composition, the damping characteristics of the MIL composites was controlled by varying the location and size of the cavities, and by varying the size and quantity of glass spheres used to fill these cavities.

These ìporousî MIL composite plates were vibration tested using an acoustic excitation and a non-contacting scanning laser vibrometer to determine their modal properties (natural frequency, mode shapes and damping properties). An extra layer of ferritic stainless steel was incorporated in several ìporousî Ti-Al3Ti composites. Vibration induced stress oscillations may produce eddy current damping in such composites, in addition to the Lodengraf damping. The dependence of damping on the processing variables i.e. the size and distribution of perforations, and the size and amount of glass beads, will be highlighted. The effect of introducing porosity on the mechanical strength and toughness will also be described.

Second International Symposium on Ultrafine Grained Materials: Superplasticity and Thermal Stability

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

Wednesday AM	Room: 210
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Terence G. Langdon, University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089 USA; Zenji Horita, Kyushu University, Dept. of Matls. Sci. & Eng., Fac. of Eng., Fukuoka 812-8581 Japan

8:30 AM Keynote
Superplasticity in Ultrafine Grain Materials: *Amiya K. Mukherjee*[1]; [1]University of California, Chem. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

One of the most important requirements for superplasticity is the presence of fine grain size having high angle boundaries that slide easily. Recent advances in materials processing have now produced new avenues for grain size refinement. Some of these avenues for producing ultrafine grain materials (including the process of high pressure torsion) will be described. Superplasticity has been revealed in ultrafine grain (including nano-sized grain) microstructure. The prediction of both high strain rate superplasticity and low temperature superplasticity have materialized. However, the flow stresses and strain-hardening rates for superplasticity in nanocrystalline materials are much higher than that in microcrystalline materials. The reason for this observation is not entirely clear at present. This research is supported by NSF-DMR-9903321.

8:55 AM
Developing Superplasticity at High Strain Rates through ECAP Processing: *Terence G. Langdon*[1]; Minoru Furukawa[2]; Zenji Horita[3]; [1]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Fac. of Eng., Fukuoka 912-8581 Japan

Processing by equal-channel angular pressing (ECAP) leads to exceptional grain refinement, typically to the submicrometer or nanometer level, and thus to an opportunity to attain superplastic ductilities at very rapid strain rates. This paper describes the specific requirements in order to achieve high strain rate superplasticity in as-processed materials and examples are given to demonstrate the feasibility of this processing method.

9:10 AM
Microstructural Evolution on Annealing of a Severely Deformed Al 3%Mg Alloy: *Jody Scott Hayes*[1]; P. B. Prangnell[1]; P. Bate[1]; [1]UMIST, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK

An Al 3%Mg alloy has been severely deformed by the ECAE process. Billets of the alloy have been subjected to multiple passes to achieve an effective strain of 10. The severely deformed microstructure was subsequently annealed at a range of temperatures. Recrystallisation and grain growth behaviour was studied. The as-processed and low temperature anneal samples have been studied using the TEM. By measuring grain widths, lengths and misorientations, the evolution of a fine equiaxed structure from a very long thin deformation structure has been investigated. Subsequent grain growth in higher temperature anneals have been studied in the FEGSEM using EBSD analysis. Using data from the TEM and FEGSEM, the annealing behaviour was modelled using Monte Carlo-Potts and Network simulations. These showed a very rapid initial change from a high aspect ratio to equiaxed grains followed by essentially normal grain growth, though the effects of boundary misorientation needs to be considered.

9:25 AM
Enhanced Formability of Superplastic AlMgZr Alloys made by Particulate Routes: *Ray Cook*[1]; Roger Grimes[2]; Richard Dashwood[2]; [1]The Aluminium Powder Company, Ltd., Forge Ln., Minworth, Sutton Coldfield, W. Midlands B76 1AH UK; [2]Imperial College of Science Technology and Medicine, Dept. of Matls., Exhibition Rd., London SW7 2AZ UK

The technique of centrifugal atomisation has been used to produce a number of AlMgZr alloy particulates with Zr contents up to 1% that have been converted into sheet by extrusion, rolling and heat treatment and their superplastic properties determined. It has been found that control of the solidification conditions during atomisation is crucial in order to maximise Zr solid solubility and achieve a fine dispersion of Al_3Zr precipitates. Superplastic behaviour is enhanced at higher Zr contents, and by thermo-mechanical processing to achieve ductilities in excess of 600% at strain rates of 0.01 s^{-1}. Alloy conditioning was found to have a significant effect on the Al_3Zr precipitate size, with coarser precipitates relating to premature failure during testing. This paper describes the atomisation process and identifies the key parameters that must be controlled for its successful operation. A summary of the superplastic performance of the product will be included.

9:40 AM Invited
Grain Refinement and Superplasticity in Magnesium Alloys: *Hiroyuki Watanabe*[1]; Toshiji Mukai[1]; Kenji Higashi[2]; [1]Osaka Municipal Technical Research Institute, Mech. Eng. Dept., 1-6-50 Morinomiya, Joto-ku, Osaka 536-8553 Japan; [2]Osaka Prefecture University, Dept. of Metal. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

Intriguing superplastic behavior, such as high-strain-rate superplasticity and/or low temperature superplasticity, can be attained when the materials are refined to extremely fine-grained conditions because of the significant grain size dependence of this phenomenon. In addition, effect of particle and grain boundary structure on superplastic flow, which is indispensable to understand the nature or limit of superplasticity, can be investigated using low-temperature superplastic materials. In the present study, fine-grained Mg-Zn-Zr alloys have been processed by powder metallurgy route or equal-channel-angular extrusion. The materials could be refined to a sub-microcrystalline structure through these two procedures. Superplastic behavior was examined for both materials at low temperatures of ~473 K in order to shed light on the mechanism of superplastic flow. Data analyses revealed some interesting features of superplasticity, which were not found until the grain sizes reduced to the order of sub-micron.

10:00 AM
Processing of an Aluminum-6061 Metal Matrix Composite by Equal-Channel Angular Pressing: *Yi Huang*[1]; Cheng Xu[1]; Sangmok Lee[1]; Minoru Furukawa[2]; Zenji Horita[3]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Fac. of Eng., Fukuoka 812-8581 Japan

An Al-6061 metal matrix composite, reinforced with 10 vol% Al_2O_3 particulates, was subjected to equal-channel angular (ECA) pressing. The grain size of the composite was reduced to the submicrometer level through processing by ECAP. The mechanical properties of the material were investigated using tensile tests at high temperatures over a range of strain rates. This paper describes and discusses the mechanical properties of the composite after ECAP and presents representative microstructures obtained using TEM.

10:15 AM Break

10:25 AM Keynote
Grain Refinement of Copper Based Alloys using ECAP: *Zenji Horita*[1]; Koji Neishi[1]; Terence G. Langdon[2]; [1]Kyushu University, Fac.

of Eng., Dept. of Matls. Sci. & Eng., Fukuoka 812-8581 Japan; [2]University of Southern California, Dept. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing (ECAP) was conducted for grain refinement of Cu alloys containing Zn and/or Zr. The ECAP samples were subjected to static annealing at elevated temperatures. Microstructures were observed by transmission electron microscopy and tensile properties were examined at elevated temperatures. Microstructural features after static annealing were compared with those of Al alloys. It is shown that recrystallization occurs discontinuously in ECAP-Cu alloys whereas grains grow continuously in ECAP-Al alloys without having the nucleation of recrystallized grains. In ECAP-Cu alloys, the recrystallization temperature depends sensitively on the contents of Zn or Zr. It is also shown that superplasticity was attained at an optimized condition for an ECAP-Cu alloy.

10:50 AM

Structure, Properties and Thermal Stability of Ultra-Fine Grain Cu-Cr-Zr Bronze: *Alexei Vinogradov*[1]; Vsevolod Patlan[2]; Kazuo Kitagawa[2]; Vladimir I. Kopylov[3]; [1]Osaka City University, Dept. Intelligent Matls. Eng., Osaka 558-8585 Japan; [2]Kanazawa University, Mech. Sys. Eng., Kodatsuno 2-40-20, Kanazawa 920-8667 Japan; [3]Physical-Technical Institute of the National Academy of Science, Minsk 220730 Belarus

The structure, thermal stability, mechanical and electrical properties are investigated of ultra-fine grain Cu-Cr-Zr bronze obtained by equal-channel angular pressing (ECAP). Strengthening mechanisms resulting from a combination of intensive cold work and subsequent aging have been taken advantage of to produce materials which have good electrical conductivity, hardness, tensile strength and excellent thermal stability, have sufficient ductility for forming and also effectively resist fatigue stressing. Optimal post-processing aging conditions were found to ensure a desired combination of properties. A special attention is paid to the fatigue behavior, which is characterized in both strain, and load controlled experiments. Fatigue mechanisms and nature of cyclic softening is discussed. Experimental data unambiguously show that the Cu-Cr-Zr bronze processed in the ECAP-aging scheme has significant advantages over currently available, commercial tempers in that it permit designs having much higher working stresses and better material utilization, thus providing a better cost effectiveness.

11:05 AM

Effect of Thermal Stability of Microstructure on the Tensile Properties: *Bing Q. Han*[1]; Terence G. Langdon[2]; [1]University of California, Dept. of Chem. & Biochem. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA; [2]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Severe plastic deformation of a commercial 2219 Al alloy was performed through equal-channel angular pressing at room temperature. Ultrafine grains of about 0.2 mym were present after 8 and 12 passes. However, superplasticity of ultrafine-grained Al 2219 alloy was not observed at temperatures from 573 to 673 K. Although there are precipitates of q¢ or q in the microstructure of the Al 2219 alloy, they are not sufficiently strong to inhibit grain growth by dynamic recrystallization. It is suggested that thermal stability of the microstructure in Al alloys after severe plastic deformation plays a significant role in the occurrence of superplasticity. The relationship between precipitates, stability of microstructure, and superplasticity of several aluminum alloys after severe plastic deformation is discussed.

11:20 AM

Effect of Pressing Strain on the Annealing Behavior of Ultrafine Grained Ferrite in a Low Carbon Steel Fabricated by Equal Channel Angular Pressing: *Kyung-Tae Park*[1]; Dong Hyuk Shin[2]; [1]Hanbat National University, Div. of Adv. Matls. Sci. & Eng., San 16-1, Dukmyung-Dong, Yuseong-Gu, Taejon 305-719 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., 1271 Sa 1-Dong, Ansan, Kyunggi-Do 425-791 S. Korea

The annealing behavior of submicrometer order grained ferrite in a low carbon steel fabricated by equal channel angular pressing was investigated. In particular, the effect of the pressing strain on the annealing behavior of submicrometer order grained ferrite was examined by imposing two different pressing strains, 4 and 8. Contrary to the general fact that more severely worked materials exhibit faster kinetic of microstructural evolution related to annealing, i.e. recovery, recrystallization and grain growth, more strained steel exhibited more sluggish recovery and recrystallization kinetics under the present experimental conditions. It is suggested that more sluggish recovery and recrystallization kinetics with increasing the pressing strain is attributed to the combined effects of the release of the accumulated strain energy by formation of equilibrated structure and carbon dissolution from pearlitic cementite during pressing. The grain growth kinetics of submicrometer order grained ferrite was analyzed by applying the general description of the grain growth law. The grain growth exponent was about 0.2 and the activation energy for grain growth, 170-180 kJ/mol, was comparable to that for grain boundary diffusion of Fe in alpha-iron. Both values were not influenced by the pressing strain.

11:35 AM

Annealing Effect on the Strength of Severe Plastic Deformed Titanium: *Alla V. Sergueeva*[1]; Ruslan Z. Valiev[2]; Amiya K. Mukherjee[1]; [1]University of California, CHMS, 1220 Bainer Hall, Davis, CA 95616 USA; [2]Ufa State Technical University, Inst. of Phys. Of Adv. Matls., 12 Marx Str., Ufa 450000 Russia

Recent studies have shown that low temperature annealing (LTA) of severe plastic deformed (SPD) materials can lead to changes in the Hall-Petch relation slope without changing grain size. The nature and mechanisms of the strengthening are not clear yet. Mostly, the increasing in strength and hardness has been attributed to recovery processes. In present study it was shown that the maximum strength (1250 MPa) of SPD Ti can be achieved by short annealing at 250-300°C. The increasing temperature and/or time of thermal treatment lead to decreasing strength of the material. TEM observation revealed highly distorted structure within individual grains with non-uniform contrast in as-prepared state, whereas, the annealing results in full grain structure relaxation. But in this case, the highly non-uniform contrast of grain boundaries and near grain boundaries regions has been observed. The analysis of possible structural changes and their effect on the tensile properties has been presented. This investigation was supported by a grant from the US National Science Foundation (NSF-DMR-9903321).

11:50 AM

Effect of Refined Grain Size on the Shape Memory Behavior of NiTi Alloys Processed by ECAE: *I. Karaman*[1]; H. J. Maier[2]; H. Rack[3]; [1]Texas A&M University, Dept. of Mech. Eng., MS 3123, College Station, TX 77843 USA; [2]Universit‰t Paderborn, Lehrstuhl f. Werkstoffkunde, Paderborn 33095 Germany; [3]Clemson University, Dept. of Cer. & Matls. Eng., Clemson, SC 29634 USA

Shape memory alloys have unique properties such as recoverability of deformation upon heating and pseudoelastic stress-strain behavior strains near 10%. To our knowledge, the effect of severe plastic deformation and the refined grain sizes below a micron has not been investigated to a great extent on the martensitic transformation that leads to pseudoelasticity and shape memory. The purpose of this study is to develop NiTi shape memory alloys (SMAs) with submicron grain sizes and to investigate the pseudoelasticity and shape memory characteristics in the presence of high volume fraction of interfaces. The materials chosen for this study are NiTi alloys with various compositions near equiatomic composition to control the phases present during selected processing routes and temperatures such as fully martensitic, fully austenitic and the mixture of martensitic and austenitic phases. The materials will be processed starting from coarse-grained precursors and processed by severe plastic deformation methods including equal channel angular extrusion (ECAE). ECAE will provide refined microstructure while conserving work piece geometry and some control over texture evolution. Several characterization techniques have been employed to identify resulting microstructure and mechanical behavior of the NiTi alloy produced. In this study, we will focus on four issues, namely (1) the effect of processing parameters on the resulting microstructures depending on the initial phase of the material (martensite or austenite), (2) the variation of transformation temperatures with the degree of deformation and the ECAE route, and (3) the monotonic tension and compression behavior in the pseudoelasticity and shape memory regions.

12:05 PM

Structure and Stability of Ultra-Fine Grain Materials: Role of Impurities and Second Phase Particles: *Nina Alexsandrovna Koneva*[1]; Anatolii Nikolaevich Zhdanov[1]; Nataliya Anatolievna Popova[1]; Evgenia Eduardovna Pekarskaya[1]; Eduard Victorovich Kozlov[1]; [1]Tomsk State University of Architecture and Building, Phys. Dept., Solyanaya Sq. 2, Tomsk 634003 Russia

The results of studied structure of ultrafine grained copper, nickel and alloys on their base, produced by severe plastic deformation(SPD), is generalized. There is a problem of real grains size, surrounded by large-angle boundaries. The sizes indicated are often underestimated. This questions is closely related to the anisotropy of grains, presence of subboundaries and structural texture of materials after SPD. Statistical analysis of distribution functions of sizes of grains and subgrains is performed. The problem of comparison of grain sizes, determined by

X-ray analysis and TEM, is discussed. Mechanisms of arising internal stress sources are analyzed. Role of grain boundaries, their joints and second phase particles is compared. SPD leads to redistribution of impurities in volume of metal. They are captured by dislocations and swept out primarily on grain boundaries and to joints of boundaries, where second phase particles are formed. The presence of particles is related to the problem of structure stability.

Surface Engineering: Science & Technology II: Oxidation/Corrosion/Nitridation

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

Wednesday AM Room: 203
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Gary L. Doll, The Timken Company, Canton, OH 44706 USA; S. V. Prasad, Sandia National Laboratories, Albuquerque, NM 87111 USA

8:30 AM Invited

Lower Thermal Conductive Thermal Barrier Coatings with Improved Cyclic Oxidation Life for Turbine Components: *Douglas E. Wolfe*[1]; *Jogender Singh*[1]; [1]Pennsylvania State University, Appl. Rsrch. Lab., PO Box 30, State College, PA 16804 USA

In order to produce lower thermal conductive thermal barrier coatings (TBC) that will allow higher engine operating temperatures, increased fuel savings, and longer component life, new coating materials and microstructural modifications are being considered. This paper discusses microstructural changes within the ZrO2-8wt.%Y2O3 (TBC) layer which dramatically lowered the thermal conductivity, while increasing the cyclic oxidation life of coated components. Altering the microstructure by introducing interfaces within the TBC has lead to a 20% reduction in the thermal conductivity and improved oxidation cyclic life (over 50%) of partially stabilized zirconia (ZrO2-8wt.%Y2O3) deposited by electron beam-physical vapor deposition on platinum-nickel-alumumide bond coated MAR-M-247. The traditional columnar microstructure of partially stabilized zirconia has been modified to produce a lower conductive TBC by periodically interrupting the condensing vapor and creating multiple interfaces. These interfaces serve as barriers for phonons, and thus decreases the thermal conductivity of the deposited coating. The thermal conductivity can further be controlled by controlling the distinctiveness and number of interfaces within the TBC layer. However, if the interfaces are too distinct, premature delamination of the layers may occur resulting in lower cyclic oxidation life. TBC samples were examined by various techniques including scanning electron microscopy (SEM), X-ray diffraction (XRD), electron probe microanalysis (EPMA), steady-state laser heat flux technique, and thermal cyclic tests. Multiple interfaced TBC exhibited better cyclic oxidation life with lower thermal conductivity as compared to conventional thermal barrier coatings (ZrO2-8 wt.%Y2O3).

8:55 AM

Factors Controlling In-Plane Cracking of Thermal Barrier Coatings: Zhehua Zhang[1]; Arne H. Swanson[1]; *Jun Kameda*[1]; Shigeo Sakurai[2]; Minoru Sato[3]; [1]Iowa State University, Ames Lab., 258 K Metals Dvlp., Ames, IA 50014 USA; [2]Hitachi, Ltd., Hitachi Work, Hatachi Japan; [3]Tohoku Electric Power Co., Ltd., Sendai 980 Japan

This paper summarizes a recent study on the in-plane cracking behavior near and away from the interface of thermally sprayed (PS) thermal barrier coatings (TBC) and thermally grown oxides (TGO) using a protruded TBC bend testing technique. The critical local tensile stress (σ^*) for in-plane TBC cracking near and away from the interface decreased and increased, respectively, with increasing substrate thickness. In order to examine how the formation of TGO and residual stresses control the in-plane TBC cracking, thermal ageing and cycling at 950°C in air were carried out. Near-interfacial cracks were initiated at slightly higher σ^* in aged specimens than in unaged specimens due to the sintering effect. The result indicates that the TGO formation itself does not cause the mechanical degradation of TBC without thermal cycling. The effect of thermal cycling on the in-plane TBC cracking is being examined to clarify the role of residual stresses.

9:10 AM

Developing a MoSi2+SiC Oxidation Resistant Coating for Mo a Prototype Refractory Metal: *Earl C. Hixson*[1]; [1]Colorado School of Mines, Metall., 920 16th St., Golden, CO 80401 USA

Refractory metals offer excellent high temperature mechanical properties, but lack sufficient oxidation resistance in air to see wide application. An oxidation resistant coating could potentially extend the usefulness of refractory metals at high temperature. Any coating system must have sufficient oxidation resistance and survive the thermal stresses of cycling between room temperature and 1600°C. The MoSi2+50wt% SiC coating system shows excellent oxidation resistance and matches the coefficient of thermal expansion of molybdenum. When combined with a Mo-Si-C-N containing diffusion barrier layer (DBL), the resulting coating system could successfully protect Mo from high temperature oxidation. Finite element models have been used to reduce the number of experiments and predict the stresses induced by thermal cycling. The finite element models have identified the DBL CTE as the critical variable in the coating stresses. Both FEM stress predictions and experimental oxidation and DBL crystallization studies will be summarized. Finally, an optimum coating architecture will be proposed.

9:25 AM

Development of Reactive Element Coatings to Improve H.T. Oxidation Performance of Iron-Chromium Alloys: *Lalgudi V. Ramanathan*[1]; Stela Maria Fernandes[1]; [1]Instituto de Pesquisas Energeticas e Nucleares-IPEN (Energy & Nuclear Rsrch. Inst.), Matls. Sci. & Tech. Ctr., Travessa R-400, Cidade Univ., Sao Paulo 05422-970 Brazil

The addition of small quantities of reactive elements (REs) to chromia and alumina forming alloys improves high temperature (HT) oxidation resistance. Traditionally, REs have been added to HT alloys in elemental form or as oxide dispersions. The REs can be also applied as coatings. The sol-gel technique is considered to be the most efficient and it generates nano-sized oxide particles. The effect of sol-gel processing parameters on RE particle size and morphology was determined. Various REs were studied and included: La, Nd, Ce, Pr, Dy, Y and Yb. Different techniques to coat alloy surfaces with RE sols were evaluated and spraying was found to be the most effective. The morphology of the coatings varied with the nature of the RE. The oxidation behavior of uncoated and RE coated specimens was evaluated from isothermal and cyclic oxidation tests. The extent to which the RE coatings influenced oxidation rate varied with oxide morphology and RE. Direct correlation between RE ion radius and extent of influence on chromia growth rate at 1000°C was obtained. The effect of applying secondary RE coats on the extent of protection was also determined gravimetrically and 3 RE oxides were selected (based on results from isothermal tests and cost). Based on results obtained in this investigation, an optimum procedure to protect chromia forming alloy components in HT oxidizing environments has been proposed.

9:40 AM

High Temperature Oxidation of Electrodeposited RE-Ni-W-P-SiC Multifunctional Composite: *Tongshun Fu*[1]; Zhongcheng Guo[2]; Shanli Ma[1]; Xiaoyun Zhu[2]; [1]China Great Wall Aluminium Company, Zhenzhou 450041 China; [2]Kunming University of Science and Technology, Fac. of Matl. & Metlgcl. Eng., Kunming 650093 China

The oxidation of the Multifunctional Electrodeposited RE-Ni-W-P-SiC Composites at high temperature is investigated. The results show that during high temperature oxidation the relationship between the mass change of pure Ni, Ni-W-P, Ni-W-P-SiC or RE-Ni-W-P-SiC coating and the oxidation time follows a mixed curve, i.e. it is approximately a linear relationship when the oxidation time is less than 60 mins while it is a power function relationship when the oxidation time is over 60 mins. The order for the oxidation rate of the four coatings is Ni> Ni-W-P> Ni-W-P-SiC>RE-Ni-W-P-SiC. The mass change of Ni-W-P, Ni-W-P-SiC or RE-Ni-W-P-SiC coating increases exponentially with a rise of oxidation temperature. The high temperature-oxidation resistance of RE-Ni-W-P-SiC composite coating is 3~4 times that of Ni-W-P alloy coating. The cross section morphologies and X-ray diffraction patterns indicate that the high temperature-oxidation resistance of RE-Ni-W-P-SiC composite coating is better than any other coatings.

9:55 AM Break

10:10 AM Cancelled
Investigation of a Low-Cost MOCVD Route for YSZ Thermal Barrier Coatings: *Susan Pran Krumdieck*

10:35 AM Cancelled
Oxidation Behavior of Bulk Dense Chromium Nitride Ceramics: *Hideyuki Kuwahara*

10:50 AM
The Corrosion Behavior of PVD-Grown WC-(Ti1-xAlx)N Films Superlattice in 3.5% NaCl Solution: *Seung-Ho Ahn*[1]; Yoon-Seok Choi[1]; Ji-Hong Yoo[1]; Jung-Gu Kim[1]; Jeon-Gun Han[1]; [1]SungKyunKwan University, Adv. Matls. Eng., 300, Chunchun dong, Jangan-Gu, Suwon 440-746 S. Korea

WC-(Ti1-xAlx)N coatings of constant changing Al concentration were deposited on S45C substrates by high-ionization sputtered PVD method. The Al concentration could be controlled by using evaporation source for Al and fixing the evaporation rate of the metals (WC alloy and Ti) (i.e., WC-Ti0.86Al0.14N, WC-Ti0.72Al0.28N, and WC-Ti0.58Al0.42N). The corrosion behavior of WC-(Ti1-xAlx)N coatings in deaerated 3.5% NaCl solution was investigated by electrochemical corrosion tests and surface analyses (EDS, XRD, AFM, and SEM). Particular attention was paid to the effects of Al target power density on the film properties related to the corrosion behavior. The measured galvanic corrosion currents between coating and substrate indicated that WC-Ti0.72Al0.28N coating showed the best resistance of the coating tested. The results of potentiodynamic polarization tests showed that the WC-Ti0.72Al0.28N coating deposited with 63W/ß≤ of Al target revealed higher corrosion resistance. This indicated that the WC-Ti0.72Al0.28N coating is effective in improving corrosion resistance. In EIS, the WC-Ti0.72Al0.28N coating showed one time constant loop and increased a polarization resistance of coating (Rcoat) relative to other samples.

11:05 AM Cancelled
Nitriding Behavior of Fe-Cr Alloys in the Temperature Range of 873-1073 K: *Hideyuki Kuwahara*

11:20 AM
Oxidation Behavior of Gama-TiAl Based Alloy Protected by Silicon Diffusion Treatment: *Xiang Yang Li*[1]; Y.-C. Zhu[1]; K. Fujita[1]; N. Iwamoto[1]; S. Taniguchi[2]; K. Nakagawa[3]; Y. Matsunaga[3]; [1]Ion Engineering Research Institute Corporation, 3rd Proj. Rsrch. Lab., 2-8-1, Tsuda-Yamate, Hirakata, Osaka 573-0128 Japan; [2]Osaka University, Dept. of Matls. Sci. & Eng., Grad. Sch. of Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [3]Ishikawajima-Harima Heavy Industry, Tokyo 135-8732 Japan

Silicon diffusion treatment to improve the oxidation resistance of a ◊-TiAl based alloy (Ti-48Al-1.3Fe-1.1V-0.3B at.%) was conducted by burying the specimen into Si powder and diffused at 800, 900 and 1000¢J for 2h and 5h in vacuum respectively. The isothermal oxidation behavior of the protected alloy was tested at 900¢J for 96h in air environment using a thermal balance. The element constitution, phase composition and morphology of Si modified layer and oxide scale were characterized by AES, XRD and SEM. The experimental results indicated that the oxidation resistance of the TiAl alloy was dramatically enhanced by Si diffusion treatment above 900¢J. The oxidation rate of 800¢J Si diffused alloy, however, is lower only in the beginning of the oxidation and accelerated shortly. It is found that Si rich layer was formed on the surface of the alloy and its thickness increased along with raising of the diffusion temperature. In addition, original surface phase of ◊-TiAl was transformed into Ti5Si3 or other Si contained phases when the Si diffusion temperature higher than 900¢J, meanwhile, a layer rich in Si still remained in the oxide scale even after long-term oxidation in this case. It is concluded that the new phase such as Ti5Si3 formed on the surface of the alloy during Si diffusion treatment above 900¢J and the existence of the Si rich layer in the oxide scale contributed to the significant improvement of the oxidation resistance of TiAl based alloy.

11:35 AM
Electronoptical Observations for Electrical Breakdown Initiation of Anodic Films during Anodizing in Molten Bisulphate Melt: *Young Seog Kim*[1]; [1]Korean Institute of Industrial Technology, Ctr. of Tech. Dvlp. & Assistance for SMEs, 472. Ka Jwa Dong, Soo-Ku, Inchon 404-254 Korea

Anodizing of aluminium at constant current density in molten eutectic bisulphate melts results in the development of amorphous anodic alumina, which has been examined by TEM with EDX facilities and other analytical procedures. Over a wide range of anodizing conditions, barrier type anodic films are developed at high current efficiency at voltages below the dielectric breakdown voltage. The first sign of incipient breakdown revealed by transmission electron microscopy of stripped films, is always the appearance of dark regions about 1,000 nm in diameter, representing local overgrowth of the film. The breakdown mechanism is closely related to thermal effects, because temperature rises at regions representing local overgrowth in the stripped films were observed at voltages close to the breakdown voltage, likely arising through impact ionization. When impact ionization occurs at the low activation energy region of the aluminium substrate, i.e. grainboundary of substrate, then, motion of excessive activated positive ions(aluminium ion) from the substrate towards the film/electrolyte interface increases the resultant field strength across the film. This field enhancement process generates further electronic current at the film/electrolyte interface and increases the temperature in the film.

11:50 AM
A Process-Structure-Property Model for Nitrocarburising in a Fluidised Bed: *D. Fabijanic*[1]; G. L. Kelly[1]; P. D. Hodgson[1]; [1]Deakin University, Sch. of Eng. & Tech., Waurn Ponds, Victoria Australia

Nitrocarburising is a thermochemical treatment designed to diffuse nitrogen and carbon in to a ferrous substrate. Resulting is the formation of a hard, wear-resistant white layer (WL) above a tough diffusion zone (DZ). The WL consists primarily of the h.c.p. epsilon-Fe2-3(C,N) and f.c.c. gamma-Fe4N, while the DZ is a nitrogen-saturated iron matrix. This study aimed at establishing the relationship between process inputs and the resulting structure and properties of a nitrocarburised layer on various ferrous substrate materials. A Design of Experiment (DoE) was performed using a Taguchi L9 orthogonal array on four pertinent process parameters; temperature, time, nitriding potential and concentration of hydrochloric acid. Through subsequent surface characterisation a process-microstructure-property model for nitrocarburising in a fluid bed was established. This model is then used to control WL and DZ depths, WL phase composition and porosity structure and produce a surface treatment with properties desired for end use.

12:05 PM
Crystallization Process of Anodic Films during Post-Breakdown in the Molten Bisulphate Melt: *SungHo Han*[1]; [1]Korean Institute of Industrial Technology, Ctr. of Tech. Dvlp. & Assistance for SMEs, 472. Ka Jwa Dong, Soo-Ku, Inchon 404-254 Korea

The morphology and composition of anodic films, formed on aluminium at various current densities, in the range 1-100 A/m2, in the molten bisulphate melt at different temperatures(418-498K), have been studied using transmission electron microscopy of ultramicrotomed film sections and ion beam thinned films. When electropolished aluminium is oxidized anodically in the molten salt, the current density, I, through the anodic films shows a marked exponential dependence on the field, E, across the film. Furthermore, the dependence of the field strength on ln i at different temperatures, i. e. the Tafel slope, dE/d ln i, was found to be approximately constant with increase of temperature. The anodic films formed in the molten salt are generally barrier type and the current efficiency for film growth is approximately 100% until electrical breakdown occurs. The morphology and composition of the films formed on aluminium during post-breakdown in the molten salt have been studied by RBS and transmission electron microscopy of ultramicrotomed film sections and ion beam thinned films. On detailed examination of the anodic films, formed at 10 Am-2 in the molten salt, after breakdown for various times in the range 30 sec °≠ 20 min., two distinct phases were revealed; i. e. amorphous and crystalline alumina.

Teaching and Learning Hydrometallurgical Science and Engineering: Applied Hydrometallurgy/Thermodynamic 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

Wednesday AM — Room: 601
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Kwadwo Osseo-Asare, Pennsylvania State University, Metals Sci. & Eng., 208 Steidle Bldg., University Park, PA 16802-5006 USA; Fiona M. Doyle, University of California, Dept. Matls. Sci. & Eng., 551 Evans Hall #1760, Berkeley, CA 94720-1760 USA

8:30 AM Keynote
Teaching and Learning Environmental Hydrometallurgy: *Fiona M. Doyle*[1]; [1]University of California, Dept. Matls. Sci. & Eng., 551 Evans Hall #1760, Berkeley, CA 94720-1760 USA

Environmental hydrometallurgy can be considered to comprise two distinctly different areas: environmentally compliant hydrometallurgy, and hydrometallurgy for environmental compliance. The latter includes both hydrometallurgical waste treatment and hydrometallurgical processes that can replace existing ones with attendant environmental benefits. This approach may find application in many fields well beyond those in which hydrometallurgy is traditionally encountered. Teaching environmental hydrometallurgy is truly coupled with learning, because this field is very much in its infancy. Students need a foundational knowledge base, but must learn to use these tools to think creatively, outside of the box. Cognizance of developments in other fields is also essential. Some of the most useful unit processes and new developments are discussed, such as biohydrometallurgy, ion exchange and membrane processes. These are examined as case studies that can be used to teach students to make future developments.

9:05 AM Keynote
Wet Chemical Processing in Integrated Circuit Manufacturing: *Srini Raghavan*[1]; [1]University of Arizona, Matls. Sci. & Eng., 147 Mines & Metallurgy, Tucson, AZ 85721 USA

The manufacturing of silicon based, high-speed integrated circuits (IC) involves a large number of wet chemical steps such as etching, rinsing, drying, plating and polishing. Principles of wet chemistry, electrochemistry and surface chemistry traditionally taught in hydrometallurgy courses can be effectively used to develop modules for a course in IC manufacturing. In this presentation, hydrometallurgical aspects of wet processing will be illustrated with two examples; copper contamination of silicon from hydrofluoric acid based solutions and chemical mechanical planarization (polishing) of copper. Opportunities for the development of environmentally benign processes using wet chemical principles will be discussed.

9:40 AM Invited
Teaching and Learning Adaptive Hydrometallurgy: *Batric Pesic*[1]; [1]University of Idaho, Col. of Mines, McClure Hall, Moscow, ID 83844-3024 USA

Hydrometallurgy spans many disciplines in the science, due to its chemistry, and technology, when particular reactions are put to use on a large, well engineered, physical scale. Speaking of physical scale of application, an additional trend is emerging, however. To many, the future and beyond belongs to the nanosized products. Although vast proportion of nanoproducts is prepared by the traditional hydrometallurgical methods, the chemical reactors and the accompanying tools, dramatically changed within the last decade. Hydrometallurgy does not belong to large physical scale any longer. Neither does it rely on chemistry alone; physics is of equal importance. The question arises regarding the teaching, if a new hydrometallurgy, nano-hydrometallurgy, curriculum should be established? If so, should it be on expense of traditional hydrometallurgy? The author will present his personal observations regarding hydrometallurgy in the US within academia, industry, and government, during the last twenty years, with his own view on dilemma to take the new nanoworld path.

10:05 AM Break

10:20 AM Invited
Utilization of an Equilibrium Calculational Program for Teaching Hydrometallurgy: Hsin-Hsiung Huang[1]; Courtney A. Young[1]; *Larry G. Twidwell*[1]; [1]Montana Tech of University of Montana, Metall. & Matls. Eng., 1300 W. Park, Butte, MT 59701 USA

Equilibrium calculational software programs can be effectively utilized to illustrate the importance of concentration, solubility, temperature, solution pH, solution potential, and ligand complexation to students studying hydrometallurgy. The calculational program STABCAL will be demonstrated. Illustrative examples for the creation of diagrams such as Eh-pH, concentration/solubility-pH and concentration/solubility-Eh will be developed to understand the fundamentals of real hydrometallurgical systems. Speciation, titration and mixing will be used to simulate or predict operational processes. Applications will include treatment of Acid Mine Drainage (Berkeley Pitlake water, in particular), leaching and selective separation of metals from electroplating sludge, and flowsheet development fortreating mercury sulfide K106 sludge.

10:45 AM Invited
Aqueous Stability Diagrams for Process Design and Analysis: The ABC Approach: *Kwadwo Osseo-Asare*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., Steidle Bldg., University Park, PA 16802 USA

A systematic method is presented for using aqueous stability diagrams as engineering tools. For a diagram of the type Eh vs. pH (i.e. Eh-pH or Pourbaix diagram), three aqueous stability domains (lakes) are recognized and labeled as A, B, C, for acidic, basic, and complexing regions respectively. Dissolution or precipitation reaction paths are identified by linking the lakes and the adjacent solid phase stability regions (hills) with corresponding chemical equations. Flowsheets are then developed by using the reactants and products of the reaction path equations as feed and product materials for the respective unit process (e.g. dissolution or precipitation). Additional concepts such as dissolution/precipitation, selectivity, and redox windows are introduced and illustrated with examples. Combined hydrometallurgical/pyrometallurgical processing is also addressed.

11:10 AM Invited
Teaching Computational Thermodynamics: Wet Flue-Gas Scrubber for Power Plants and Metallurgical Furnaces: *Y. K. Rao*[1]; [1]University of Washington, Dept. of Matls. Sci. & Eng., Box 352120, Seattle, WA 98195 USA

The computation of equilibrium-state is illustrated by the flue-gas-scrubbing operations that use a slurry of lime/limestone and freshwater to convert sulfur oxides (SO2+SO3) to solid products CaSO4.2H2O and CaSO3.0.5H2O. The equilibrium compositions and the respective amounts of the gaseous, aqueous and solid phases that occur in the five-phase heterogeneous (Ca-O-C-H-S-N-Cl) system are computed by means of the iterative equilibrium constant method; the number of species (gaseous, condensed, aqueous ionic and aqueous non-ionic) exceeds thirty. The flue-gas entering the scrubber may contain CO2, O2, N2, H2O(g), NO, N2O, SO2, SO3, and HCl. The computation is presented for 25°C and 101.325 kPa or 1 atm pressure.

11:35 AM Invited
Chemical Modeling of High Temperature Aqueous Processes: *Haixia Liu*[1]; Vladimiros G. Papangelakis[1]; [1]University of Toronto, Dept. of Cheml. Eng. & Appl. Chem., 200 College St., Toronto, Ontario M5S 3E5 Canada

Chemical modeling constitutes a very powerful tool to predict and understand the behavior of complex aqueous processing systems. Needless to add is the educational aspect associated with building such models. In recent years, chemical modeling has undergone tremendous development. The new capabilities of various software have changed the way scientists approach the objective to simulate aqueous solutions in hydrometallurgical process. This paper presents new developments and strategies to simulate high temperature aqueous processing systems. The acid pressure leach of nickeliferous laterites is used as an example. Calculation of standard thermodynamic properties of aqueous species was performed with the revised Helgeson-Kirkham-Flowers (HKF) model as well as with the Density model. Nonidealities, including weak ion pair formations, were treated through the Bromley-Zemaitis ion interaction method. The above models were implemented through the OLI-Systems-μ Commercial software. Pure binary solutions like Al2(SO 4)-H2SO4, MgSO4-H2SO4, NiSO4-H2SO4,Fe2O3-H2SO4 were modeled first followed by progressively more complex solution mixtures. Finally, simulation results were compared with available experimental solubility data and very good agreement was found.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Furnace Integrity and Smelting Slags

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead, Zinc, and Tin 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

Wednesday AM	Room: 607
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Mineo Hayashi, Mitsubishi Materials Corporation, Process & Tech. Dept. Met. Co., 1-5-1 Ohtemachi Chiyoda-ku, Tokyo 100-8117 Japan; David B. George-Kennedy, Kennecott Utah Copper Corporation, 8315 West 3595 S., PO Box 6001, Salt Lake City, UT 84044-6001 USA

8:30 AM
Kennecott Flash Converting FurnaceñDesign Improvements 2001: *Chris J. Newman*[1]; Matthew M. Weaver[1]; [1]Kennecott Utah Copper Corporation, Copper Smelter, 12000 W. 2100 S., PO Box 329, Magna, UT 84044 USA

In January 1999, a redesign of the Kennecott Flash Converting furnace settler was initiated to enhance furnace integrity and extend furnace life. A detailed assessment was made of all technology available around the world before making a decision on the design features to be incorporated in the new design. The new design includes the following: redesign of all sidewall cooling elements to include the latest cooling element technology; redesign of the furnace hearth to include a stadium hearth at the reaction shaft end of the furnace; redesign of the furnace hearth to include a stepped hearth design adjacent to the furnace side walls; addition of flat settler roof cooling elements around the reaction shaft; replacement of reaction shaft transition elements with an improved design and improvements to the furnace cooling element monitoring systems. The Flash Converting furnace was shut down on a planned basis in July 2001 to implement these improvements after 29 months of operation, converting more than 1,000,000 tonnes of matte. During the 30 day shut down, the improvements were successfully implemented, with furnace start up in August 2001.

9:05 AM
Improved Technology for Water-Cooled Copper Blocks: *Allan MacRae*[1]; [1]Kvaerner Metals E&C, 12657 Alcosta Blvd., Ste. 200, San Ramon, CA 94583 USA

A new copper alloy for coils in cast copper cooling blocks has been identified and samples prepared for metallurgical examination and destructive testing to compare with cast cooling blocks manufactured from copper and nickel-copper (NiCu) alloy pipes. The new copper alloy allows for the formation of a metallurgical bond between the pipe and the cast copper during the casting process, which does not normally occur with pure copper or nickel-copper alloy pipes. The alloyís coefficient of thermal expansion is also much closer to that of pure copper compared with NiCu alloys, so there is less strain induced at the pipe-cast copper interface due to differential thermal expansion. It is expected that this new pipe coil alloy and associated casting techniques (Patents Pending) will allow for the production of cast copper cooling blocks that have greatly enhanced service life in high heat load applications compared to existing technologies.

9:30 AM
Current Practice in Taphole Design: *Melissa Trapani*[1]; Andrew Kevin Kyllo[1]; Neil B. Gray[1]; [1]The University of Melbourne, Dept. of Cheml. Eng., Parkville, VIC 3010 Australia

The removal of molten products from metallurgical furnaces is one of the most hazardous and labour intensive processes in a non-ferrous smelter. The proximity of operators to the molten metal and slag during tapping and the oxygen lancing that facilitates the flow have led to many reported injuries on tapping floors. In most cases, tapholes are opened and closed periodically leading to extreme thermal cycling in the tapping assembly. Consequently, refractory wear rates are high and unpredictable in the taphole. This paper provides a review of the current state of taphole development, and gives a methodology which is now being applied to gain a better understanding of the taphole region, to allow design improvements.

9:55 AM
Non-Isothermal Study of Refractory Dissolution by Copper Containing Calcium Ferrite Slag: *Kim Fagerlund*[1]; Tuija Suortti[1]; Pekka Taskinen[1]; [1]Outokumpu Research Oy, PO Box 60, Pori FIN-28101 Finland

A novel method for studying refractory erosion and dissolution/degradation under a temperature gradient was developed. To overcome conventional isothermal refractory testing, a new ìcold fingerî technique, based on a crucible test with a cooling element, has been developed to provide practical information on penetration and dissolution of refractory materials under cooled conditions. This technique was adopted and applied to several refractory-slag systems. Commercially available magnesia chrome refractory bricks against copper containing calcium ferrite type of slag were tested and compared using both a conventional crucible test and the new cooling method. The experiments were conducted between 1300-1420°C under a controlled atmosphere of nitrogen or air. It was noticed, that the type of porosity had a significant effect on penetration of magnesia chrome refractories. Furthermore, the copper containing calcium ferrite slag caused severe slag attack and refractory failure in some cases, due to its fluidity and corrosive nature. Characteristics of the penetration and dissolution mechanisms were analyzed from the sample microstructures.

10:20 AM Break

10:35 AM Cancelled
Wear of Magnesia-Chrome Brick in PS Converter: *Yongfeng He*

11:00 AM
Slags of Suspension Smelting of Chalcopyrite Ores and Copper Matte Converting: Heikki Jalkanen[2]; *Jaakko Poijarvi*[1]; Hanna Pajari[3]; [1]Outokumpu Harjavalta Metals Oy, Teollisuuskatu 1, Harjavalta FIN-29200 Finland; [2]Helsinki University of Technology, Lab. of Metall., Vuorimihentie 2, PO Box 6200, Espoo FIN-02015 Finland; [3]Outokumpu Pori Copper Oy, PO Box 60, Pori FIN-28101 Finland

The slag formation mechanisms in suspension smelting and matte converting affect the composition and properties of molten and solidified slags. The final mineralogical composition and morphology of solidified slags, that is of great importance for slag cleaning by froth flotation, results from the state of molten slag before solidification and processes taking place during solidification and cooling. In suspension smelting, oxidation processes take place in the reaction shaft whereas final slag and matte forms in the settler due to reactions of condensed oxidation products and slag forming agents. During slag solidification and cooling, copper and sulfur dissolved in the molten slag are rapidly precipitating in metallic or sulfide form and metals present in the raw materials are distributed in a distinct manner between soldified slag and non-silicate solidification products. Smelting and converting slags differ remarkably in their mineralogical composition and morphology despite resulting principally from the same chemical processes. Slag forming, solidification, and precipitation phenomena are discussed on the basis of mineralogical and morphology analyses of smelter slags and laboratory scale experiments.

11:25 AM
Trends in Development of Autogenous Smelting of Sulfide Materials using Oxygen-Flame Smelting Processes: V. M. Paretsky[1]; *Andrey V. Tarasov*[1]; [1]State Research Institute of Non-Ferrous Metals ìGintsvetmetî, 13, Acad. Korolyov St., Moscow 129515 Russia

There are many technical, economic, and environmental advantages to be gained by the use of a single-stage process for production of white metal. However, the changeover to a single-stage smelting process for the production of white metal requires solutions to a number of technical issues, the most serious of which is the requirement for a more stringent approach to selection of slag compositions. This approach should be based on the following major principles: homogeneity of high iron slags with high Fe^{3+}, selection of a slag composition with a new distribution of copper between white metal and slag, the need to process gold-bearing quartz flux for most smelters, the formation of slags well-suited for subsequent slag cleaning, and selection of an appropriate furnace design taking into consideration the high reactivity of the slags. At the Gintsvetmet Institute, two single-stage processes have been developed for the production of white metal and highly basic slag, including both the processing technology and the required equipment. They are based on the oxygen-flame smelting technology well suited for autogenous smelting with production of calcium ferrite slags. These versions are the oxygen-flame smelting process (KFP Process), currently being introduced at the Kovoguty Krompahy Smelter (Slovakia) and the flame-type sparged smelting process (FBP Process) being introduced at the ALmalyk Copper Smelter

(Uzbekistan). In order to provide a more rigorous basis for selection of slag compositions for autogenous smelting processes, researchers at the Gintsvetmet Institute conducted studies into the compositions and structures of slags produced on a semi-commercial scale as a result of KFP and FBP processes, as well as slag from a full-scale KFP furnace in Almalyk. The studies were carried out with the aid of mineralogical microscopy, Cameca microprobe analyses, and nuclear gamma-resonance spectroscopy. Solidified and quench slags were investigated with different cooling rates of 10^3°C/s (bar samples) and 10^6°C/s (superfast quenching by spinning-disc method). In the latter case, the structure of liquid slag was preserved (super-cooled liquid) permitting a more definite assessment of the true structure of slag in the molten state. The results obtained make it possible to give certain recommendations with respect to reduction in copper loss in slags in autogenous smelting processes.

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

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

Wednesday AM Room: 305
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: B. Gleeson, Iowa State University, Ames, IA 50011 USA; W. J. Quadakkers, Forschungszentrum J¸lich, J¸lich 52425 Germany

8:30 AM
The Effect of Moisture on Secondary Spallation of Alumina Scales on Y-Doped ReneíN5: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls. Div. 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

The single crystal superalloy ReneíN5 (with and without Y-doping or hydrogen annealing) was cyclically oxidized at 1150°C for 1000 hours. Primary spallation was observed for un-doped, un-annealed samples. However, after considerable scale growth (500 hr.), even the adherent alumina scales formed on Y-doped samples exhibited secondary delayed interfacial spallation during subsequent water immersion tests. Spallation was monitored by weight change and image analyses of the area spalled to bare metal. Hydrogen annealing (prior to oxidation) reduced spallation both before and after immersion, but without measurably reducing the bulk sulfur content of the Y-doped alloys. Only the carbon content was measurably reduced, which may account for the increased scale adhesion upon hydrogen annealing. The strain energy released was followed by acoustic emission, indicating a rapid succession of crack growth bursts ~1 m/s. A proposed failure method is proposed in which a contaminated interface allows moisture ingress and a local decrease in interfacial toughness.

9:00 AM
Effect of Water Vapor on the Oxidation Behavior of Alumina and Chromia Forming Superalloys: *K. Onal*[1]; G. H. Meier[1]; F. S. Pettit[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The oxidation behavior of several alumina and chromia forming superalloys have been examined and compared for dry air and wet air at 900°C and 700°C, both under cyclic and isothermal conditions. The results show that water vapor causes the oxidation of the alloys to be increased during cyclic exposures. The scale adherence is also affected by cyclic exposures, especially in the presence of water vapor. Under these conditions substantial amounts of oxide spallation are observed, whereas isothermal exposures in air with steam do not show the same effect. Possible mechanisms for these effects of water vapor will be proposed.

9:30 AM
Effects of Water Vapor on the High-Temperature Oxidation of Alumina-Forming Coatings and Ni-Base Superalloys: *M. C. Maris-Sida*[1]; G. H. Meier[1]; F. S. Pettit[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Superalloys develop oxidation resistance by forming protective scales of alumina or chromia during exposure to oxidizing conditions. As has been presented in the literature, water vapor has been observed to deleteriously affect the oxidation of superalloys. Isothermal oxidation experiments have been performed on a number of Ni base superalloys and coatings on these alloys in dry air and air saturated with water vapor (=0.05, 0.1 and 0.5 atm) for short times (10 minutes and 1 hour at 1100°C). Also, cyclic oxidation exposures under the same conditions were carried out for several hundreds of cycles. Results from these experiments show that the high temperature oxidation of superalloys is influenced by water in the gaseous state in two important ways: the oxide scales crack and spall sooner in air containing water vapor; and the development of continuous alumina scales is inhibited in a water vapor environment. Mechanisms will be proposed for these observed effects.

10:00 AM
High Temperature Oxidation Behavior of Some Superalloys in Steam Environment: *V. Desai*[1]; D. Tamboli[2]; [1]University of Central Florida, AMPAC, 12443 Research Pkwy., Ste. 404, Orlando, FL 32826 USA; [2]Ashland Specialty Chemical Company, R&D, 5200 Blazer Pkwy., Dublin, OH 43017 USA

For land based gas turbines, steam cooling hot gas path components offers many advantages over conventional compressed air cooling. However, superalloys are not designed to operate in steam environment at high temperatures and understanding oxidation behavior of these alloys at high temperatures warrants attention. Three superalloys with varying low to high Al content were subjected to short and long-term high temperature oxidation in flowing steam conditions, slightly above atmospheric pressure. Moderate Al content alloy showed considerable internal oxidation, weight loss and non-steady behavior even after a long exposure period. This study indicated different oxidation characteristics in steam compared to that in air. In particular, it was determined that the transition of these alloys (moderate Al content) from Chromia forming (non-protective) to Alumina forming (protective) alloy was prolonged. X-ray photoelectron spectroscopy of the surface oxide revealed a chemical difference in the air formed and steam formed oxide.

10:30 AM
Effects of Water Vapor on Oxidation of Chromium Containing Alloys at 2000°F: *M. J. McNallan*[1]; Y. S. Park[2]; [1]University of Illinois, CME Dept., MC 246, 842 W. Taylor St., Chicago, IL 60607 USA; [2]Haynes International, Eng. & Tech. Grp., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The presence of water vapor in oxidizing environments can affect the oxidation kinetics of alloys by increasing the formation of volatile species. The effect of increasing water vapor content in 5% increments from dry air to air containing 20% water vapor has been investigated for three nickel based alloys and a cobalt based alloy, all of which obtain oxidation resistance from formation of chromium oxide scales (Alloy X, Alloy 617, Alloy 188, and Alloy 230). Long term tests were performed at 2000°F with temperature cycles to room temperature at 1008-hour intervals. Both weight changes and internal attack characterization are reported. The effects of alloy composition on the behavior are discussed.

11:00 AM
Role of Water Vapour in Chromia Scale Growth at Low Oxygen Partial Pressure: *David J. Young*[1]; [1]University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney, NSW 2052 Australia

The oxidation behaviour of pure chromium and ODS-Cr alloys in Ar-H_2-H_2O and Ar-O_2-H_2O was studied at 1000°C. At high oxygen potentials, the addition of H_2O to the gas had negligible effect on scaling behavior. However, at low oxygen potentials, when the $PH_{20}PH_2$ ratio was held constant, the oxidation rate increased with water partial pressure. Increasing values of $PH_{20}PH_2$ led to more rapid rates. At fixed PH_{20} values, the rate increased with increasing PH_2. Compact scales were formed under all conditions. In addition Cr_2O_3 blades grew on the scale surface when pure chromium was reacted with H_{20}/H_2 mixtures but not in reaction with O_2H_2O. These blades did not form when Y_2O_3 dispersion strengthened material was reacted. Detailed consideration of local equilibrium between H_2O(g) and Cr_2O_3(s) led to the conclusions that oxide growth was sustained by diffusion of chromium vacancies and that adsorption of H_2O on oxide exposed to low oxygen activity gas led to formation of hydroxyl species. This model is shown to be consistent with the complex effects of gas composition on scale growth and blade formation.

11:30 AM

Water Vapor and Oxygen/Sulfur Impurity Effects on Simultaneous Oxidation and Nitridation in Cr2Nb Laves Phase Reinforced Cr: M. P. Brady[1]; *P. F. Tortorelli*[1]; L. R. Walker[1]; [1]Oak Ridge National Laboratory, Metals & Cer., MS 6115, Oak Ridge, TN 37831-6115 USA

The oxidation and nitridation behavior of Cr(Nb) solid solution and intermetallic Cr2Nb Laves phase was studied individually and in combination at 950°C or 1100°C in dry air, humid air, and N_2-$4H_2$. Greatly increased subscale nitridation was observed in Cr_2Nb-matrix alloys exposed at 950°C in both dry and humid air when the starting levels of oxygen and sulfur impurities in the alloys were reduced. At 1100°C, little effect of alloy impurity level was evident; however, exposure of Cr_2Nb-matrix alloys in humid air resulted in greatly increased subscale nitridation as compared with exposure in dry air. Pure Cr and Cr(Nb) solid solution alloys were relatively insensitive to water vapor or impurity oxygen/sulfur effects under the conditions studied. Possible synergistic effects between sulfur and water vapor and the beneficial role sulfur may play in reducing Cr_2O_3 scale permeability to nitrogen are discussed.

Advances in Metallic Glasses: Mechanical and Other Physical Properties, and Applications - 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

Wednesday PM — Room: 212
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Daniel B. Miracle, Air Force Research Laboratory, Matls. & Mfg. Direct, 2230 Tenth St., AFRL/MLLMD, Dayton, OH 45433 USA; David A. Rigney, The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

2:00 PM

High Temperature Deformation Behaviors of the Bulk Metallic Glass $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ Alloy: *Kwang Seok Lee*[1]; Tae Kwon Ha[1]; Sangho Ahn[2]; Young Won Chang[1]; [1]Pohang University of Science and Technology, Ctr. for Adv. Aeros. Matls. (CAAM), Pohang 790-784 S. Korea; [2]Research Institute of Industrial Science and Technology, Matls. & Proc. Rsrch. Ctr., Pohang 790-600 S. Korea

We have examined the deformation behaviors of a $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ bulk metallic glass that has a wide supercooled liquid region over 100K before crystallization. In compression testing in the supercooled liquid region, two types of nominal stress-strain curves were obtained. At near T_g and at strain rates higher than 10^{-3} s^{-1}, the metallic glass were fractured at maximum stress without plastic deformation. But at temperatures ranging from 401°C to 431°C, plastic deformation has been shown at strain rates ranging from $2x10^{-4}$ s^{-1} to $2x10^{-2}$ s^{-1}. Applying these compression test conditions to tensile test, superplastic deformations with maximum elongation over 500% have been observed at relatively high strain rate of $2x10^{-2}$ s^{-1} at the temperatures ranging from 401°C to 431°C. However, at the same initial strain rate, the test samples were observed to exhibit brittle fracture at the temperatures below T_g and above T_x. The superplastic properties such as flow stress and elongation were found to be very sensitive to test temperature and initial strain rate. DSC, XRD and TEM analysis for the mechanically tested specimens have been performed to investigate the changes of microstructure, thermal stability and crystallization behavior during deformations with various test conditions.

2:20 PM

Comparison of Fatigue Behavior of a Zirconium Based Bulk Amorphous Metal in Vacuum and Air: *William Hutchison Peter*[1]; R. Buchanan[1]; C. T. Liu[2]; P. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Eng. Dept., 227 Dougherty Engineering Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

Exciting recent advances have led to ìbulk amorphous materialsî (BAMs) with fabrication diameters as large as 20 mm. Although the mechanical behavior of BAMs has been studied, fatigue characteristics in controlled environments has not been well performed. Early fatigue studies have resulted in fatigue strengths lower than anticipated. It is suspected that environmental effects degrade the fatigue life. Fatigue testing of zirconium-based bulk metallic glass has been performed in a 3.5% NaCl electrolyte at room temperature. Results of these tests have shown a substantial decrease in fatigue lifetimes when compared with those from tests conducted in air. The next step toward understanding the corrosion-fatigue phenomenon is to compare fatigue results in air and vacuum. This comparison will define any detrimental effects of humidity on fatigue lifetime of a Zr-based BAM. This research was jointly sponsored by the NSF Integrative Graduate Education and Research Training (IGERT) Program on ìMaterials Lifetime Science and Engineeringî (DGE 9987548) and the Division of Materials Sciences and Engineering, US DOE under contract DE-AC05-OR22725 with UT-Battelle, LLC.

2:40 PM

Cyclic Deformation Behavior and Fatigue Fracture of Bulk Zr-Al-Ni-Cu Metallic Glass with Fully Amorphous and Partially Crystalline Structures: *Zhang Hai Feng*[1]; Zhang Qing Sheng[1]; Wu Shi Ding[1]; Ding Bing Zhe[1]; Hu Zhuang Qi[1]; [1]Institute of Metal Research, Shenyang Natl. Lab. for Matls. Sci., Wenhua Rd. 72, Shenyang, Liaoning 110016 China

This work is an investigation of cyclic deformation behavior and fatigue fracture of a bulk amorphous alloy with quenched-in crytallites. Experimental measurements indicated that fully amorphous alloy was absence of cyclic hardening or softening. For the bulk amorphous alloy with fine quenched-in crystallites had the same mode as fully amorphous alloy, neither cyclic hardening nor cyclic softening. The netlike array of shear bands for amorphous specimen. The fatigue crack initiation occurred on the surface for the fully amorphous alloy and specimen with fine crystallites. However, for the specimen with larger crystallites the crack originated from a big broken crystallite near the surface. The striation pattern on the amorphous matrix differed from that on the surface of crystallites. The average striation spacing on amorphous area was much larger than that on the crystallite.

3:00 PM

Effects of Hydrogen and Open-Volume Defects on Deformation and Fracture Behavior of Zr-Ti-Ni-Cu-Be Bulk Metallic Glass Alloys: *Daewoong Suh*[1]; Reinhold Dauskardt[1]; [1]Stanford University, Dept. of Matls. Sci. & Eng., Stanford, CA 94305-2205 USA

The effects of hydrogen and open-volume defects (free volume) on deformation and fracture behavior of Zr-Ti-Ni-Cu-Be bulk metallic glass alloys (Vitreloy 1) are presented in an attempt to better understand mechanisms for flow and fracture of metallic glasses. A range of thermomechanical behavior, including viscoelastic deformation, hardness, fracture, fatigue crack growth, glass transition and crystallization kinetics are examined with varying amounts of hydrogen and open-volume defects. The nanoscale structure of the metallic glass was examined using positron annihilation spectroscopy, high-resolution transmission electron microscopy, small-angle X-ray and neutron diffraction, and X-ray photoelectron spectroscopy. It was found that hydrogen charging and low temperature annealing increased the time scale for viscoelastic relaxation in the amorphous microstructure, both leading to sluggish atomic relaxation behavior and severe embrittlement. The reduced atomic mobility is believed to suppress stress relaxation at crack tips and hence induce brittle fracture behavior. Both hydrogen charging and annealing result in changes in the distribution of open-volume defects. Such changes are discussed in an attempt to elucidate the fundamental mechanisms of deformation and fracture.

3:20 PM Break

3:40 PM

Sliding of Metallic Glass: Experiments and MD Simulations: *David A. Rigney*[1]; Xi-Yong Fu[2]; Michael L. Falk[3]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Merck Research Laboratories, WP78-304, Sumneytown Pike, West Point, PA 19486 USA; [3]University of Michigan, Matls. Sci. & Eng., & Appl. Phys., Ann Arbor, MI 48109-2136 USA

The sliding behavior of metallic glass was studied using simple pin-on-disk tests and molecular dynamics (MD) simulations. Friction coefficients and wear rates of a Zr-Ti-Cu-Ni-Be alloy were similar to those reported for ductile materials, e.g., normal crystalline metals. Sliding caused plastic deformation, transfer, mechanical mixing and reamorphizing of devitrified material. In vacuum, a softer layer developed adjacent to the interface. In air, an additional harder layer appeared when oxidation products mixed in. MD simulations involved a two-component 2D amorphous system. Simulations of tensile tests showed elastic/perfectly plastic response, strain rate dependence and void formation. Simulations of sliding showed decreased density near the interface, suggesting an increase in free volume during shear, but no void formation. Subsurface displacement profiles were similar to those reported in experiments on crystalline materials and were consistent with flow patterns expected for flow near a boundary. The MD results on mechanical mixing suggest relevance to other processes, including mechanical alloying, friction welding, formation of nanocrystals, erosion and deformation at very high strain rates.

4:00 PM

An Instrumented Indentation Study of Mechanical Deformation in Bulk Metallic Glass: *Raj Vaidyanathan*[1]; Ming Dao[2]; G. Ravichandran[3]; Subra Suresh[2]; [1]University of Central Florida, AMPAC & MMAE, 12443 Research Pkwy., Ste. 404, Orlando, FL 32826 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Rm. 8-139, 77 Massachusetts Ave., Cambridge, MA 02139 USA; [3]California Institute of Technology, Div. of Eng. & Appl. Sci., 1200 E. California Blvd., Pasadena, CA 91125 USA

Instrumented sharp indentation experiments at the nano- and micro-length scales were carried out in an attempt to quantify the deformation characteristics of a bulk metallic glass. The experiments were accompanied by detailed three-dimensional finite element simulations of instrumented indentation to formulate an overall constitutive response. By matching the experimentally observed continuous indentation results with the finite element predictions, a general Mohr-Coulomb type constitutive description was extracted to capture the dependence of multiaxial deformation on both shear stresses and normal stresses. Constrained deformation of the material around the indenter results in incomplete circular patterns of shear bands whose location, shape and size are captured well by the numerical simulations. The surface deformation features are also consistent with mechanisms such as localized shear flow, serrated yielding and adiabatic heating, which are observed during macroscopic mechanical tests.

4:20 PM

Deformation Behavior of Bulk Ni-Based Glass Composites Containing a Ductile Metal Synthesized by Warm Extrusion of Gas Atomized Powders: *D. H. Bae*[1]; M. H. Lee[1]; E. Rozhkova[2]; D. Sordelet[2]; D. H. Kim[1]; W. T. Kim[3]; [1]Yonsei University, Metlgcl. Eng., 134, Shinchon-dong, Seodaemum-ku, Seoul 120-749 Korea; [2]Iowa State University, Matls. Sci. & Eng., 107 Metals Development, Ames, IA 50011 USA; [3]Chonju University, Phys., 36 Naedok-dong, Chonju 360-764 Korea

Ductile metal reinforced metallic glass matrix composites are prepared by warm extrusion of gas atomized powders for the purpose of improving ductility of the monolithic metallic glass. A mixture of amorphous $Ni_{59}Zr_{20}Ti_{16}Si_2Sn_3$ powders and ductile metal powders, either Cu or low-carbon steel, were extruded in the supercooled liquid region of the amorphous powder with an extrusion ratio of 5. The volume fraction of ductile metal ranged between 0 and 30%. After extrusion all powders were elongated along the extrusion direction; no porosity was visible. The extruded materials were tested under uniaxial compression. Samples containing a ductile phase exhibited slightly lower fracture strengths (e.g., to 1.85GPa for 10 vol% of Cu compared to 2.0 GPa for 0 vol%). Ductility of the composite is improved as the volume fraction of ductile metal increases due to effect on stabilization against shear localization and crack propagation. Further detailed deformation mechanism in the composite will be presented.

4:40 PM

The Role of Structural Relaxation in the Plastic Flow Behavior of Metallic Glasses: *Vitaly Khonik*[1]; [1]State Pedagogical University, Dept. of Gen. Phys., Lenin St 86, Voronezh 394043 Russia

Plastic flow of metallic glasses (MGs) is crucially dependent on the kinetics of irreversible structural relaxation (SR). For samples stored at room temperature after production, the rate of structural relaxation is very low at temperatures T < 300 to 400 K and maximal at T > 400 K. Correspondingly, one can distinguish two stages of irreversible SR: the stage of ìslowî SR and the stage of ìfastî SR. Several experiments show that MGsí plastic behavior under the ìslowî SR stage closely resembles localized flow of crystals being deformed via the dislocation mechanism. In particular, predeformed samples display a number of dislocation-like relaxations. Plastic deformation of MGs under the ìfastî SR stage is homogeneous and viscoelastic. The mechanism of this deformation should be interpreted as structural relaxation oriented by external stress. One can derive a set of simple equations describing the kinetics of homogeneous plastic flow within the framework of such notions.

5:00 PM

Phase Transformations in Metallic Glasses under Pressure: *J. Z. Jiang*[1]; [1]Technical University of Denmark, Dept. of Phys., Bldg. 307, DK-2800 Lyngby Denmark

In this talk, I will present experimental results of crystallization process in metallic glasses under pressure [1]. Several interesting aspects, e.g., crystallization temperature vs. pressure, formation of quasicrystals, time-dependent nucleation process, and TTT diagram, will be addressed. [1] J.Z. Jiang et al, Appl. Phys. Lett. 77, 3553 (2000); N. Wanderka, J.Z. Jiang et al., ibid. 77, (2000) 3935; Y.X. Zhuang, J.Z. Jiang et al., ibid. 77, 4133 (2000); ibid. 79, 743 (2001) ; N. Nishiyama, A. Inoue, and J.Z. Jiang, ibid. 78, 1985 (2001); J.Z. Jiang et al., ibid.79, 1112 (2001); J.Z. Jiang et al., Europhys. Lett. 54, 182 (2001); J.Z. Jiang et al., Phys. Rev. B. (2001) 1st September (in press).

Aluminum Reduction Technology: Inert Anodes

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

Wednesday PM — Room: 6B
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Les Edwards, CII Carbon, 1615 E. Judge Perez Dr., PO Box 1306, Chalmette, LA 70044 USA

2:00 PM

Inert Anodes: An Update: *R. P. Pawlek*[1]; [1]Technical Info Services & Consulting, Le Forum des Alpes, Ave. du Rothorn 14, CH-3960, Sierre Switzerland

During the last few years the development of inert anodes for the primary aluminium industry has advanced considerably and announcements have influenced the stock market. This update reviews results of tests on (1) anodes made of ferrites (Ni, Co and Zn) alone or in combination with nickel oxide and copper leading to the final composition Ni1-x-yFe2-xMyO4, where M is zinc and/or copper, x is from 0 to 0.5 and y is from 0 to 0.6; (2) anodes made of oxides (Ni, Li, Sn, Sb, Cu) alone or in combinations; and finally (3) anodes made of combinations of metal powders including Ni, Al, Fe, Cu and Zn. The studies examined especially solubility in cryolite alumina melts, but also performance in electrolysis tests from which the results are discussed. Solubility laboratory tests revealed that compositions with limited corrosion (1-2 cm/y) exist and could be successful not only in laboratory but also in industrial cells.

2:25 PM

Electrolysis and Post-Testing of ìInertî Cermet Anodes: *Odd-Arne Lorentsen*[1]; Jomar Thonstad[2]; [1]Norsk Hydro ASA, Rsrch. Ctr., Matls. Dvlp., PO Box 2560, N-3907 Porsgrunn Norway; [2]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Sem Selandsv. 6, N-7491 Trondheim Norway

Stoichiometric nickel ferrite cermet anodes with 17% copper metal added, making a cermet, were electrolysed for 50 hours in alumina-saturated cryolite-based bath. Bath samples were frequently withdrawn from the cell and analysed with respect to the anode constituents. Steady-state concentrations of iron and copper were below their solubility concentrations, while the nickel concentration was a little higher. Precautions had to be taken to avoid unwanted precipitation of solid alumina and cryolite at the cathode. Post-tests of the anodes showed that copper was depleted in the outer zone of the anode, and the composition of the copper-rich alloy (Cu-Ni-Fe) was changing from the interior toward the surface. However, the metal-depleted surface seemed to be dense, and no voids/pits from the lost metal grains were found in this region.

2:50 PM

The Development of a Metallic Anode and Wettable Cathode Coating and their Tests in a 20-kA Prototype Drained Cell: *Curtis McMinn*[1]; Olivier Crottaz[2]; Vittorio BellÚ[2]; Thinh Nguyen[2]; Vittorio de Nora[2]; [1]2125 Chickasaw Dr., Florence, AL 35630 USA; [2]MOLTECH Systems, Ltd., c/o MOLTECH S.A., 9 Rte. de Troinex, Carouge/GE Switzerland

The MOLTECH Group has tested a large range of non-carbon metallic anode materials and designs, modeling the more promising options prior testing them for their retrofittability into drained configuration cells using the required wettable cathode coating developed by MOLTECH and extensively tested in commercial cells of many types. Extensive experience has been gained in design and operating conditions, as MOLTECH has moved to successful testing and operation of a prototype 20-kA cell. This paper will present the results of this research and development work, including a discussion of the experience gained in starting up and operating Non-Carbon Metallic Anodes at the 20-kA cell size, superstructure design issues for retrofitting Hall-HÈroult cells into a drained cell configuration, and cell monitoring and control issues. It will conclude with a resume of the status of the cell and anode tests, discussing some of the major problems which have been solved and which are still to be solved.

3:15 PM
Fate of SOFC-Type Inert Anode for Production of Primary Aluminum: *Robert A. Rapp*[1]; Yunshu Zhang[1]; [1]The Ohio State University, Dept. Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Research supported by the DOE-OIT has evaluated the possibility to use yttria-stabilized zirconia tubes, supplied internally with a metallic anode and fed with reformed natural gas, as an inert anode for retrofit in the Hall-Herault Cell. In principle, electrically-driven titration of oxide ions from alumina-fed cryolite through the oxide-ion conducting electrolyte tubes would lead to a confined anodic reduction site providing significant reductions in cell voltage, cost of production, and release of greenhouse gases. Detailed studies of the solubilities of alumina, zirconia and yttria over a very wide range of cryolite bath ratios (acid-base properties) showed that no bath composition exists where alumina would be sufficiently soluble while the electrolyte components were sufficiently insoluble. Therefore, the SOFC-type anode is not chemically compatible with any cryolite-base solvent system. Solid-state probes were developed which indicated the acid-base state of any cryolite-base melt, and clarified the role of complex anion formation.

3:40 PM Break

3:50 PM
An Alternate Fused Electrolyte/Solvent Suited to the SOFC-Type Anode for Electrowinning Aluminum: *Robert A. Rapp*[1]; [1]The Ohio State University, Dept. Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

In cryolite-base melts, the acid-base range is greatly restricted by the formation of complex oxyfluoride anions. But oxides dissolve in fused sodium sulfate melts to form uncomplexed oxide anions over a wide acid-base range. Extensive previous measurements of oxide solubilities in fused sodium sulfate at 1200K show that in a very basic melt (where NaAlO2 becomes stable) the solubility of alumina is about 10 mol %, while those for zirconia and yttria are very much lower. Therefore, at a temperature suited to the YSZ electrolyte, the chemical conditions required for the SOFC-type inert anode should be satisfied. However, liquid Al is not compatible with fused sodium sulfate, but should react to form a sodium-aluminum sulfide ìmatteî. A preliminary experiment indicated very slow reaction between Al and sodium sulfate, and the YSZ electrolyte is known to support oxide ion titration under such melt conditions. Therefore,the benefits from a SOFC-type inert anode might be realized upon a change in the solvent for alumina to sodium sulfate. Novel modular vertical anode geometries are suggested.

4:15 PM Cancelled
The Corrosion Rate of Cermet Inert Anode with Variety Scale in Aluminum Electrolysis: *Zhao Qun*

Aluminum Sheet and Plate Rolling & Finishing Technology and Applications: Aluminum Rolling-Metallurgy and Microstructure

Sponsored by: Light Metals Division, Aluminum Association,
Program Organizers: Michael H. Skillingberg, The Aluminum Association, Inc., Washington, DC 20006 USA; John P. Brandimarte, Precision Coil, Inc., Clarksburg, WV 26302-2650 USA; Karl Eminger, Alcoa, Inc., Alcoa Mill Products, Lancaster, PA 17604 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Jeff J. Kadilak, Alcan, Inc., Oswego, NY 13126 USA; Leland R. Lorentzen, Nichols Aluminum, Davenport, IA 52801 USA; Douglas N. McLeod, ARCO Aluminum, Inc., Louisville, KY 40242 USA; Ken Schreckengast, Coastal Aluminum Rolling Mills, Inc., Williamsport, PA 17701 USA

Wednesday PM Room: 608
February 20, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: J. Daniel Bryant, Alcoa, Inc., Alcoa Technical Ctr., 100 Technical Dr., Alcoa Center, PA 15069-0001 USA; Michael H. Skillingberg, The Aluminum Association, Inc., 900 19th St. N.W., Ste. 300, Washington, DC 20006 USA

2:00 PM
Texture Evolution in Cold Rolled Direct Chill Cast and Continuous Cast Aluminum Alloys: *Wenchang Liu*[1]; Xiyu Wen[1]; Tongguang Zhai[1]; James G. Morris[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

The hot bands of direct chill cast (DC) and continuous cast (CC) AA 1100 aluminum alloys were annealed at 399C for 3 hours, and the hot bands of DC and CC AA 5182 aluminum alloys were annealed at 454C for 3 hours. The annealed hot bands were then cold rolled to different reductions. The ODFs of the cold rolled samples were determined by X-ray diffraction in order to compare the texture evolution of DC and CC aluminum alloys during rolling. The texture volume fractions were computed by a new method, in which the Euler space representing all possible crystallographic orientations in rolling was subdivided into the cube, r-cube, Goss, r-Goss, beta fibre, and random orientation regions based on the slip pattern combined with the characteristics of microstructure and texture. Empirical formulas of the texture volume fractions and true strain were constructed to predict the texture in cold rolled aluminum alloys. The effect of the process method (DC vs. CC) and Mg content on the texture after annealing and the texture evolution during the subsequent cold rolling is discussed.

2:25 PM Cancelled
The Evolution of Microstructure and Texture of DC AA3004 Aluminum Alloy through Different Previous Processes of the Hot Band: *Xiangming Cheng*

2:50 PM Cancelled
Microstructure and Local Crystallography of Rolled Al-1%Mn Single Crystals of {001}<110> Orientation: Zhengjie Li

3:15 PM Cancelled
Simulating the Deformation Texture Evolution of Aluminum Alloys during Cold Rolling by using Artificial Neural Network: *Jiantao Liu*

3:40 PM Break

3:55 PM
On the Orientation Hardening and Softening of Strip Cast Sheet of AA5052 Aluminum Alloy during Hot Rolling and Cold Rolling: *Xiyu Wen*[1]; Wenchang Liu[1]; Tongguang Zhai[1]; Jiantao Liu[1]; Xiangming Cheng[1]; J. G. Morris[1]; [1]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

In this paper, an investigation of the work-hardening characteristics of strip cast sheet of AA5052 aluminum alloy during hot rolling and cold rolling is carried out using X-ray crystallographic data. The texture changes during hot rolling and cold rolling reductions were followed by changes in the orientation distribution function (ODF). The orientation hardening and softening caused by a change in the lattice reorientation can lead to either hardening or softening of the matrix, and can be separated from normal dislocation hardening at the grain level. A numerical method is used to derive the rate of orientation hardening and softening and to determine six individual texture components (Cube, Rotated Cube, Goss, Brass, S and Copper components) directly from the ODF data in the alloy sheet. The developments of strain instability during hot rolling and cold rolling are also studied at the grain level. The difference of orientation hardening and softening of the alloy sheet between hot rolling and cold rolling is analyzed and discussed. The significance of orientation hardening and softening on the instability of the sheet metal during hot rolling and cold rolling is also discussed.

4:25 PM
Development of a High Strength Fin Stock Aluminium Alloy: Part II: *Scott W. Haller*[1]; A. Burger[2]; K. Vieregge[2]; A. Haszler[2]; A. Wittebrood[3]; J. van der Hoeven[3]; [1]Corus Aluminum Rolled Products, 9523 Chipping Dr., Richmond, VA 23237 USA; [2]Corus Aluminium Walzprodkte GmbH, Carl-Spaeter-Strasse 10, 56070, Koblenz Germany; [3]Corus Research, Development & Technology, Postbus 10000 (3G16), 1970 CA, IJmuiden The Netherlands

The emphasis in the automotive industry toward heat exchanger units with reduced overall weights and decreased sizes (compact designs) has created a trend for heat exchanger materials with improved performance characteristics. This trend, coupled with the continuous changes in heat exchanger coolants and refrigerants requiring increased system pressure capabilities, has resulted in the development of new materials with higher strengths and improved corrosion resistance characteristics-targeting all components of modern automotive heat exchangers. In order to meet these demands, Corus Aluminium has developed new families of improved alloys for heat exchanger tubes and core plates that are suitable for vacuum and/or controlled atmosphere brazing (CAB). To further complete this development, a program was started to develop a series of high strength alloys for heat exchanger fin applications. This paper presents an overview of the latest laboratory developments and details the plant production trials of the improved Corus Aluminium fin stock alloys.

4:55 PM
A Measurement Capability Study Comparing the Effect of Lubricant Type on Earing of Can Body Stock: *Gary J. Mahon*[1]; Ed Creed[1]; *Jeff J. Kadilak*[2]; [1]Alcan International, Ltd., Banbury Lab., Southam Rd., Banbury, Oxfordshire OX16 2SP UK; [2]Alcan Sheet Products, Oswego Works, PO Box 28, Oswego, NY 13126 USA

The earing test is an important material evaluation technique for sheet products which are to be used in applications where the material will be subjected to a drawing operation. Historically, the most important of these is the draw and re-draw which occurs during manufacture of can bodies. The height of the ears produced during drawing is not only a function of the crystallographic texture of the sheet, but is also strongly affected by the conditions used during the cup drawing operation itself. These include the die clearances, drawing speed, clamping load and surface friction. This paper reports the findings of a statistical study into the reproducibility of the earing test measurements on cups drawn using petroleum jelly as the lubricant (Vaseline) and an industrial grease (Esso Unirex EP2). The study will demonstrate the importance of lubricant type on test reproducibility and the value of the Measurement Capability methodology.

Cast Shop Technology: Grain Refining & Filtration

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

Wednesday PM	Room: 6A
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Jean-Pierre Martin, Alcan, Arvida Labs. & Dvlp. Ctr., 1955 Mellon Boul., Jonquiere, Quebec G7S 4K8 Canada; Jennifer Black, Vesuvius, PO Box 788, Alfred, NY 14802 USA

2:00 PM Keynote
Grain Refining of Commercial Aluminum Wrought Alloys: *Men Glenn Chu*[1]; [1]Alcoa, Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

Grain refining of aluminum alloys using aluminum-based master alloys containing inoculant particles is practiced daily in most ingot casting plants. The main reasons for grain refining are to reduce ingot cracking during casting and improve ingot fabricability during subsequent thermal mechanical processing. Industrial grain refinement practices are often established based on past experience and not necessarily optimized. Much progress has been made in recent years in the understanding of mechanism of grain refinement and the characteristic of the Al-Ti-B grain refiners. These advances now enable the establishment of grain refining practices for industrial applications based on a scientific basis. In this paper, an overview of current understanding on the mechanism of grain refinement will be presented. The effect of solidification conditions on the effectiveness of grain refiners and the impact of the grain refiner product characteristics on the ingot metal quality will be discussed. The trend of adapting commercial Al-Ti-C grain refiner for critical product applications and recent development of new grain refining techniques including gas-liquid in-situ reaction and acoustic cavitation will also be reviewed.

2:30 PM
A Comparison of Grain Refiner Master Alloys for the Foundry: *D. A. Boot*[1]; P. S. Cooper[1]; D. H. St John[2]; A. K. Dahle[2]; [1]London and Scandinavian Metallurgical Company, Ltd., Aluminium, Fullerton Rd., Rotherham, S. Yorkshire S60 1DL England; [2]University of Queensland, Dept. of Mining, Minls. & Matls. Eng., St Lucia, Queensland QLD 4072 Australia

Grain refiners are widely used in the foundry. They are considered to provide benefits in a number of ways including improved feeding during solidification, reduced and more evenly distributed porosity, and reduced hot tearing. There are a number of refiners for the foundry industry used industrially or mentioned in the literature. A programme of laboratory tests has been carried out, which started with a prescreening of a wide range of potential refiners, using standard laboratory grain refining tests. The better performing refiners were then tested in a permanent mould casting, designed to simulate conditions at the spoke-rim junction of a wheel casting. Results are presented in terms of the effect of refiner on grain size, hot tearing and porosity distribution. Recommendations are made in terms of the optimum refiners for the foundry.

2:55 PM
Investigation on the Grain Refining Behaviors of Al-Ti-C Grain Refiners from Different Suppliers: *Tzu-Hsin Wang*[1]; Kuo-Feng Hsu[1]; Chin-Sheng Chan[1]; [1]China Steel Corporation, Steel & Aluminum R&D, PO Box 47-111, Hsiao Kang, Kaohsiung 81233 Taiwan

From the point of practice it was interesting to know that whether the same type of Al-Ti-C grain refiners from different suppliers provided the same grain refining behaviors or not. The behaviors of commercially available Al-3Ti-0.15C grain refiners from two famous suppliers were investigated using various adding amount, melt temperature and holding time. It was found that the grain refining ability with adding amount of the two grain refiners showed different tendency. Their responses to the aluminum melt temperature were also different. One was sensitive to the melt temperature, the other one was not. One of them showed temperature dependent progressive fading of grain refining ability while held in the melt for a long time, the fading of the other one was not temperature dependent. It was interesting to know that their fading of grain refining ability could not be regained by stirring the melt.

3:20 PM
The Performance of TiB2 and TiC Bearing Grain Refiners in Twin Roll Strip Casting: *Y¸cel Birol*[1]; Murat Dundar[2]; Selda Ucuncuoglu[1]; Osman Cakir[1]; Abdullah Soner Akkurt[2]; [1]Marmara Research Center, Matls. Technologies, Gebze, Kocaeli 41470 Turkey; [2]Assan Aluminum, Proc. Dvpt., Tuzla, Istanbul Turkey

The master alloys based on the Al-Ti-B system have been used extensively for grain-refining aluminum alloys(1). The TiB2 particles in commercial Al-Ti-B master alloys, however, are rather coarse and have a tendency to agglomerate both during the manufacturing process and after being released into the melt, leading to a number of quality problems. These quality-related problems have created a big demand for an acceptable replacement for Al-Ti-B master alloys. The Al-Ti-C system has thus received particular attention as TiC has been assumed for a long time to be the nucleant for aluminum treated with hypo-peritectic Ti concentrations. TiC particles are claimed to be smaller than the TiB2 particles and are less prone to agglomeration. The Al-3%Ti-0.15%C alloy of this new generation grain refiners have been used commercially for several years in a wide range of alloy systems. Much of the work reported on this alloy, however, has been from laboratory scale testing while data on commercial applications is rather scarce. In the present work, commercially available Al-3%Ti-0.15%C grain refiner was employed in the twin roll strip casting of Al-Fe-Si alloys. Its grain refining potency was compared with that of the Al-5%Ti-1%B master alloy, the standard grain refiner in the aluminum industry.

3:45 PM Break

4:00 PM
The Effects of Rod Grain Refiners with Differing Ti/B Ratio on Ceramic Foam Filtration: *Nicholas Grant Towsey*[1]; Wolfgang Schneider[1]; Hans-Peter Krug[1]; [1]VAW Aluminium AG, Georg-von-Boeselager-Str. 25, Bonn 53117 Germany

The effects of 3%Ti-1%B and 3%Ti-0.15%C rod grain refiners on ceramic foam filtration was previously presented. It was shown that such grain refiners apparently change the filtration behavior leading to lower average filtration efficiencies. The present paper reports the results of follow up investigations into the effects of other grain refiner compositions, viz. 3%Ti-0.2%B, 1.2%Ti-0.5%B and 5%Ti-1%B, on the efficiencies of 50ppi ceramic foam filters. In general terms, it can be said that the new findings tend to support the contention that the alteration of filtration behavior is associated with the boride phase of the Ti-B grain refiners. Together with parallel investigations on relative grain refining potency, an ultimate goal may be to add grain refiner of a certain composition at a lower rate after a ceramic foam filter providing a consistently high quality grain refiner can be supplied. The desired balance may then be realized of optimized ceramic foam filter performance, consistently low inclusion content of metal delivered to the direct chill mould and lowest possible grain refiner costs.

4:25 PM
LiMCA Comparison of a Bed Filter and a Two Stage Ceramic Foam Filter: *D. Corleen Chesonis*[1]; David H. DeYoung[1]; David E. Lake[2]; Neil R. Ridler[2]; [1]Alcoa, Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]Alcoa Manufacturing G.B., Ltd., Waunarlwydd Works, Swansea SA1 1XH Wales

The filtration performance of a bed filter and a two stage ceramic foam filter were compared based on LiMCA inclusion measurements. The bed filter uses a packed bed of alumina mesh and balls to remove inclusions. The two stage filter uses two ceramic foam filter plates; a coarser pore filter is fitted directly over a finer pore filter with a small gap between the two plates. The objective is to use the coarser filter to remove large inclusions so the finer filter can remove more and smaller inclusions without plugging. The main advantage of the two stage filter is to avoid downtime and recovery loss from having to flush the bed filter for alloy changes. LiMCA data was obtained with the bed filter for nine casts and with the two stage filter for five casts. Casts with the two stage filter included three ppi combinations and two filter factors.

4:50 PM Cancelled
Investigation of Inclusion Re-Entrainment during Filtration: *Duygu Kocaefe*

5:15 PM
Metal Quality Analysis at a Continuous Twin Roll Caster: *Edward M. Williams*[1]; Lawrence D. Ray[2]; Tom F. Higdon[3]; [1]Alcoa, Ingot, 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]Alcoa, Flexible Pkgg. R&D, 2101 Reymet Rd., Richmond, VA 23237 USA; [3]Alcoa, Hot Spring Continuous Rolling Plant, Hwy. 270, Jones Mill, AR 72105 USA

LiMCA, Alscan and PoDFA measurements of inclusion and hydrogen concentrations were made at the Hot Springs Continuous Rolling plant casthouse. The purpose of this work was to evaluate the metal quality effects of dramatically reducing and/or eliminating the chlorine used in the in-line degasser. In addition to the reduced chlorine tests, the use of a bonded particle vertical filter, which can be changed in situ during casting, was evaluated. The trial took place at a twin roll continuous caster.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: The Effect of Segregation on Environmental Cracking

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

Wednesday PM	Room: 307-308
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Harvey B. Solomon, General Electric, Dept. of Matls. Sci. & Eng., Schenectady, NY USA; David L. Pope, University of Pennsylvania, Dept. of Matls. Sci. & Eng., Philadelphia, PA 19104 USA

2:00 PM Invited
Hydrogen Segregation at Interfaces and its Role in Embrittlement: *Howard K. Birnbaum*[1]; [1]University of Illinois, Matls. Rsrch. Lab., Urbana, IL 61820 USA

Impurity segregation to interfaces generally (but not in every case) has the effect of decreasing the strength of the bonding across that interface and increasing the probability of interfacial fracture. Professor McMahonís research has shown the importance of impurity segregation in determining the mechanical properties and fracture of metals. One important case, that has not been extensively studied is the segregation of hydrogen to interfaces and in particular to grain boundaries. This presentation will review what is known about the segregation of hydrogen to grain boundaries and will relate this segregation to the embrittlement of a number of model systems. The emphasis will be on using the information available to discuss the mechanisms by which hydrogen embrittlement occurs at grain boundaries. It will be shown that hydrogen segregation at grain boundaries plays an important role in at least two mechanisms of hydrogen embrittlement. In one case, NiñSñH, the synergistic effects of S and hydrogen segregation will be discussed.

2:35 PM Invited
Effects of Grain Boundary Chemistry and Microstructure on the Intergranular Stress Corrosion Cracking of an Al-Mg Alloy: *Russell H. Jones*[1]; Donald R. Baer[1]; Michael J. Danielson[1]; Valery Y. Gertsman[1]; John S. Vetrano[1]; Charles F. Windisch[1]; [1]Pacific Northwest National Laboratory, Matls. Scis., PO Box 999, Richland, WA 99352 USA

Low strength Al-Mg alloys, such as AA5083, are susceptible to intergranular stress corrosion cracking (IGSCC) with thermal aging. Grain boundary chemistry and microstructure measurements have been made using AES and AEM/HRTEM and the role of Mg and Cu on corrosion and stress corrosion measured by XPS and stress corrosion crack growth analysis. Grain boundary enrichment of Mg increases from a ratio of 1.4 to 2.5 upon aging from 1 hr to 100 h at 175C, while grain boundary enrichment of Cu is observed in the solution treated condition and reaches a level of 10 after 100 h at 175C. During this same thermal treatment there is an increase in the volume fraction of the beta phase (Al3Mg2) precipitated at grain boundaries. Precipitation proceeds from triple points after 1 h at 175C to more randomly along the grain boundary after 100 h. Corrosion studies of Al implanted with Mg and Cu performed in an XPS system with an in situ corrosion cell showed that both Mg and Cu contribute to the corrosion rate of Al. Increased IGSCC rates occur coincident with these grain boundary microchemistry and microstructure changes. These results will be discussed in terms of anodic dissolution and hydrogen induced crack growth mechanisms for low strength Al-Mg alloys. Research supported by the Office of Basic Energy Sciences of the US Dept. of Energy.

3:10 PM
High Temperature Strength of Two Cryomilled Aluminum Alloys: B. Dehiya[1]; K. Tsuchiya[2]; *J. R. Weertman*[1]; [1]Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA; [2]Toyohashi

University of Technology, Production Sys. Eng., Toyohashi, Achi 441 Japan

Cryomilled aluminum alloys have been found to retain considerable strength at elevated temperatures. Small dispersoids of aluminum oxynitride particles in the tens of nm size range stabilize the microstructure to temperatures approaching the melting temperature. Al-Al2O3 and Al-Al3Ti cryomilled alloys showed little strain hardening and even strain softening, with yield point behavior at elevated temperatures. Strain to failure at elevated temperatures is strain rate sensitive and fracture appears to switch from ductile to intergranular, especially in the Al-alumina material, as the strain rate drops. This research was partially sponsored by Exxon.

3:30 PM Invited
Grain Boundary Segregation and Precipitation on Intergranular Stress Corrosion Cracking of Austenitic Stainless Alloys in High-Temperature Water Environments: *Stephen Michael Bruemmer*[1]; [1]Pacific Northwest National Laboratory, Matls. Interfaces & Characterization, PO Box 999, Richland, WA 99352 USA

Environment-induced intergranular degradation continues to be a critical issue for Fe- and Ni-base stainless alloys in high-temperature water. While the understanding of sensitization has eliminated many problems in oxygenated environments, intergranular stress corrosion cracking remains a common mode of component failure. Grain boundary composition has not been directly linked to degradation in non-sensitized stainless alloys, but considerable new data is available demonstrating complex interactions among segregation, precipitation and environmental cracking. This paper will review and highlight current understanding of grain boundary compositions in Fe- and Ni-base stainless alloys due to both equilibrium and non-equilibrium processes. Beneficial and detrimental segregation of alloying and impurity elements will be discussed along with the influence of second-phase precipitation. High-resolution observations of crack-tip corrosion reactions will be used to illustrate complex effects of local chemistries and precipitate phases on crack advance.

4:05 PM
Analysis of Hydrogen-Induced Decohesion at a Particle/Matrix Interface: *Petros Sofronis*[1]; Yueming Liang[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Theoretl. & Appl. Mech., 216 Talbot Lab., 104 S. Wright St., Urbana, IL 61801 USA

Hydrogen-induced decohesion along grain boundary carbides is a form of material degradation observed experimentally in Ni-base alloys. This decohesion leads to void formation along grain boundaries accompanied by subsequent ductile shearing of the ligaments between voids, thereby causing an intergranular crack to form and propagate. In this paper, the transient hydrogen diffusion around a precipitate is simulated in conjunction with the plastic and elastic deformation correspondingly in the matrix and the particle. The finite element method is used to solve the relevant boundary value problem allowing for decohesion to occur along the particle/matrix interface, and calculate the hydrogen distribution around the precipitate. The thermodynamic theory of Rice and Hirth specialized to the regime of ìfast decohesionî is used to describe the interfacial traction-separation constitutive law. The numerical results show that even at small initial hydrogen concentrations, hydrogen can weaken the macroscopic behavior of the material even under ìfast decohesionî conditions.

4:25 PM
On the Correlation between Grain Boundary Segregation, Faceting and Embrittlement in Bi Doped Copper: *Wilfried Sigle*[1]; Li-Shin Chang[2]; Wolfgang Gust[1]; Manfred R¸hle[1]; [1]Max-Planck-Institut fuer Metallforschung, Seestrasse 92, D-70174 Stuttgart Germany; [2]National Chung-Hsing University, 250, Kuo-Kuang Rd., 402 Taichung Taiwan

Copper Σ19a {331} bicrystals grown by the Bridgman technique have been doped with 14, 25 and 65 at.ppm Bi, respectively. After annealing between 600 and 800∞C the segregation was quantified using energy dispersive X-ray spectroscopy in a dedicated scanning transmission electron microscope. Grain boundary faceting on low-index planes was observed and the segregation level was significantly higher at the facets compared to the non-faceted segments. The fraction of faceted grain boundaries was found to increase with increasing annealing temperature implying that the faceting transition is thermally activated. Only the completely faceted grain boundary exhibits brittle behaviour. Faceting appears to be a necessary prerequisite for grain boundary embrittlement of the Σ19a {331} grain boundary by the fact that it allows to incorporated enough Bi atoms. It is suggested that this embrittling mechanism may be a general phenomenon in Bi-doped Cu.

4:45 PM
Corrosion and Stress Corrosion Cracking of Al-Mg Alloys: *Clyde Briant*[1]; Sharvan Kumar[1]; Ping Wang[1]; Zhengfu Wang[1]; [1]Brown University, Div. of Eng., 182 Hope St., Providence, RI 02912 USA

Aging Al-Mg alloys can make them very susceptible to corrosion and stress corrosion cracking because of the precipitation of beta-phase along the grain boundaries. This phase is anodic with respect to the matrix in these alloys and is preferentially corroded. In this paper we examine the effect of small additions of scandium and silver on this intergranular corrosion. Corrosion tests were run by placing the samples in nitric acid for one day and stress corrosion tests were performed by pulling the samples slowly to failure in an NaCl solution. Both sets of tests show that the scandium containing alloy and the alloy that contained neither addition had greater corrosion resistance than the alloys that contained silver. These results are interpreted in terms of the difference in the amount of beta phase precipitated along the grain boundaries.

5:05 PM
On the Stabilization of Grain Boundaries by Solute Segregation: *Reiner Kirchheim*[1]; [1]Universit‰t G^ttingen, Inst. f¸r Materialphysik, Hospitalstr. 3-7, G^ttingen D-37073 Germany

It will be shown that a certain total area of grain boundaries is in metastable thermodynamic equilibrium in the presence of solute atoms and, therefore, grain coarsening is stopped as there will be no driving force. This is in contradiction to the generally accepted interpretation, where solute drag, i.e. zero mobility of the boundaries stops grain coarsening. Equations for the grain boundary area or the grain diameter, respectively as a function of temperature and solute concentration will be derived based on the excess solute concept of Gibbs. The resulting equations are compared with experimental results. Detailed information about the segregation of phosphorous to grain boundaries in nanocrystalline nickel is obtained from measurements using the tomographic atom probe.

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

Wednesday PM	Room: 401
February 20, 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: Wednesday 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

Wednesday PM	Room: 619-620
February 20, 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

Keynote
Computational Modeling of Thermo-Mechanical Phenomena: *Christopher Bailey*[1]; [1]University of Greenwich, 30 Park Row, Greenwich, London SE10 9LS UK

Many industrial processes involve materials that are subject to temperature change and thermal stress. Examples range from the casting of large metallic products to the cooling and reliability of small electronic components. Thermally induced stress is a major concern as it can lead to material damage and product failure. Material properties, thermal and mechanical, and process conditions such as the size and location of a feeder in metals casting, or the power dissipation in a computer chip, will govern the magnitude of these stresses. Temperature may also be influenced by fluid flow, for example, the airflow over a computer chip in a laptop will govern the rate of heat extraction, hence the temperature gradients in the chip and the evolving thermal stresses. This paper provides details on the governing equations for heat transfer (temperature) and solid mechanics (stress), their degree of coupling, and the numerical techniques used to solve them. Three examples are discussed to illustrate the use thermomechanical modelling. These also provide an insight into the degree of coupling required between the equations. The first example, involves thermal cycling of electronic components where a prescribed temperature field is applied. As temperature is known, this example only requires the solution of the stress equation. The second example also involves the modelling of an electronic component where the temperature field is also calculated. In both of these examples, the stress calculation is dependent on the temperature field, but the temperature calculation is not dependent of the solution of the stress equation (one-way coupling). The final example provides details on modelling the metals casting process. Both the temperature and stress equations are solved but, unlike the previous two examples, in this case the temperature field is dependent on the results from the stress calculation (two-way coupling).

Computational Modeling of Materials, Minerals & Metals Processing:Thermo-Mechanical Modelling - 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

Wednesday PM	Room: 619
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Chris Bailey, University of Greenwich, 30 Park Row, Greenwich SE10 9LS UK; Brian G. Thomas, University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., Urbana, IL 61801 USA

2:45 PM
Mathematical Modeling of Mean Flow Stress during Hot Strip Rolling of Nb Microalloyed Steels: *Ki Bong Kang*[1]; Sang Hyun Cho[2]; John J. Jonas[2]; [1]POSCO, Rsrch. Lab., 1 Goedong-dong Nam-gu, Pohang, Kyungbuk 790-785 S. Korea; [2]McGill University, Dept. of Metall. Eng., 3610 University St., Montreal, Quebec H3A 2B2 Canada

The recrystallization behavior of Nb microallyed steels was studied using hot torsion testing with the aim of modeling the recrystallization processes taking place hot rolling. It is shown that the recrystallization kinetics depend on steel composition and processing conditions. The stress-strain curves were determined in order to derive new equations for the peak stress, peak strain, mean flow stress and softening kinetics. The peak strain is influenced by the presence of alloying element; their addition, which has a solute effect, retards the rate of grain boundary motion, shifting the peak to the right. The addition of Ni to the steel results in a significant increment in the activation energy for hot deformation, but Cr has the opposite effect. It was also found that the deformation activation energy in these steels was not altered by the addition of Mo and Nb. The kinetics of static and metadynamic recrystallization were characterized and appropriate expressions were formulated for the recrystallization kinetics. The rate of metadynamic recrystallization increases with strain rate and temperature and is observed to be independent of strain, in contrast to the observation for static recrystallization.

3:10 PM
Modeling of Residual Stresses and Mechanical Behavior of Glass-Infiltrated Spinel Ceramic Composites: *Anil Saigal*[1]; Edwin R. Fuller[2]; Said Jahanmir[2]; [1]Tufts University, Mechl. Eng., 200 College Ave., Medford, MA 02155 USA; [2]National Institute of Standards and Technology, Cer. Div., Bldg. 223, Gaithersburg, MD 20899 USA

All-ceramic crowns, including glass-infiltrated alumina and spinel composites, are coming into widespread use because of their superior aesthetics and chemical inertness. This study investigates the residual stresses that are developed in these composites as a result of cooldown from the glass-infiltration temperature to room temperature due to slight mismatch in the coefficients of thermal expansion and its effect on the mechanical behavior of these composites. Two-dimensional finite element simulations were performed using an object oriented finite element program OOF. The OOF program is a combination of two programs. The first program, PPM2OOF, is designed to read an image file such as a micrograph. The individual pixels that constitute the micrograph may be collected into groups and their material properties assigned. PPM2OOF is then used to create the finite element model/mesh that OOF then reads. The average residual stresses are found to be tensile in the alumina and spinel matrix and compressive in the infiltrated-glass. There is large variation in residual stresses and strains from location to location with presence of locations at which the glass is under tensile stress. The crack initiation and initial propagation in glass-spinel composites is at the glass-spinel interface in both the glass and the spinel. The presence of residual stresses can lead to lower crack initiation stresses and degrade the mechanical properties of the composites.

3:35 PM Break

3:50 PM
Simulation of Fatigue Stress Life (S-N) Diagrams for Ti-6Al-4V Alloy by Application of Artificial Neural Network: S. McShane[1]; *Savko Malinov*[1]; J. J. McKeown[1]; Wei Sha[1]; [1]The Queens University of Belfast, Sch. of Civ. Eng., Belfast, Northern Ireland BT7 1NN UK

A model is developed for prediction of fatigue stress life S-N diagrams for Ti-6Al-4V alloy at different conditions, using Artificial Neural Network (ANN). Different factors which have influence on the S-N diagram are involved as input parameters of the NN, namely microstructure, environment, texture, test/work temperature, surface treatment and stress amplitude. As the output the S-N fatigue diagram is simulated and plotted. The model is based on standard multilayer feed forward neural network. The NN is trained with a comprehensive data set collected from the literature, using Levenberg-Marquardt training algorithm in combination with Bayesian regularisation. A very good performance of the trained Neural Network is achieved. Good correspondence between ANN prediction and experimental S-N diagrams is observed. Using the model S-N diagrams for different conditions are predicted and analysed. Graphical User Interface is developed for use of the model. We believe that the model will be important for practical applications in solving various problems on fatigue behaviour of the titanium alloy.

4:15 PM
Prediction of Lateral and Normal Force-Displacement Curves for Flip-Chip Solder Joints: *Daniel Wheeler*[1]; Daniel Josell[1]; James A. Warren[1]; William E. Wallace[1]; [1]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

We present the results of experiments and modeling of flip-chip geometry solder joint shapes under shear loading. Modeling, using Surface Evolver, included development of techniques that use an applied vector force (normal and shear loading) as input to determine a vector displacement of the pads connected by the solder joint (standoff height and misalignment). Previous solutions solved the converse problem: fixed displacements used to determine required applied force. Such solutions were inconvenient for applications, where the applied force (chip weight) is known. Also, for geometric and materials studies of solder joint shapes involving multiple parameters, determining the equilibrium displacement from applied force by bracketing solutions could become computationally expensive. Measurements of solder joint standoff height and misalignment as functions of the applied force (normal and shear), solder volume and pad diameter are presented. Experiments were carried out for solder ball diameters from 15 mil (0.029 mm^3 volume) to 6 mil (0.0019 mm^3 volume) on pads of diameter 0.64 mm and 0.35 mm. Fitting of simulation to experimental results gave optimised values for the contact angle and surface tension of the solder which were consistent with measured and literature values.

4:40 PM
Simulation of the Plastic Behavior during Mechanical Testing of Galvanized Steel using the Finite Element Method: *Adriana Salas*[1]; *Martha Patricia Guerrero Mata*[1]; Rafael Col·s[1]; RenÈ Garza[2]; [1]Universidad Autonoma de Nuevo Leon, FIME-Programa Doctoral en Materiales, Cd. Universitaria, San Nicolas de los Garza, Nuevo Leon 66450 Mexico; [2]Galvak, SA de CV, Quality Assurance, Ave. de la Juventud 340 Nte, Col Cuauhtemoc, San Nicolas de los Garza, Nuevo Leon 66450 Mexico

The use of the finite element method (FEM) for studying the plastic behavior of the metals has become a common practice over the few past years, because it has been proved to be a good means of evaluating processes parameters, and predicting and improving the processes, avoiding the traditional trial and error technique. FEM was used via a commercial package (Ansys) to create a three dimensional model to simulate tension uniaxial simple tests for a galvanized low carbon steel. The aim of the work was to analyze the plastic behavior of a distribution of grains on the zinc cover of a set of galvanized low carbon steel samples. The samples were drawn a mesh with lines 2 mm apart on one of their faces. The samples were deformed at room temperature, at constant low crosshead speed, stopping deformation at different times. Before and during the stops of the testing the deformation measurements were recorded for further calculations in an image analyzer in order to compare these with the computational predictions, as well as validate the numerical model. The results of this study can be extended to other testing conditions, besides the model can used for obtaining constitutive equations.

Computational Phase Transformations: Kinetics of Phase Transformations and Microstructure Evolution

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

Wednesday PM	Room: 201
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Duane Johnson, University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1302 W. Green St., Urbana, IL 61801 USA

2:00 PM
First Principles Computations of Phase Transitions in Oxides and Metals: *Gerbrand Ceder*[1]; Anton Van der Ven[1]; John Reed[1]; [1]MIT, 77 Massachusetts Ave., Rm. 13-5056, Cambridge, MA 02139 USA

First principles energy methods can be used to study the thermodynamics and kinetics of phase transitions in great detail. In ab initio thermodynamics, a systemís Hamiltonian is coarse-grained to a model which only contains the most relevant degrees of freedom. In multi-component systems with configurational disorder this leads to a cluster expansion, or generalized Ising Hamiltonian for the energy. The effective interactions in this Hamiltonian can contain the contribution of non-configurational entropy, due to electronic, vibrational or magnetic excitations, and can be easily equilibrated with Monte Carlo Simulation. The methodology for first principles kinetics in alloys is less well established. Besides a correct thermodynamic description, information on the activation energies is now required. In addition, the Monte Carlo method now has to be treated as a kinetic path evolution, rather than as a sampling method. We will present several successful case studies of studying phase transformations and diffusion in transition metal oxides with first principles methods. Several transition metal oxides display reversible and irreversible phase transitions at room temperature. These phase transitions are usually due to a very high mobility of the transition metal ion and/or to the spinodal-like instability in the thermodynamics. High ion mobility can be achieved in oxides through charge disproportionation reactions which allow the ion to obtain its most favorable hybridization at every point along the migration path. This insight has lead to several propositions for engineering the increased stability of these materials. Under some circumstances, chemically embrittled fracture can also be modelled as a phase transition in the appropriate variable space. We show an example of H-embrittlement in metals.

2:30 PM
Ordering Trends in bcc-Based Alloys: *Patrice E.A. Turchi*[1]; Vaclav Drchal[2]; Josef Kudrnowsky[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct. (L-353), PO Box 808, Livermore, CA 94551 USA; [2]Institute of Physics, Acad. of Scis. of the Czech Republic, Na Slovance 2, Praha 8, CZ 182-21 Czech Republic

A majority of the phase diagrams for alloys made of two bcc-based transition metals indicates in the solid phase the existence of a solid solution in the entire range of alloy composition. Using a first-principles electronic-structure approach departures from ideal solid solution behavior are predicted. The methodology is based on the Generalized Perturbation Method applied to the fully relativistic Tight-Binding Linear Muffin-Tin Orbital description of the electronic structure of the chemically random configuration of the alloy. Finite temperature effects are accounted for with the generalized mean-field Cluster Variation Method. Ordering trends and stability properties are rationalized as functions of simple electronic parameters. Work performed under the auspices of the U. S. Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

3:00 PM
Multiscale Modeling of Precipitate Microstructure Evolution: Venu Vaithyanathan[2]; *Christopher Mark Wolverton*[1]; Long-Qing Chen[2]; [1]Ford Motor Company, MD 3028/SRL, PO Box 2053, Dearborn, MI 48121 USA; [2]Pennsylvania State University, Dept. of Matls. Sci., 106 Steidle Bldg., State College, PA 16801 USA

We demonstrate how three ìstate-of-the-artî techniques may be combined to build a bridge between atomistics and precipitation microstructure: 1) first-principles total energy and linear-response calculations, 2) a mixed-space cluster expansion approach, and 3) diffuse-interface phase-field modeling. The first two methods are used to construct the thermodynamic driving forces for a phase-field microstructural model of θí Al_2Cu precipitates in Al: bulk free energies, interfacial energies, and elastic strain energies. The incorporation of these energetic properties, obtained from atomistics, into a continuum microstructural model represents a significant step towards predictive modeling of precipitate microstructure evolution.

3:30 PM Break

3:45 PM
Microstructural Evolution in Thermal Gradients: *William C. Johnson*[1]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

Microstructural evolution of thin films on compliant substrates subjected to a temperature gradient through the thickness of the film is examined numerically using Cahn-Hilliard and phase field models. Compositional and transformation strains, strains between the substrate and film, and thermal stresses are considered for a binary alloy which can exist in several different phases. The temperature gradients alter the kinetics of spinodal decomposition, the sequence of phase formation, and the resultant steady-state profiles which evolve with time as compared to isothermal transformations. The effects of substrate material parameters and thickness on evolution are explored. This work is joint with S. Wise, Y. Zhen, P.H. Leo, and is supported by the US Department of Energy through GrantDE-FG02-99ER45771.

4:15 PM
Spinodal Decomposition and Layered Growth in Multiphase Films: *Perry H. Leo*[1]; [1]University of Minnesota, Aeros. Eng. & Mech., 107 Akerman Hall, 110 Union St. S.E., Minneapolis, MN 55455 USA

The microstructural evolution of multiphase films is investigated using a Cahn-Hilliard type equation. The problem is formulated for a binary alloy, and allows for multiple phases, varying temperature, and elasticity. Elastic fields arise from compositional self-strain and thermal mismatch. The governing partial differential equation is solved using a dual-grid finite difference technique that guarantees mass conservation. Results show that quenching the system leads to decomposition at pre-existing interfaces, which may increase the number of layered phases in the system. These extra layers may persist for very long times. Results also show that because of elasticity, the phases that appear may not be equilibrium phases in the absence of elasticity. This work is joint with Y. Zhen, W.C. Johnson and S. Wise, and is supported by the US Department of Energy through Grant DE-FG02-99ER45770.

4:45 PM
Impact of Nucleation Conditions on Transformations: *J. M. Rickman*[1]; [1]Lehigh University, Matls. Sci. & Eng., Bethlehem, PA 18015 USA

We examine quantitatively the impact of various nucleation conditions on the temporal evolution of a phase transformation, with particular emphasis on thin-film systems. Specifically, we characterize the transformation kinetics and resulting microstructures associated with catalyzed nucleation on crystalline defects. This is accomplished via the calculation of non-equilibrium correlation functions, as well as related descriptors of spatial point processes, using both computer simulation and complementary analytical methods. In order to connect our models more closely with experiment, we also discuss image processing strategies that have been employed to interpret relevant TEM micrographs.

5:15 PM
A Phase-Field Model for Coherent Microstructure Evolution in a Thin Film Constrained by a Substrate: Yulan Li[1]; S. Y. Hu[1]; Z. K. Liu[1]; *L. Q. Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

A three-dimensional phase-field model has been developed for studying the stability and evolution of coherent microstructure evolution in thin films constrained by a substrate. Elastic solutions are derived for both elastically anisotropic and isotropic thin films with arbitrary domain structures, subject to the mixed surface stress-free and substrate constraint boundary conditions. Electric field in the film is calculated taking into accout the long-range electric dipole-dipole interactions under short-circuit, open-circuit or mixed electric boundary conditions. A specific example of a [001] orientated PbTiO3 film heteroepitaxially grown on a [001] cubic substrate is considered. We investigated the effect of substrate constraint, temperature and depolarization on the volume fractions of ferroelectric domain variants, domain-wall orientations, surface topology, domain shapes, and their temporal evolution for a cubic-to-tetragonal ferroelectric phase transition. It is shown that the shapes of a-domains with tetragonal axes parallel to the film surface are significantly different from those of c-domains with tetragonal axes perpendicular to the film surface. For the substrate constraints and temperatures under which both a1- and a2-domains coexist, both types of a-domains are present with their tetragonal axes perpendicular to each other, and the domain wall orientations deviate from the 45 degree orientation generally assumed in thermodynamic analyses. It is demonstrated that a substrate constraint results in sequential nucleation and growth of different tetragonal domains during a ferroelectric phase transition. The effect of depolarization on the domain shape and domain variant volume fraction will be discussed.

Creep Deformation: Fundamentals and Applications: High Temperature and Advanced Materials

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

Wednesday PM	Room: 214
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: J. D. Whittenberger, NASA-Glenn Research Center, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

2:00 PM Invited
Microstructure and Creep Strength of Fourth Generation Single Crystal Superalloys: *Hiroshi Harada*[1]; [1]National Institute for Materials Science, High Temp. Matls. Rsrch. Grp., 1-2-1 Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan

Fourth generation single crystal(SC) Ni-base superalloys with plutinum group metals additions have been developed along the guidelines given by our alloy design computer programs. In the present paper, the relationship between microstructures and creep properties of the alloys are examined and compared with their mother alloy TMS-75, our third generation SC superalloy. The role of larger negative lattice misfit as well as microstructural stability will be discussed for understanding the excellent creep resistance at higher temperature and lower stress creep conditions, e.g., over 400h rupture life at 1100C/137MPa, with the developed fourth generation SC superalloys.

2:25 PM
Effect of Microstructure on Creep in Directionally Solidified Eutectic NiAl-31Cr-3Mo: *J. Daniel Whittenberger*[1]; S. V. Raj[1]; [1]NASA-Glenn Research Center, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Directional solidification of eutectic NiAl-31Cr-3Mo (at. %) can yield a combination of reasonable room temperature toughness (~20 MPa√m) and good creep strength (~100 MPa at 1300 K and 10^{-7} s^{-1}) which makes this material attractive for use in elevated temperature, oxidizing environment conditions. The 1200-1400 K deformation properties of as-cast NiAl-31Cr-3Mo and alloy rods directionally solidified at growth rates between 7.6 to 508 mm/h have been characterized in compression at strain rates ranging from 10^{-3} to 10^{-9} s^{-1}. In addition extensive light optical microstructural analysis of the directionally solidified NiAl-31Cr-3Mo has been undertaken to determine the average spacing, cell size, intercellular region thickness, etc. This presentation will focus on correlations between microstructure and creep properties and the connection of microstructure with the underlying mechanisms controlling deformation in this eutectic.

2:45 PM
Microstructure Design Near Grain Boundaries for Creep Resistant Tempered-Martensitic 9Cr Steels for 650°C USC Boilers: *F. Abe*[1]; T. Horiuchi[1]; M. Taneike[1]; K. Kimura[1]; S. Muneki[1]; H. Okada[1]; [1]National Institute for Materials Science Japan

National Institute for Materials Science (formerly, National Research Institute for Metals) has been conducting the research and development of advanced ferritic steels for application to thick section boiler components of ultra-supercritical (USC) power plant at 650°C.The project involves the improvement of creep strength, oxidation resistance and creep-fatigue properties for high-Cr ferritic steels, including welded joints. This paper describes the microstructure design near grain boundaries for the improvement of long-term creep strength at 650°C. A tempered-martensitic steel ASME T91 (9Cr-1MoVNb steel) exhibits a rapid decrease in creep strength at long times above 10,000 h at 600 to 650°C. The TEM observations show that preferential recovery of lath martensitic microstructure proceeds in the vicinity of prior-austenite grain boundaries at low stresses and long times above 10,000 h, involving agglomeration of carbonitrides and coarsening of lath near grain boundaries. The preferential recove ry of microstructure causes the transition from the primary or transient creep to the tertiary or acceleration creep at shorter times, which results in higher minimum creep rate and shorter creep life. Suppression of preferential recovery of lath martensitic microstructure near grain boundaries has been examined by optimizing the distributions of precipitates for a tempered-martensitic 9Cr-3WVNb steel and for a carbon-free martensitic Fe-12Ni-9Co-10W-0.2Ti-0.1Al alloy which is a modification of low-carbon maraging steel.Homogeneous dispersion of fine M23C6 carbides at lath boundaries has been achieved for up to long times for the 9Cr-3WVNb steel with small amount of boron. This results in stabilization of lath martensitic microstructure near grain boundaries and hence improves long-term creep strength. The enrichment of boron into M23C6 carbides becomes more significant with reaching prior-austenite grain boundaries. Homogeneous dispersion of fine V(Nb)-nitrides and intermetallic compounds at lath boundaries as well as within lath near grain boundaries has been also obtained for the 9Cr-3WVNb steel with minimized carbon and the Fe-12Ni-9Co-10W-0.2Ti-0.1Al alloy, respectively. Based on the experimental results, the optimization of microstructure near grain boundaries will be described.

3:05 PM
Creep Behavior of Binary NiAl: *S. V. Raj*[1]; [1]NASA Glenn Research Center, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Constant load creep tests were conducted on binary NiAl. Two types of primary creep transients were observed depending on stress and temperature. First, at low temperatures and stresses, there was an initial sharp decrease in the creep rate soon after the application of the applied stress followed by inverse primary creep, where the creep rate approached a steady-state value. Second, at high temperatures and stresses, normal primary creep was observed after the specimen was loaded. These observations suggest that primary creep in binary NiAl is limited by dislocation mobility. Power-law creep with a stress exponent of about 6 and activation energy of about 300 kJ/mol was observed between 1000-1300 K. At stresses below 25 MPa, a new mechanism was observed with a stress exponent of about 2 and an activation energy of about 100 kJ/mol. This mechanism was observed to be grain size dependent.

3:25 PM
Creep Behavior at 1050°C of a New Generation Single Crystal Superalloy: *FrÈdÈric Diologent*[1]; Pierre Caron[1]; Alain Jacques[2]; Pierre Bastie[3]; [1]Onera, DMMP, 29 Av de la Division Leclerc, Ch,tillon 92322 France; [2]LPM, UMR 7556, Ecole des Mines de Nancy, Parc de

Saurupt, Nancy 54042 France; 3LSP, UMR 5588, UniversitÈ Joseph Fourier, Saint-Martin-díHÈres 38402 France

We have compared the creep behavior at 1050∞C of two nickel-based single crystal superalloys: the first generation AM1 alloy and the new generation MC-NG alloy containing rhenium and ruthenium and showing a very high creep strength at elevated temperatures. The creep life of MC-NG was longer than that of AM1, but both alloys did not show similar creep behavior. The creep stages of each alloy were analyzed to understand this discrepancy. Attention was focused on the evolution of the γ/γí lamellar microstructure during the creep deformation. The determination of γ/γí lattice mismatches in creep-deformed MC-NG suggests that the microstructural evolution, and therefore the creep behavior, is related to the variation in the difference of lattice mismatch between the horizontal and vertical γ/γí interfaces. The analysis of the dislocation structures evidenced some differences between the two alloys which could contribute to the creep behavior modifications.

3:45 PM Break

3:55 PM
Creep Behavior and TEM Observation of the First and Third Generation Ni-base Single Crystal Superalloys: *Takao Murakumo*[1]; Toshiharu Kobayashi[1]; Shizuo Nakazawa[1]; Yutaka Koizumi[1]; Hiroshi Harada[1]; [1]National Institute for Materials Science Japan, High Temp. Matls. Rsrch. Grp., 1-2-1 Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan

Ni-based single crystal superalloy has been developed by Re addition. Actually, the creep rupture life of a first generation SC superalloy TMS-26 is shorter than that of TMS-75, a third generation Ni-base SC superalloy at 900°C/392MPa. However, the steady state creep region and rupture life of TMS-26 are indeed longer than those of TMS-75 at 1100°C/137MPa. In this study, we observe microstructures with TEM, and differences in creep mechanism are discussed. At lower temperature and higher stress, it is effective to delay rafting for extending creep rupture life. On the other hand, at higher temperature and lower stress, it is expected that rupture life becomes longer when raft structure is stable. In the case of this study, all of factors benefit TMS-26 for rafting rather than TMS-75, resulting in better creep strength at 1100°C/137MPa and shorter creep life at 900°C/392MPa.

4:15 PM
On the Influence of Shear Loading Direction on the Creep Response of Super Alloy Single Crystals: *Kazim Serin*[1]; Muthuswamy Kamaraj[2]; Matthias Kolbe[3]; Gunther Eggeler[1]; [1]Ruhr-Universitat-Bochum, Inst. for Matls., Universitatsstr. 150, Bochum 44780 Germany; [2]Indian Institute of Technology, Dept. of Metlgcl. Eng., Madras 600036 India; [3]DLR, Inst. for Space Simulation, Koln 51170 Germany

Recently a shear creep test technique for superalloy single crystals was developed which can be used at temperatures up to 1100∞C [1]. This technique allows to load specific macroscopic crystallographic slip systems. So far the test technique was used to study microstructural processes associated with high temperature and low stress creep [e.g. 2,3]. In the present paper we report a complete set of shear creep data for the super alloy single crystal material CMSX4. Constant stress tests are performed on four macroscopic crystallographic slip systems. We discuss the shape of individual creep curves as well as the stress and the temperature dependence of the secondary creep rate. In addition an attempt was made to study the influence of a change of load direction. One specimen was shear loaded on the {111} plane in <01-1> direction until a shear strain of 6.5% was reached. Then the test was interrupted and continued in <0-11> direction. After this change creep rates were observed to be ten times higher than bef ore. This result is discussed on the basis of the underlying dislocation processes which govern high temperature and low stress creep. [1] C.Mayr, G.Eggeler, G.A.Webster, G.Peter, Mat. Sci. Eng., A199 (1995) pp. 121-130; [2] M.Kamaraj, C.Mayr, M.Kolbe, G.Eggeler, Scripta Mater., 38 (1998) pp. 589-594; [3] G.Eggeler, A.Dlouhy, Acta Mater., 45 (1997) pp. 4251-4262.

4:35 PM
Structure and High-Temperature Mechanical Behavior Relationship in Nano-Scaled Multilayered Materials: *Nathan Allan Mara*[1]; Alla Sergueeva[1]; Amiya K. Mukherjee[1]; [1]University of California-Davis, Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Nanometer-scale polycrystalline multilayered films (layer thickness less than 100 nm) have been the subject of many recent experimental and theoretical studies. These fine-scale composite materials typically exhibit high yield strength, often approaching the theoretical strength at room temperature. Most attempts to characterize the mechanical behavior of such thin films have been carried out using nanoindentation and scanning force microscopy or their combination and there is little data regarding their mechanical behavior at elevated temperatures. In the present investigation, the microstructure and mechanical properties of polycrystalline Cu-Nb nanolayered composites prepared by magnetron sputtering and electrodeposited Cu-Ni nanolayered composites were evaluated. Samples were tested in uniaxial tension at temperatures ranging from 25 to 550°C at different strain rates. The high strength of these new materials is attributed to their layered, nanoscale structure and a variety of related strengthening m echanisms. This investigation is supported by NSF, Division of Materials Research, grant NSF-DMR-9903321.

4:55 PM
Characterization of Key Modeling Parameters in the Modified Jogged-Screw Model for Creep of Equiaxed Gamma TiAl: *Karthikeyan Subramanian*[1]; Gopal B. Viswanathan[1]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Transmission Electron Microscopy studies on crept samples of an equiaxed Ti-48Al alloy deformed to strains near the minimum strain rate show a microstructure dominated by unit 1/2[110] type dislocations. These dislocations are pinned by jogs of varying heights. The jogged-screw model is adopted, where the rate controlling step is assumed to be the non-conservative dragging of the jogs along the length of the screw dislocations. The presence of tall jogs and the existence of a stress-dependent upper bound to the height of tall jogs which can be dragged have been incorporated into the model. These modifications lead to excellent agreement with experimental data. Simulation of the movement of these jogged-screw segments have lead to the identification of the key microstructural parameters that control the creep rate. Attempt has been made to characterize these modeling parameters as a function of stress, strain and temperature. The evolution of the creep curve is also qualitatively predicted.

5:15 PM Discussion Session

Deformation and Stresses in Small Volumes: Deformation Mechanisms

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

Wednesday PM	Room: 303
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Scott Mao, University of Pittsburgh, Mechl. Eng., 3700 O'Hara St., Pittsburgh, PA 15261 USA

2:00 PM Invited
TEM Investigation of Nanoindentation Induced Deformation Structures in W/NbN Nanolaminates: *Michael F. Savage*[1]; Donald K. Kramer[1]; Aric Lin[1]; Tim Foecke[1]; Scott A. Barnett[2]; [1]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899 USA; [2]Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA

Understanding the deformation mechanisms in nanolaminated composites is an important step towards developing constitutive laws that describe their mechanical behavior. Several mechanisms have been proposed, however experimental corroboration is difficult due to the small length scales associated with these materials and limited means of mechanical testing. The nanoindentation test is an attractive choice as a mechanical probe due to the ease of sample preparation and available analysis procedures. The measure of a materialís resistance to plastic deformation, the hardness, is often related to yield strength through Taborís relationship, in which hardness is three times the yield stress, or according to Johnsonís spherical cavity model. However, these models assume that the material is a homogeneous isotropic solid. Their applicability to nanolaminated composites is unknown. In this study, the deformation mechanisms in single crystal tungsten and epitaxial W/NbN superlattices with bilayer spacings from 5 nm to 30 nm are investigated by means of a novel indentation technique. Cross-section transmission electron microscopy is then used to study the deformation mechanisms, and compare the size and shape of the plastic zones underneath the contact. Differences within the range of bilayer spacings and between the superlattices and tungsten single crystal will be discussed.

2:30 PM
Quantitative In Situ Nanoindentation of Thin Films: *Andrew M. Minor*[1]; Eric A. Stach[2]; J. W. Morris[1]; [1]University of California-Berkeley and Lawrence Berkeley National Laboratory, Dept. of Matls. Sci. & Matls. Sci. Div., One Cyclotron Rd., MS 66-200, Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., MS 72, Berkeley, CA 94720 USA

The fundamental processes that initiate deformation can be measured and observed directly through a nanoindentation experiment. Typically, these discrete microstructural events are examined ex situ, and ex post facto, in which important details may be missed or lost. Recently, we have developed a method for quantitative, in situ nanoindentation in an electron microscope. The initial application of this new technique will be presented, in which we have studied the onset of deformation during the nanoindentation of aluminum and other metallic thin films. The force-displacement curve developed shows the characteristic ìstaircaseî instability at the onset of plastic deformation. This instability corresponds to the first appearance of dislocations in a previously defect-free grain. These results represent the first real time measurements and observations of the discrete microstructural events that occur during nanoindentation.

2:50 PM
Dislocation Emission and Subsequent Interactions with Sigma = 3 Boundaries in Epitaxial Au Thin Films: *Gene A. Lucadamo*[1]; Douglas L. Medlin[1]; [1]Sandia National Laboratories, Thin Film & Interface Sci., MS 9161, PO Box 969, Livermore, CA 94551-0969 USA

We report on the origin and nature of dislocation arrays formed near facet junctions in (111) Au thin film bicrystals. Using room temperature and in situ hot stage transmission electron microscopy (TEM), we have studied dislocations at the junctions between orthogonal $\Sigma=3$ {111} and $\Sigma=3$ {112} faceted grain boundaries. Dislocation arrays were observed with b=1/6<112> in {111} twin boundaries near {112} facet junctions. Experiments conducted in the TEM at elevated temperature showed that the arrays originated from the emission of secondary grain boundary dislocations (SGBDs) and that the motion of the emitted dislocations was coupled with the migration of the {112} facet junctions. The SGBDs accommodated a rotational misorientation between adjacent crystallographic domains. This interpretation was corroborated by measurements showing that the domains in the vicinity of the facet junctions deviated slightly ($<1°$) from the exact $\Sigma=3$ coincident-site-lattice (CSL) orientation. We propose that the driving forces for this motion are provided primarily by the climb stress between the SGBDs arising from the discontinuous character of the {112} boundary at the intersection with the horizontal twin plane. This work was partially supported by the US Department of Energy, Office of Basic Energy Sciences, under contract number DE-AC04-94-AL85000.

3:10 PM
Deformation of Small Volumes of Material Studied using Strained Layer Superlattice Structures: *Andrew J. Bushby*[1]; David J. Dunstan[2]; Patricia Kidd[3]; Anthony Kelly[4]; [1]Queen Mary, University of London, Dept. of Matls., Mile End Rd., London E1 4NS UK; [2]Queen Mary, University of London, Dept. of Phys., Mile End Rd., London E1 4NS UK; [3]Philips Analytical Research Centre, Cross Oak Ln., Redhill RH1 5HA UK; [4]University of Cambridge, Dept. Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Mechanical studies of semiconductor superlattices have shown that the onset of plastic deformation under an inhomogeneous stress is a process that takes place simultaneously across a finite volume of the order of a micron across. The deformation behaviour of materials under contact loading is of prime technological importance, and a criterion for yielding is important as a design tool. As technology moves increasingly to smaller scales in thin film coatings, nanostructured materials and micro-electro-mechanical systems, existing yield criteria fail rather badly. The ability to incorporate known internal stresses, and to vary the stress and thickness of individual layers in a semiconductor superlattice, is a very powerful tool, opening up new possibilities for investigations that cannot be achieved by varying external stresses on a specimen that is sensibly homogeneous. In this way, from the initial yield stress of single-crystal strained-layer superlattices under indentation, we demonstrate a new criterion, of which the key feature is that it is to be averaged over a finite volume. By careful design of the superlattice stress in individual layers, the size and position of the initial yield volume can be determined and the yield criteria deduced.

3:30 PM Break

4:00 PM
Heteroepitaxial Aluminum Thin Films on Silicon: A Model System for Understanding Thin Film Plasticity: *Eric A. Stach*[1]; Karla Balzuweit[1]; Ulrich Dahmen[1]; David Nowak[2]; Shefford P. Baker[2]; [1]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, MS 72-150, Berkeley, CA 94720 USA; [2]Cornell University, Dept. of Matl. Scis. & Eng., 214 Bard Hall, Ithaca, NY 14853 USA

Through careful choice of substrate orientation and deposition temperatures, it is possible to grow aluminum heteroepitaxially on silicon. In particular, single crystal, bi-crystal and tri-crystal geometries have been demonstrated. Because of the orientation relationships established, it becomes possible to determine the resolved shear stresses on particular slip systems during thermal cycling. In prior work, we have used in-situ transmission electron microscopy to determine how dislocations mediate plastic deformation during cooling from elevated temperatures in bicrystalline films. Dislocations are introduced into the film by nucleation at grain boundary cusps. These events occur in ensembles at particular temperatures, where resolved shear stresses are on individual dislocations are on the order of 300 to 350 MPa. Herein, we correlate these observations with x-ray microdiffraction studies of strain relaxation and extend these studies to single and tri-crystal microstructures. Direct comparison of these observations with polycrystalline films will be presented, allowing insight into the general behavior of dislocations in thin metal films.

4:20 PM
N-Edge Dislocations in Thin Bent Foils and N-Screw Dislocations in Circular: *Johannes Weertman*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Anomalous hardening was observed by St^lken and Evans in thin bent nickle foils and by Fleck, Muller, Ashby and Hutchinson in twisted copper rods of small diameter. The anomalous hardening has been explained as a caused by the presence of N_dislocations. (Here ëNí stands for geometrically necessary or non-redundant dislocations and ëRí stands for redundant or statistically stored dislocations.) St^lken and Evans attribute the anomalous hardening in bent foils to N-edge dislocations. They assume that the flow stress is proportional to the square root of the sum of the R-dislocation density and the N-dislocation density. Anomalous hardening occurs when the latter density is greater than the former. Fleck and Hutchinson have the same explanation and ascribe the anomalous hardening in torsion rods to N-screw dislocations that are oriented parallel to the axis of the torsion specimen. Hurtado and Weertman analyzed anomalous hardening with N-screw dislocations that lie in the planes perpendicular to the torsion axis. However, they disagree that the anomalous hardening is simply the result of swamping of the total dislocation density by N-dislocations (because insufficient R-dislocation exist to pin N-dislocations). We will show in this talk it is not possible for the anomalous hardening in torsion to arise from twist boundaries formed by combining N-screw dislocations that are parallel to and N-screw dislocations that are perpendicular to the specimen axis because the sign of the N-screw dislocations in one set of dislocations is opposite that of the other. If they could have formed the case for hardening by N-dislocation swamping would be strengthened. The N-screw dislocations all move in the same radial directions (towards the center of the torsion bar) and are not likely to hinder each others motion significantly. The central theme of this talk is that N-dislocation density swamping is not needed for anomalous hardening. The imposition of the condition that the N-dislocation density is less than or equal to the R-dislocation density everywhere in a sample leads to anomalous hardening (by reducing the N-dislocation density and the amount of plastic deformation produced at a given stress level). That this N-dislocation skimping condition does so is shown explicitly in detail for the problem of the thin bent foil.

4:40 PM
Stress Effects in SiO2 Thin Films: *Terry J. Delph*[1]; Ralph J. Jaccodine[1]; Ming-Tzer Lin[1]; [1]Lehigh University, Mechl. Eng. & Mech., 19 Memorial Dr. W., Bethlehem, PA 18015 USA

Stress effects on the oxidation of silicon become of considerable importance in the design of sub-micron structures, where many instances of anomalous oxide thinning have been observed. We describe the state of the art in modeling these stresses. We further report upon recent work upon the oxidation of planar silicon substrates. Oxidation of planar substrates can result in substantial levels of compressive stress. Experimental results from oxidizing substrates tested in bending implicate the substrate strain as one of the factors influencing the thinning behavior.

Fatigue and Creep of Metal Matrix Composites: Creep 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

Wednesday PM	Room: 206
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Efrain CarreÒo-Morelli, University of Applied Sciences of Western Switzerland, Competence Grp. Matls. & Design, Rte. de Rawyl 47., Sion CH-1950 Switzerland; David C. Dunand, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

2:00 PM Keynote
Creep Behavior of Metal Matrix Composites: A Comparison with Pure Metals and Alloys: *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matl. Sci., Los Angeles, CA 90089-1453 USA
There are important differences between the creep behavior of metal matrix composites and unreinforced pure metals and metallic alloys. This paper examines these differences and develops procedures for data analysis.

2:40 PM Invited
Creep Cavitation in Aluminum Containing High Volume Fractions of Alumina Dispersoids: Bing Q. Han[1]; *David C. Dunand*[2]; [1]University of California, Dept. of Mechl. & Aeros. Eng., Irvine, CA 92717-3975 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA
Density measurements were used to quantify creep cavitation in pure aluminum containing very high volume fractions (25-42 vol.%) of alumina dispersoids with a mean diameter of 280 nm. In the range of creep temperatures studied (370-450∞C), the cavity fraction increases linearly with strain, i.e. the ratio of cavity fraction to strain is constant. This cavitation ratio (i) decreases with increasing stress at constant temperature; (ii) is weakly dependent on temperature at constant stress; (iii) increases with increasing volume fraction of dispersoids at constant stress and temperature; (iv) is independent of matrix grain size. The latter result indicates that cavitation occurs at dispersoid/matrix interfaces, not at matrix grain boundaries. Results are compared to existing creep cavitation models in pure metals and metal matrix composites.

3:10 PM
Creep Deformation and Rupture Behavior of Laminated Metal Matrix Composites: *S. B. Biner*[1]; [1]Ames Laboratory, Iowa State Univ., 208 Metals Dvlp., Ames, IA 50011 USA
In this study, the creep behavior at 250 Co of laminated composite consists of 6061-Al alloy layers and 2014-Al-20vol%SiC particulate reinforced composite layers was investigated. In spite of the absence of delamination between the layers, the observed creep rupture times of the laminated composite were much shorter than those seen for its constituent phases. This behavior is explained with a model based on the laminate theory. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-Eng-82.

3:30 PM Break

3:50 PM Invited
Creep Behavior of Composite Lead-Free Electronic Solder Joints: *K. N. Subramanian*[1]; [1]Michigan State University, Dept. Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA
Electronic solders used in surface mount technology need to be reasonably strong to maintain the structural integrity, and should also be reasonably compliant to avoid component failures by the stresses that develop due to CTE mismatches during thermal excursions experienced in service. Service life of such solder joints depend not only on the steady state creep-rate but also on the strain for onset of tertiary creep. Roles of compatible intermetallic reinforcements introduced into lead-free Sn-Ag solders by either in-situ or mechanical means on both secondary creep-rate and strain for onset of tertiary creep will be discussed. Composite approach to increase the solder joint service life will be evaluated on the basis of time duration spent in steady state creep condition as a function of stress and temperature. Acknowledgment: Project funded by Composite Materials and Structures Center at Michigan State University.

4:20 PM
Advances in the Use of Neutron Diffraction to Study Strain Partitioning in Composites: *Mark A. Bourke*[1]; David C. Dunand[2]; Ersan ‹st¸ndag[3]; [1]Los Alamos National Laboratory, MST, MS H805, Los Alamos, NM 87545 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., 1083†MLSB, 2225†N.†Campus†Dr., Evanston, IL 60208-3108 USA; [3]California Institute of Technology, Dept. of Matls. Sci., 309 Keck Laboratory, MC 138-78, 1200 E. California Blvd., Pasadena, CA 91125 USA
In the summer of 2001, a load frame-furnace suite will be implemented at Los Alamos National Laboratory on a new neutron scattering spectrometer called the Spectrometer for Materials Research at Temperature and Stress (SMARTS). The value of neutron diffraction for studying mechanical strain response in multi-phase polycrystalline systems is that it can resolve phase-specific strain, averaged over a volume representative of the bulk response. Thus by recording diffracted spectra during loading of e.g. a metal/ceramic composite, the elastic strain in the metal matrix can be distinguished from the strain in the ceramic reinforcement. At elevated temperature, creep in the metal alters the composite load partitioning which is reflected in changes to the lattice strain. The new system is noteworthy by virtue of its uniaxial 250KN/1500°C load-temperature capacity under vacuum or controlled atmosphere. This talk will illustrate the capabilities by example. Some systems that are planned for investigation in 2001 include Al-Al_2O_3, Ti-SiC, SiC-Si_3N_4, WC-Co and Waspaloy.

4:40 PM
High Strain Rate Superplasticity of TiN and TiB2 Particulates: *Tsunemichi Imai*[1]; Liang Zhen[2]; Makoto Takagi[3]; [1]National Institute of Advanced Industrial Science and Technology Japan; [2]Harbin Institute of Technology China; [3]Aichi Institute of Technology Japan
TiN and TiB2 particulate reinforced aluminum composites fabricated by powder metallurgical method were extrued and hot-rolled to build fine microstructures and effect of aluminum alloy matrix on the high strain rate superplasticity (HSRS) was investigated. TiN/2014 Al composite exhibits the m value of 0.30~0.43 in the temperature of 773~818K and in the strain rate range higher than 10-1s-1 and maximum total elongation of 370% at the strain rate of 3x10-1s-1 and at 818K, although TiN/2017 Al indicates about 200% at the strain rate of 3x10-2 ~ 3x10-1s-1 and at 818K. And TiN/1N90 pure Al could produce about 200% elongation at the strain rate of 2x10-1s-1 and at 923K. But TiN/Al-1Mg could not produce large elongation. On the other hand, maximum elongation of TiB2/Al composites was about 100%, but the flow stresses of TiB2/2017, TiB2/1N90 and TiB2/Al-Mg composites are the same value as those of TiN/2017, TiN/1N90 and TiN/Al-Mg, respectively although the flow stress of TiB2/2014 is larger than those of TiN/2014. It is thought that HSRS of TiN/2014 aluminum composite is effected by small amount of alloy elements to build optimum partial melting phases at the interfaces and grain boundaries.

Fundamentals of Advanced Materials For Energy Conversion: Hydrogen & Tritium Storage II - Complex Hydrides

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

Wednesday PM	Room: 613
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Karl Gross, Sandia National Laboratories, Analytl. Matls. Sci. Dept., PO Box 969, MS9403, Livermore, CA 94551-0969 USA; Renato G. Bautista, University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA

2:00 PM Invited
Complex Hydrides for Light-Weight Hydrogen Storage: *Karl J. Gross*[1]; E. H. Majzoub[1]; G. Sandrock[1]; [1]Sandia National Laboratories, Analytl. Matls. Sci., PO Box 969, MS9403, Livermore, CA 94551-0969 USA

An on-board supply of high-purity hydrogen is requisite for the development of fuel cell powered vehicles. The discovery that hydrogen can be reversibly absorbed and desorbed from catalyst-enhanced complex hydrides (the alanates) has created an entirely new prospect for lightweight hydrogen storage[1]. Unlike the common interstitial metal hydrides, these compounds release hydrogen through a series of decomposition reactions. The breakthrough for storage applications is that chemical doping with Ti, Zr and other transition metals allows these reactions to take place reversibly under moderate conditions. First investigations have focused on NaAlH4 which has a theoretical reversible hydrogen capacity of 5.5 wt.%. Phase transitions and crystal structure modifications were observed using X-ray powder diffraction during in situ thermal decomposition of this compound. Doping procedures have been dramatically improved through a dry mechanical milling process using TiCl3 as a precursor. Direct synthesis combined with dry doping has completely eliminated hydrocarbon contaminants found in previous wet doping techniques. This is critical for PEM fuel cell applications. Reversible capacities of nearly 5 wt.% at 125∞C are now being achieved at rates required for many hydrogen storage applications. [1] Bogdanovic and Schwickardi, J. Alloys and Compounds Vol. 253,1 (1997).

2:30 PM Invited
Mechanochemically Induced Solid-State Transformations of Complex Aluminohydrides: *Vitalij K. Pecharsky*[1]; Viktor P. Balema[2]; [1]Ames Laboratory, Dept. of Matls. Sci. & Eng., Iowa State University, Ames, IA 50011-3020 USA; [2]Ames Laboratory, Iowa State University, Ames, IA 50011-3020 USA

Recent interest in complex aluminohydrides as potential near ambient, ultra-high capacity, solid-state sources of hydrogen (e.g., for fuel cells) calls for an improved understanding of the processes occurring in these hydrogen-containing materials during both the release and the uptake of hydrogen. Lithium aluminohydride ($LiAlH_4$) rapidly releases 5.2 wt.% of hydrogen (i.e., nearly four times more than $LaNi_5$-based alloys) during mechanochemical processing at ambient conditions in the presence of transition metal catalysts. Using a variety of solid-state characterization techniques, the mechanochemical transformations in a series of $LiAlH_4$-metal catalyst systems have been examined and preliminary insights on their mechanism have been obtained. Surprisingly, the well-known hydriding-dehydriding catalysts, such as Ni and Pt, are practically inactive in the mechanochemical dehydriding of $LiAlH_4$. The latest developments within this extended family of materials will be discussed.

3:00 PM
Hydrogen Absorbing Properties and Crystal Structure of Ti-V-Mn BCC Solid Solution Alloys: *Yumiko Nakamura*[1]; Etsuo Akiba[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST), Energy Elect. Inst., AIST Tsukuba Central-5, 1-1-1, Higashi, Tsukuba, Ibaraki 305-8565 Japan

Ti-V based ternary BCC solid solution alloys are eminently suitable for use in on-board hydrogen storage. We have been focusing particularly on Ti-V-Mn BCC solid solutions since they show a phase transition distinguished from other BCC alloys: they form a mono-hydride and a di-hydride subsequently. Thus we investigated the crystal structure of the two kinds of hydrides by X-ray and neutron powder diffraction. As a result, it was found that both hydrides have an FCC metal sublattice. However, hydrogen atoms occupy the O-sites in the mono-hydride (NaCl structure), in contrast, they occupy the T-sites in the di-hydride (CaF2 structure). The Ti-V-Mn alloys forming NaCl type mono-hydrides show two plateaus in the P-C isotherms. This makes the hydrogen-solution region narrow and increases the effective hydrogen storage capacity.

3:30 PM Break

3:45 PM Invited
Solid State ^1H, ^{27}Al, and ^{23}Na NMR Studies of Doped and Undoped $NaAlH_4$: Kristin K. Kumashiro[1]; Walter Niemczura[1]; *Craig M. Jensen*[1]; [1]University of Hawaii, Dept. of Chem., Honolulu, HI 96822 USA

The dehydriding of solid $NaAlH_4$ is markedly accelerated and rendered reversible at moderate conditions upon mixing the hydride with a few mole percent of selected transition metal complexes. Since the initial report of this effect by Bogdanovic in 1997, the hydrogen cycling performance of the doped hydride has been the subject of intensive investigation and practical improvements. It now appears that doped $NaAlH_4$ could possibly be developed as a viable means for the onboard storage of hydrogen. However, the nature of the dopants has remained an enigma. Also a fundamental understanding of why the kinetic enhancement results upon doping is lacking. In order to elucidate the changes that occur upon doping, we have conducted solid-state NMR studies employing range of single- and double-resonance techniques. We have observed unexpected spectral features that will be discussed in the context of structural models.

4:15 PM Invited
High-Pressure Hydrides of Iron and its Alloys: *V. E. Antonov*[1]; M. Baier[2]; V. K. Fedotov[1]; B. Dorner[3]; G. Grosse[2]; A. I. Kolesnikov[1]; E. G. Ponyatovsky[1]; G. Schneider[2]; F. E. Wagner[2]; [1]Institute of Solid State Physics RAS, 142432 Chernogolovka, Moscow Dist. Russia; [2]Technische Universitaet Muenchen, Physik-Dept. E 15, D-85747 Garching Germany; [3]Institut Laue-Langevin, BP 156, 38042 Grenoble, Cedex 9 France

Hydrides of iron and iron-based alloys are thermodynamically stable only at high hydrogen pressures exceeding a few GPa and rapidly loose hydrogen under ambient conditions. Nevertheless, these hydrides can be retained in a metastable state at atmospheric pressure and low temperatures, if previously cooled at the high pressure to liquid nitrogen temperature. The review will discuss the current state of studies on phase transformations in the Fe-H and related systems and also on the composition, crystal structure and physical properties of the hydrides, both under high hydrogen pressures and in the ìquenchedî metastable state at ambient pressure. The studies at ambient pressure include magnetization measurements, X-ray and neutron diffraction, Mˆssbauer and neutron spectroscopy.

4:45 PM Invited
The Measurement and Analysis of the Kinetics of Hydride Formation in Bulk Metallic Samples: *Joseph Bloch*[1]; [1]Nuclear Research Center-Negev, Phys., PO Box 9001, Beer Sheva 84190 Israel

The hydriding of bulk metals and alloys is a gas-solid reaction in which a hydride product layer is initially nucleated adjacent to the gas-solid interface and continues to grow until a complete transformation of the parent (alpha) phase into the hydride phase. The combination of measurements of the overall hydriding kinetics of samples having defined shapes and dimensions, together with examinations of partially hydrided samples, enables the presentation of the reaction rate in terms of an appropriate reaction model. A common and important type of topochemical development of the hydride in bulk metals and alloys involves a hydride layer formed on the metallic surface. The continuity of the layer can affect the reaction rate. The rate of hydrogen absorption in a sample, in the presence of a hydride layer, is usually analyzed using a model combining several sequential steps in which the hydrogen is transferred from the gas phase into the reaction site. Coupling the flux equations across the layer under proper steady state conditions, results in a complex rate equation in which the steady state rate of hydrogen absorption (proportional to the hydride layer velocity) is expressed in terms of the pressure, temperature, the rate constants of the sequential steps and the critical concentrations of hydrogen in the hydride. Generally, the rate constants of the individual microscopic processes are comparable in magnitude, so that the overall rate is not controlled by any of the specific sequential steps. However, there are two limit cases related to the first stage, the adsorption, for which the general rate equation is much simpler, namely, the fast and the slow adsorption approximations. Using this limit cases, equations are derived for the steady state hydriding rate of a given system. These equations provide means to anticipate the pressure dependence of the steady state absorption rate under low pressures, close to Peq and under very high pressures. At the low pressure regime both the fast and slow absorption cases yield linear pressure dependence. For the very high pressure range, it is found that the rate become independent of the applied pressure. The rate is given then by a combination of the individual rate constants of the system and the critical hydrogen concentrations. The model is shown to apply in several real cases, such as ZrCo, LaNi5 and titanium.

5:15 PM
Catalytic Mechanism of Reversible Hydrogen Storage by Titanium-Doped Lithium Alanates: *Jun Chen*[1]; H. T. Takeshita[1]; H. Tanaka[1]; T. Kiyobayashi[1]; N. Takeichi[1]; T. Sakai[1]; [1]National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., Ikeda, Osaka 563-8577 Japan

The alkali metal alanates such as MAlH4 and M3AlH6 (M = Li, Na) have high hydrogen capacity and low material cost, but their hydride formations and decompositions are irreversible, thereby making them impractical for industrial application. The recent work showed that doping alkali metal aluminum hydrides with titanium, zirconium, or iron could form reversible hydrogen storage systems. Surprisingly, effective mechanistic explanation for the catalytic phenomenon is not available. Thus, the aim of this article is to prepare Ti-doped MAlH4 and M3AlH6 (M = Li, Na) with nanocrystallites by means of ball milling and chemical synthesis, and to study their effects on dehydriding and rehydriding properties. The methods of X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetry (TG), and differential scanning calorimetry (DSC), and X-ray photoelectron spectroscopy (XPS) were used. It is found that the reversible hydrogen storage characteristics, depending on how to prepare them, are related to the catalytic activity of the doped Ti state. The result shows that both the homogenous distribution of Ti-catalyzed nanocrystallites and the Ti-catalyst with an electron attacking of Ti0 € Ti4+ (Ti0/Ti3+/Ti4+) defect site take the role to be optimizing the reversible hydrogen storage.

Fundamentals of Structural Intermetallics: Fatigue and Fracture of TiAl

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

Wednesday PM	Room: 615-616
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Kwai S. Chan, Southwest Research Institute, Dept. of Matls. Sci., 6220 Culebra Rd., San Antonio, TX 78284 USA; Chong-Soo Lee, Pohang University of Science and Technology, Matls. Sci. & Eng., San 31, Hyoja-dong, Pohang 790-784 Korea

2:00 PM Invited
An Experimental Study of Fatigue Crack Initiation in Lamellar Gamma Titanium Aluminides: *Andrew H. Rosenberger*[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMN, 2230 Tenth St., WPAFB, OH 45433-7817 USA

The damage tolerant design philosophy encouraged by the US Air Force, as documented Engine Structural Integrity Program, requires that aeroengine components be lifed based an understanding of the fatigue and fatigue crack growth behavior of the material. The structural application of advanced intermetallic materials will require an application of this philosophy. However, the stress-life fatigue curve for gamma titanium aluminides is very flat while the fatigue crack growth rate versus stress intensity factor curve is very steep. These indicate that crack initiation plays a dominant role in the their fatigue failure. Hence, an understanding of the mechanism of crack initiation is paramount in their structural application and the development of new, advanced alloys. This study examined the mechanisms of crack initiation in lamellar gamma TiAl under fatigue loading and found significant differences compared to the cracking phenomena observed in monotonic tension or compression.

2:30 PM
Effects of Surface Crack and Notch Condition on the Fatigue Life of Lamellar TiAl Alloys: *Ze-Wen Huang*[1]; Stephen John Trail[2]; Paul Bowen[1]; [1]The University of Birmingham, IRC in Matls./Sch. of Metall. & Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK; [2]Cosworth Racing, Ltd., The Octagon, St. James Mill Rd., Northampton NN5 5RA UK

Both coarse-grained and fine-grained lamellar TiAl alloys have been studied to determine the high cycle fatigue (HCF) performance for a number of surface and notch conditions. All alloys studied have provided good HCF resistance in plain-sided and polished condition. The ratio of „FL/„0.2 is around 0.7-0.8 for fine-grained alloys and even >1 for a coarse-grained alloy. However, the surface damage caused by electro-discharge machining and the sensitivity to notches are found to be strongly dependent on lamellar colony size. The fine-grained alloys were much more sensitive to surface cracks and notches than the coarse-grained alloy, while notch sensitivity reduces as a result of notch strengthening at the notch root due to localised micro-plastic deformation. The difference in surface and notch sensitivity is explained based on a change in controlling mechanism for crack initiation and propagation, owing to a change in lamellar colony size and therefore a change in yield strength.

2:50 PM
Cyclic Stress-Strain Behaviour of a Cast Gamma-Titanium-Aluminide Alloy: *Anne-Lise Gloanec*[1]; Gilbert HÈnaff[1]; Denis Bertheau[1]; Mustapha Jouiad[1]; [1]ENSMA, LMPM, 1, ave. Clement Ader, Teleport 2-BP 40109, Futuroscope, Chasseneuil 86961 France

Gamma titanium aluminide alloys have attractive high temperature properties which make them excellent candidates for aeronautical applications. The present study was undertaken in order to investigate the low cycle fatigue behaviour of the cast alloy Ti-48Al-2Cr-2Nb (atomic %) with a lamellar structure. Tests were carried out at a constant strain rate of 10-3s-1, at two temperatures (room temperature and 750∞C) with different total-strain-amplitudes (from 0.2% to 0.8%), in air. The strain ratio (Re=emin/emax) values of -1 and 0 were used. The cyclic-stress-strain behaviour is characterised by an important hardening at room temperature and no hardening and no softening at 750∞C. At both temperature a Bauschinger effect was noticed. In order to correlate the cyclic-stress-strain behaviour with fundamental deformation mechanisms, TEM observations have been undertaken. In particular the respective role of twining and dislocations cells is examined with respect to temperature and mechanical loading parameters.

3:10 PM
R-Curves for Toughness and Fatigue Crack Propagation in Gamma-TiAl: The Effect of Microstructure: *Reinhard Pippan*[1]; Andreas Tesch[1]; Manuel Beschliesser[2]; H. Kestler[3]; [1]Austrian Academy of Sciences, Erich Schmid Inst. of Matls. Sci., Jahnstrasse 12 A-8700 Austria; [2]University of Leoben, Inst. f¸r Metallkunde und Werkstoffpr¸fung, Franz-Josef-Strasse 18, Leoben Austria; [3]Plansee AG, Reutte Austria

The increase of both-fracture and fatigue resistance as a function of crack extension-is studied in different Gamma-TiAl microstructures. Ti-46.5 at% Al-4at% (Cr, Nb, Ta, B) sheets-a fine grained near microstructure and coarse grained designed fully lamellar microstructures with different lamellar spacing are investigated. The basic idea of the applied technique is to start the toughness or fatigue crack growth experiments on pre-cracked specimens with a minimum contribution of crack tip shielding (or of extrinsic contributions to the fracture resistance). In order to minimize the effects of pre-cracking, we used specimens with very sharp notches (root radius about 10µm) and very short pre-cracks (lengths between 10-50µm) produced at the smallest possible cyclic compression loading. The influence of the microstructure on the intrinsic toughness and intrinsic fatigue mechanism as well as the increase of extrinsic mechanisms are presented and discussed.

3:30 PM
High Cycle Fatigue Properties of a Pre-Alloyed PM TiAl Alloy: Thomas Virginie[1]; Popoff Fabienne[1]; *Thomas Marc*[1]; [1]ONERA, DMMP, 29 ave. de la Division Leclerc, BP72, Chatillon, Cedex 92322 France

Based on our collaborative studies aimed to develop a competitive processing route for near-net shape TiAl components using Powder Metallurgy (PM), there is still a need to establish whether interstitial contamination may be harmful in specific cyclic conditions of aerospace gas turbine engines. Pre-alloyed powders of composition Ti-47Al-2Cr-2Nb have been produced by inert gas atomization. Higher interstitial contamination has been detected both in the smallest and largest powder particles. HCF tests were conducted at RT in air on a series of powder compacts consolidated by HIP. Low data scatter in the as-HIPed condition reflects no clear incidence of powder size on fatigue properties. Results also indicated that this PM material exhibits a particularly high endurance limit by comparing to other gamma materials tested under similar conditions. Detailed SEM fractography was carried out to identify the fracture initiation mechanisms. The possibility for an initial withdrawal of the most contaminated powder particles before consolidation was finally addressed in the present paper.

3:50 PM Invited
Thermo-Mechanical Fatigue Properties of Ti-46.5Al-5Nb: Hongfu Xiang[1]; Yuyou Cui[1]; *Rui Yang*[1]; Jinlong Wen[2]; Shouxin Li[2]; Rui Xu[3]; [1]Institute of Metal Research, CAS, Titanium Alloy Lab., 72 Wenhua Rd., Shenyang 110016 China; [2]Institute of Metal Research, CAS, State Key Lab. for Fatigue & Fracture of Matls., 72 Wenhua Rd., Shenyang 110016 China; [3]Liaoning Technical University, Dept. of Matls. Sci. & Eng., Fuxin 123000 China

Fatigue properties are of primary concern for gamma titanium aluminide alloys intended for off-ground component applications. For many low-risk, stationary applications the alloy is subject to the joint action of cyclic thermal and applied stresses. In this work the thermomechanical fatigue behavior of cast Ti-46.5Al-5Nb with fully lamellar microstructure was studied using smooth bar samples, under synchronized temperature and load control. Triangular and trapezoidal wave forms were used for 200~600°C and 300~700°C cycling in air, respectively, with the maximum load varying in the range of 0.71~0.97 of the ultimate tensile strength of the alloy. Slightly better fatigue life was found under the trapezoidal test condition than the triangular. This was interpreted as a result of creep deformation blunting the fatigue crack tip. Crack initiation and growth were characterized by examining tested samples and will be discussed in relation to microstructure and cast defects.

4:20 PM
Microscopic Characterisation of Gamma-TiAl Diffusion Bonded Joint Interfaces: *Cesar Justino Buque*[1]; Uwe Lorenz[1]; Stefan Eggert[1]; Fritz Appel[1]; [1]GKSS Research Centre Geesthacht, Institute for Materials Research, D-21502 Geesthacht Germany

The present paper is addressed to the problems which occur at the bond interfaces of diffusion bonded titanium aluminide alloys. The investigations were performed on Ti-45Al-10Nb (TNB alloy), Ti-45Al and Ti-54Al by SEM combined with EBSP analysis and EDX analysis. During the diffusion bonding static and dynamic recrystallisation occurs, which is controlled by the chemical composition and microstructure of the starting material. For instance, bonds of the TNB alloy have fine alpha-2 grains which are surrounded by gamma grains. The gamma grains exhibit preferentially middle orientations in the direction perpendicular to the bond interface. The induced alpha-2 particles are formed by the dissolution of alpha-2 lamellae during the recrystallisation process. The investigations showed, that high titanium content is necessary for alpha-2 grains to form during the bonding process. The recrystallisation process is associated with incompatibilities of elastic deformation. The resulting local stresses are relaxed by mechanical twinning and dislocation glide.

4:40 PM
Atomistic Simulations of Fracture in Lamellar Titanium Aluminides: Ashwin A. Ramasubramaniam[1]; *William A. Curtin*[1]; Diana Farkas[2]; [1]Brown University, Div. of Eng., Providence, RI 02912 USA; [2]Virginia Tech, Dept. Matls. Sci., Blacksburg, VA 24060 USA

Atomistic studies of fracture in lamellar Ti-Al are reported. Cracks in Ti3Al lamellae blunt; few dislocations reach the TiAl. Cracks in TiAl emit dislocations that arrest at the Ti3Al interface and then cleave. Cracks at the TiAl/Ti3Al interface blunt and deflect into the TiAl. A model for blunt crack dislocation emission and cleavage, extended to include blunting and shielding by dislocations, predicts (i) the number of dislocations at cleavage scales linearly with lamellar thickness t and (ii) the toughness scales with the square root of t. The simulations on TiAl agree with these scalings; results for Ti3Al can also be rationalized. Thin layers of TiAl between Ti3Al lamellae are thus the ìweak linksî controlling fracture in fully lamellar Ti-Al. Use of these nanoscale results in microscale models of fully lamellar Ti-Al to predict macroscopic crack growth and toughness are discussed.

5:00 PM
The Effect of Single and Multiple Overloads on the Fatigue Crack Growth of High Strength Titanium Aluminides: *Timothy Paul Halford*[1]; Paul Bowen[1]; [1]The University of Birmingham, Dept. of Metall. & Matls., Edgbaston, Birmingham B15 2TT UK

Samples of fully lamellar titanium aluminides with randomly orientated colonies were exposed to single and multiple overloads. The damage caused by these exposures was monitored by acoustic emission to determine its extent, its microstructural location, and the manner in which it is introduced in single and cyclic loading. The samples were then exposed to cyclic fatigue loading to failure, revealing the effect of overload damage upon fatigue life. The results were compared for alloys covering both fine and coarse-grained microstructures to identify mechanisms of initiation and growth.

5:20 PM
Production of Gamma-TiAl Sheets with Improved Superplastic Properties by Pack Rolling: *Marat R. Shagiev*[1]; Gennady A. Salishchev[1]; F. H. (Sam) Froes[2]; [1]Institute for Metals Superplasticity Problems of Russian Academy of Sciences, 39 Khalturin Str., Ufa 450001 Russia; [2]University of Idaho, Inst. for Matls. & Adv. Proc., Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

The sheets of γ-TiAl based alloys with improved superplastic properties are of a great interest for aerospace applications. Such properties can be achieved through the grain refinement to micron and submicron level. Grain refinement may also lead to considerable decrease in the temperature intervals of brittle-to-ductile transition and, consequently, of pack rolling. Therefore, the smaller the grain size in rolling preform and the lower the rolling temperature, the higher superplastic properties in the sheet. Besides the microstructure improvement in γ sheets, decrease in rolling temperature will decrease their cost because of energy savings and use of the cheap can materials. The temperature intervals of pack rolling of several γ alloys were defined based on tensile properties at strain rates corresponding to rolling ones. Optimization of texture and rolling regimes were performed in order to eliminate anisotropy of superplastic properties in the sheet. Pack rolling temperature of Ti-50Al alloy with homogeneous microcrystalline structure was found to be 950∞C while that of Ti-45Al-2Cr-2Nb-0.45B alloy was 1100∞C. At 1000∞C and strain rate of 10^{-3} s^{-1}, both sheets exhibited elongations of about 200% and steady state flow stresses of 140 MPa.

Fundamentals of Structural Intermetallics: Structural Intermetallics

Poster Session: Wednesday 6:00 PM – 9:00 PM

The Thermal Phase Stabilization Process of Wurtzite Boron Nitride: *Ali Solaiman Alwatban*[1]; [1]Riyadh Technical College, Mechl. Tech., PO Box 53699, Riyadh 11593 Saudi Arabia

Boron and nitrogen are the two elements that form boron nitride compounds of which a number of polymorphs exist. Hexagonal boron nitride (hBN) has the same structure as graphite, with boron and nitrogen atoms arranged alternately. The chemical bonds between boron and nitrogen within the layer are strong and covalent (sp2). On the other hand, the chemical bonds between the atoms in adjacent layers are weak (van der Waals type). One of the most common form of boron nitride which can be derived from the hexagonal form is wurtzite. In this paper the x-ray diffractometry, transmission electron microscopy and ESR spectroscopy have been utilized in order to investigate the phase stabilization process of wurtzite boron nitride, and the effect of the phase transformation of wurtzite boron nitride into a cubic form (cBN) under high.

Microstructural Evolution of PST-Titanium Aluminum during Microstrain Compression at 1023 K: *Abhishek Gupta*[1]; *J^rg M.K. Wiezorek*[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The creep resistance of lamellar TiAl appears to be related to the ìsoftî mode behavior, which is considered to be unaffected by the presence of the lamellar interfaces, yet lamellar refinement improves the creep strength. The present systematic study investigates the microstructural evolution and interface related deformation processes active during elevated temperature loading of lamellar TiAl. The evolution of microstructures in PST TiAl during constant low strain rate loading along ìsoftî and ìhardî orientations at 750°C was studied by scanning and transmission electron microscopy. Deformation modes active under these different loading conditions and changes in morphology and microstructural metrics of the lamellar phases have been determined. Instability mechanisms operative at such straining and temperature conditions have been identified. Implications for the deformation behavior of PST-TiAl are discussed. Financial support from the School of Engineering of the University of Pittsburgh is acknowledged.

Carburisation and Metal Dusting of Fe-Al Alloys: *AndrÈ Schneider*[1]; [1]Max-Planck-Institut f¸r Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, Duesseldorf 40237 Germany

Iron aluminides are known for their resistance to high temperature oxidation and sulphidation. Only little information is available about carburisation and metal dusting of Fe-Al alloys. Carburisation ($a_C < 1$) and metal dusting ($a_C > 1$) experiments with Fe-Al samples were conducted in CO-H_2-H_2O and CH_4-H_2 gas mixtures at 800∞C. The kinetics of the carbon transfer was measured using thermogravimetric analysis. Computer simulations using the software DICTRA have been conducted in order to predict the formation of the carbides M_3C, MC, M_3C_2 and k-carbide (Fe_3AlC_x) during carburisation. In these calculations the carbides were treated as dispersed particles in the Fe-Al-X (X = Ti, V, Nb) matrix.

Anomalous Effects of Alloying with Nb on Yield Strength of MoSi2: *Adel A. Sharif*[1]; Amit Misra[1]; Terence E. Mitchell[1]; [1]University of Michigan-Flint, Eng., 303 E. Kearsley St., MSB, Rm. 213, Flint, MI 48502 USA

The effects of alloying with 1 at% Nb on the yield strength of MoSi2 were investigated from room temperature to 1600°C. Anomalous solid solution softening at low temperatures and rapid solid solution hardening at high temperatures were observed by alloying. The mechanisms resulting in these anomalies were investigated. Ambient temperature softening may be attributed to the effects of Nb on stabilizing the 1/2<111> dislocations with lowering the stacking fault energy. At elevated-temperatures, dislocation substructures were consistent with a viscous-glide controlled behavior in (Mo,Nb)Si2 alloys as compared to the easy cell structure formation in unalloyed MoSi2. At the intermediate (600-1200°C) temperature range, the anomalous increase in the yield strength observed in unalloyed MoSi2 appears to be suppressed by Nb alloying.

Phase Transformations in the Synthesis of Intermetallics and Composites: *B. A. Greenberg*[1]; *E. P. Romanov*[1]; *L. A. Rodionova*[1]; *E. V. Shorohov*[2]; [1]Institute of Metal Physics Ural Division RAS, 18 S. Kovalevskaya, GSP-170, Ekaterinburg 620219 Russia; [2]Russian Federal Nuclear Center-VNIITF, Snezhinsk 456770 Russia

The microstructure of alloys produced by explosion loading of powders of pure metals and nanocrystalline diamond was examined. A quasispherical loading scheme with a conservation capsule was used. The stress on the contact surface was 50 GPa. Alloys Ni3-Al, Ti3-Al and TiAl were synthesized. Some characteristic microstructures were revealed, which could serve as indicators of the processes taking place in the conservation chamber under explosion loading. The chains of transformations were reconstructed for each synthesized alloy. The system under study was captured in a kind of a trap and failed to reach the equilibrium state. The presence of nanocrystalline diamond had a considerable effect on phase transformations during explosion loading. Fragments of the corresponding phase diagrams, which included both intermetallic reactions and formation of carbides, were reconstructed. Diamond was graphitized. The graphitization process was accompanied by appearance of carbon nanostructures, such as onion-like carbon structures, etc. The outlooks for a wider use of the alloys as functional materials thanks to the introduction of carbon nanostructures have been discussed.

Phase Transformations in Hydrogenated Ti-Nb: *Daniela Zander*[1]; David Olson[1]; Dan Eliezer[2]; [1]Colorado School of Mines, Dept. of Metlgcl. & Matls. Eng., Golden, CO 80401-1887 USA; [2]Ben-Gurion University of the Negev, Dept. of Matls. Eng., POB 653, Beer-Sheva 84105 Israel

The fabrication of Ti-based alloys can be strongly enhanced with hydrogen as a temporary alloying element; for example due to lower stresses and/or lower temperatures. Further in refractory metals or alloys hydrogen has a large potential to promote superplasticity. Until now very few results are known about the non-equilibrium conditions of phase transformations in Ti-Nb alloys due to hydrogen charging at high fugacities as well as from the gas phase. The objective point of our present research is to investigate in detail the influence of hydrogen on the phase stability in Ti-Nb (20-45 wt.% Nb) alloys. The influence of hydrogen on Ti-Nb was studied by means of TDA and microhardness test; the microstructure was investigated by x-ray diffraction, SEM and TEM. Hydrogenation of Ti-Nb was found to exhibit a significant effect on the microhardness. Hydrogen influences strongly the slip mechanism in the bcc-phase and assists in lowering the deformation energy. So the hydrogen influence on the mechanism of plastic deformation of Ti-Nb is of considerable interest. The observed microstructural investigations are expected to provide significant conclusions on the microstructural mechanism how hydrogen influences the plasticity of Ti-Nb. Furthermore the influence of deformation on the hydrogen absorption and desorption behavior as well as microstructural transformations of Ti-Nb are studied and discussed in detail.

The Protection and Creep Behavior of Ti Aluminides with (TiAlCr) Coatings: *German Fox-Rabinovich*[1]; George Weatherly[1]; David Wilkinson[1]; Anatoliy Kovalev[2]; Christoph Leyens[3]; Da Xu[1]; [1]McMaster University, Matl. Sci. & Eng., 1280 Main St. W., Hamilton L8S 4L7 Ontario; [2]Surface Phenomena Research Group, Metallophysical Institute, CNIICHERMET, 9/23 2-nd Baumanovskaya St., Moscow 107005 Russia; [3]DLR-German Aerospace Center, Inst. of Matls. Rsrch., Cologne D-51170 Germany

Ternary TiAlCr alloys have recently attracted attention as protective coatings for the gamma Ti-aluminide family of alloys due to their excellent oxidation stability in air at elevated temperatures. The common feature of these alloys is the formation of a stable alumina layer at the surface during oxidation. Two ternary compounds have been studied extensively, i.e. Ti0.25Al0.67Cr0.08 (t-phase) and Ti0.28Al0.44Cr0.28 (Laves-phase) and their behavior compared to the alumina forming binary Ti0.25Al0.75 (b-phase) alloy. We have shown that in order to achieve an oxidation resistance greater than the binary TiAl3 the components in a multi-component alloy must interact synergistically during oxidation. Cr alloying increases the activity of Al in the alloy and, equally important, reduces the Ti activity, thus establishing the thermodynamic driving force for Al2O3 to form. Cr therefore forces other elements to act together and form a protective alumina layer on the surface. Preliminary results will also be presented showing how coatings made from these alloys behave during flexural creep testing.

Reactive Processing of Nickel-Aluminide Intermetallic Compounds: *Hexiang Zhu*[1]; Reza Abbaschian[1]; [1]University of Florida, Dept. of Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

NiAl have been fabricated by reactive sintering compacts of ball-milled powder mixtures containing Ni and Al. The reaction mechanism, as well as phase and microstructural development, was investigated by quenching the compacts from different temperatures during reactive hot compaction, and analyzed by XRD, SEM and EDS. It was found that the reaction process was strongly affected by pressure, heating rates, heat loss from the sample to the environment. The application of 50MPa prior to the reaction resulted in the intermetallic-formation reaction initiating at a temperature (480°C) much lower than that (~550°C) when no pressure was applied. At high heating rate (50°C/min), when the heat loss is small, the formation of NiAl occurs rapidly via combustion reaction. On the other hand, if the heat loss is significant, as in slow heating rate (10°C/min), the reaction process is slow and controlled by solid-state diffusion. The phase formation sequence for the slow solid-state reaction was determined to be: NiAl3, Ni2Al3, NiAl, NiAl (Al-rich) + Ni3Al, NiAl.

Quantitative Analysis on Boundary Sliding and its Accommodation during Superplastic Deformation of Two-Phase Ti3Al-xNb Intermetallics: *Ji Sik Kim*[1]; Young Won Chang[2]; Chong Soo Lee[2]; [1]Sangju National University, Dept. of Adv. Matls. Eng., Gajang-dong 386, Sangju, Kyung-buk 742-711 S. Korea; [2]POSTECH, Dept. of Matl. Sci. & Eng, San 31, Hyoja-dong, Pohang, Kyung-buk 790-784 S. Korea

A study has been made to investigate boundary sliding and its accommodation with respect to the variation of grain size and a/b volume fraction during deformation of two-phase Ti3Al-xNb intermetallics. Step strain rate tests and load relaxation tests have been performed at 950, 970 and 990∞C to obtain the flow stress curves and to analyze the deformation characteristics by the theory of inelastic deformation. The results show that, due to the dislocational accommodation of both a and b phase, the accommodation modes for fine and coarse-grained materials are in good agreement with the mixed mode of the isostress and isostrain-rate models. The sliding resistance analyzed for the different boundaries is lowest in the a/a boundary, and increases on the order of a/a << a/b = b/b, which plays an important role in controlling the superplasticity of the alloys with various a/b phase ratios.

Phase Stability of L12-Based Alloys in Al-Ti-Cr System: *J. K. Lee*[1]; M. W. Oh[1]; M. H. Oh[2]; D. M. Wee[3]; [1]KAIST, Dept. of Matls. Sci. & Eng., Taejon 305-701 Korea; [2]Kumoh National University of Technology, Dept. of Matls. & Eng., 188, Sinpyung-dong, Kumi, Kyungbuk 730-701 Korea; [3]The Center for Advanced Aerospace Materials, POSTECH, Dept. of Matls. Sci. & Eng., Knut, Kumi 730-701 Korea

Compressive tests on Al-21Ti-23Cr (L12+Cr2Al), Al-21Ti-15Cr (L12+Al17Cr9) and Al-30Ti-15Cr (L12+TiAl+TiAlCr) alloys were performed to investigate the effects of alloy composition on the phase stability of L12-based alloys below 1000°C. In Al-30Ti-15Cr alloy with relatively higher Ti content, yield strength increased very significantly after exposure at 800°C than other two alloys. Microstructure observation and phase identification confirmed that Al-30Ti-15Cr alloy with Ti-rich composition exhibited the full decomposition of L12 phase after exposure at 800°C despite the partial decomposition of L12 phase in Al-21Ti-23Cr and Al-21Ti-15Cr alloys with Ti-lean composition under same conditions. This results from the fact that L12 phase field was shifted to the direction of Al-rich corner composition with decreasing temperature in Al-Ti-Cr phase diagram. In three-point bend tests to investigate the correlation between phase stability and fracture toughness, the fracture toughness of Al-30Ti-15Cr alloy was more degraded after exposure at 800°C than other two alloys because Al-30Ti-15Cr alloy exhibited poor phase stability due to higher Ti content. This elucidated that the poor phase stability resulted in the degradation of fracture toughness of L12-based alloys. Therefore, L12-based alloys with lower Ti content would be more appropriate by considering phase stability coupled with fracture toughness.

Composites of Iron Aluminides Containing Al_2O_3 and TiC: *Joachim H. Schneibel*[1]; S. C. Deevi[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Chrysalis Technologies, Inc., Richmond, VA USA

Sintered and injection molded parts based on iron and stainless steel powders are extensively used around the world for a variety of industrial applications such as tool bits, fasteners, gears, and various automotive components. Composites based on iron aluminides offer high hardness, excellent wear resistance, and good oxidation and corrosion resistance. In this paper we discuss the processing techniques, microstructure, and mechanical properties of FeAl-based composites such as FeAl/Al_2O_3 liquid phase-sintered with and without transition metal carbides. While FeAl does not wet Al_2O_3, addition of TiC to FeAl/Al_2O_3 improves the wetting. Because of residual porosity, the room temperature flexure strength of a FeAl-16.5TiC-16.5Al_2O_3 (vol. %) composite produced by liquid phase sintering was only 300 MPa. Further processing by hot forging at 1000∞C removed the residual porosity and increased the strength to 1000 MPa. Interestingly, the Al_2O_3 and TiC particulates did not degrade the fracture toughness significantly and room temperature values of 27 MPa $m^{1/2}$ were observed. Research at the Oak Ridge National Laboratory (ORNL) was sponsored by Chrysalis Technologies Incorporated under the Work for Others Program, IAN 14B480501, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Intermetallic Compounds in Diffusion Couples of Fe with an Al-Si Eutectic Alloy: *Shant Prakash Gupta*[1]; [1]Indian Institute of Technology, Matls. & Metlgcl. Eng., I. I. T. Kanpur, Kanpur, UP 208 016 India

The phase equilibria in the Fe-Al-Si system have been studied in the temperature range 1020 to 1115°C using ternary diffusion couple experiment involving pure Fe and high purity Al-Si eutectic alloy. A number of binary and ternary Intermetallic compounds have been observed in the diffusion couples cooled at different rates from the annealing temperatures. The microstructure and composition of the Intermetallic compounds have been described and compared with the results published earlier. In all, nine ternary Intermetallic compounds have been identified. Except for the tau3 and tau5 phases, compositions of tau1, $\tau 2$, tau4, tau6, tau7, tau9, tau10 phases agree quite well with the work of Muravíeva and coworkers. The tau8-phase composition has not been observed in this investigation which may be due to very high Si in the intermetallic compound. The invariant reactions originally proposed by Takeda et al are postulated to be responsible for the observed microstructures. The tau10 phase has been observed only in diffusion couples rapidly cooled from the annealing temperature and is proposed to be a metastable phase.

Control of Phase Transformation Process and Improvement of Shape Memory Effect in NiAl Martensite Including Ternary Elements: *S. H. Kim*[1]; M. H. Oh[2]; D. M. Wee[3]; [1]KAIST, Dept. of Matls. Sci. & Eng., Taejon 305-701 Korea; [2]Kumoh National University of Technology, Dept. of Matls. & Eng., 188, Sinpyung-dong, Kumi, Kyungbuk 730-701 Korea; [3]The Center for Advanced Aerospace Materials, POSTECH, Dept. of Matls. Sci. & Eng., Knut, Kumi 730-701 Korea

The Ni-rich b-NiAl alloys, which are potential materials for high temperature shape memory alloys, show the thermoelastic martensite transformation, and it can make their shape memory effect. But the transformation to Ni5Al3 phase occurring at 450~550°C during heating of NiAl martensite interrupts the reversible martensite transformation, consequently the shape memory effect in NiAl martensite canít show any more. The phase transformation process in binary Ni-(33~37)Al martensite was investigated by DTA, and we could understand that the condition of reversible martensite transformation was not the b-Ni5Al3 transformation but the M-Ni5Al3 transformation occurring at 250~300°C. Therefore the transformation temperature of M-Ni5Al3 determined the critical temperature of shape memory effect. For verifying the critical temperature, the phase transformation process was investigated for various ternary Ni-33Al-X alloys (X = Cu, Co, Fe, Mn, Cr, Ti, Si, Nb). As a result, Ti, Si and Nb addition was very effective to lower the Ms temperature, and they could help the Ni-33Al-X alloys to have the shape memory effect. Especially Si and Nb addition could raise the transformation temperature of M-Ni5Al3, and it would be helpful for the shape memory effect at higher temperature.

Mechanical Properties of Fully Lamellar TiAl-Mo-V-Si Alloy: *Sadao Nishikiori*[1]; [1]Ishikawajima-Harima Heavy Industries Company, Matl. Tech. Dept. Rsrch. Inst., 3-1-15 Toyosu, Koutou-ku, Tokyo 135-8732 Japan

In general, refractory metals regard as the beta stabilizer. Increasing of these elements leads to produce a fully-lamellar structure with the beta phase surrounding lamellar colonies. This microstructure has not been attributed to the increase in creep strength. Influence of alloying on microstructure and creep behavior in the TiAl-Mo-V-Si alloy was investigated. Then, as-cast and heat treated microstructures were analyzed as well crept samples. As a result, we developed the fully-lamellar alloy which shows a creep rupture life of well over 2160ks at 1073K and 220MPa and the minimum creep rate of 2x10-5 h-1 at 1073K and 200MPa. Moreover, effect of the micro-alloying of carbon on the lamellar colony size and creep resistance in this developed alloy was also discussed. In addition, with an industrial view to applications, this work mentions castabilty on sub-production scale and effect of surface reaction formed during casting on fatigue properties.

Processing of Ruthenium Aluminide Alloys: *Sebastien Rosset*[1]; David R. Johnson[1]; [1]Purdue University, 1289 Materials & Electrical Engineering Bldg., W. Lafayette, IN 47907-1289 USA

Ruthenium aluminide (RuAl) is a promising high temperature structural material due to its high temperature melting point and room temperature toughness from qualitative tests. However, this unusual room temperature toughness remains unexplained due to lack of single crystal deformation work. Promising results have been found on preliminary experimentations using arc-zone melting. Large grains of several millimeters in diameter have been obtained when growing samples of 50.5 and 52 at% Al. Furthermore, microstructure analysis suggests that a ruthenium-ruthenium aluminide liquid film forms at the grain boundaries and promotes a fast grain growth. Additionally, an advantage of this technique is that it does not require melting of the whole sample. Present work is focused on the control of parameters such as composition and microstructure in order to obtain single crystal specimens suitable for compression and bending tests.

Characterization of the Microstructure and Properties of Welded TiAl Sheet Materials: *Shenavia Wilkerson*[1]; Mario Arenas[1]; Viola L. Acoff[1]; [1]The University of Alabama, Metlgcl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Two gamma TiAl sheet materials were investigated to characterize the weld microstructure and mechanical properties. Spot welding was performed on the design fully lamellar materials. Each specimen cracked catastrophically immediately after welding. Preliminary results showed that all cracks were parallel to the direction of rolling. There were also some smaller cracks within the fusion zone. The primary annealed material was used for making butt welds. For the specimens welded transverse to the rolling direction, there were cracks parallel to the welding direction (normal to the rolling direction) and the cracking was much more severe than those for the specimens welded in the longitudinal direction. The fusion zone hardness of the transverse specimens was slightly lower than that for the longitudinal samples. This corresponded to a coarser fusion zone microstructure for the transverse samples. For the specimens investigated, a preliminary weld structure-property relationship will be established as a function of texture.

Tensile Properties of HVOF-Sprayed Iron Aluminide Coatings: *Terry C. Totemeier*[1]; Richard N. Wright[1]; W. David Swank[1]; [1]INEEL, PO Box 1625, MS 2218, Idaho Falls, ID 83415 USA

The tensile properties of Fe3Al coatings produced by High-Velocity Oxy-Fuel (HVOF) thermal spraying have been investigated at room and elevated temperature using 0.75 mm-thick dogbone specimens. Coatings were sprayed onto steel substrates at three particle velocities. Residual stresses in the coatings were characterized by curvature measurements and X-ray diffraction; subgrain sizes and microstrains were measured using X-ray line broadening analysis. Tensile tests were performed at room temperature on coatings in the as-sprayed condition and after annealing at 800C for two hours. As-sprayed coatings were also tested at 700C. The fracture stresses of the coatings at room temperature increased with increasing particle velocity and were markedly higher after the annealing treatment. The coatings showed no ductility at room temperature. The coating strengths were considerably reduced at high temperature, and plastic deformation occurred. The variations in tensile properties with particle velocity and annealing treatment correlate with changes in microstructure and residual stress state.

The Nitridation of Iron Alumindes in a H2/NH3/Ar Gas Mixture: *Wu Kai*[1]; H. Y. Tsai[1]; H. H. Hsieh[1]; J. P. Chu[1]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., 2, Pei-Ning Rd., Keelung 20224 Taiwan

The nitridation behavior of pure Fe and two iron aluminides (Fe-28 and -40 at.%Al) was studied over the temperature range 500-700°C in a H2/NH3/Ar gas mixture. The results indicated that the nitridation kinetics of both pure Fe and iron aluminides followed the parabolic rate law. The nitridation rates of iron aluminides decreased with increasing Al content and temperature. The scales formed on pure Fe consisted of mostly Fe4N and minor Fe3N, while the scales formed on Fe-Al alloys were a heterophasic mixture of mostly Fe4N and minor amounts of Fe3N and AlN. The formation of AlN is responsible for the reduction of the nitridation rates of pure iron. This work was supported by the National Science Council of Republic of China under the Grant No. NSC 89-2216-E-019-008.

Ordered Beta and Omega Phases in Highly Alloyed, High Strength TiAl Alloys: *Ze-Wen Huang*[1]; Wayne Voice[2]; Paul Bowen[1]; [1]The University of Birmingham, IRC in Matls./Sch. of Metall. & Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK; [2]Rolls-Royce plc, Aeros Grp., PO Box 31, Derby DE24 8BJ UK

TiAl alloys with significant additions of refractory metals, such as Nb, Zr and W, have been developed aimed at increasing the liquidus temperature thus improving high temperature strength and creep resistance. It has been found that ordered beta and omega phases form in as-cast highly-alloyed TiAl alloys which have a detrimental effect on mechanical properties. The retained beta and omega phases can be removed by HIPping in some alloy systems but both phases reform when subjected to long-term exposure at 700∞C. On the other hand, alloys containing ordered beta and omega show no further reduction in mechanical properties after long-term thermal exposure. In order to understand the specific behaviour of ordered beta and omega, a detailed study has been conducted for highly alloyed, high strength TiAl alloys. The effects of retention, removal, re-formation, and Oswald coarsening of ordered beta and omega on mechanical properties are discussed.

General Topics in Waste Treatment and Minimization: Resource Reclamation and Contaminant Control

Sponsored by: Extraction & Processing Division, Process Mineralogy Committee, Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Cheryl Dahlin, US Department of Energy, Albany Research Center, Albany, OR 97321-2152 USA; Jasper Kwong, US Department of Energy, Albany Research Center, Albany, OR 97321-2198 USA

Wednesday PM	Room: 604
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Cheryl L. Dahlin, US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97374 USA; Jasper Kwong, US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97374 USA

2:00 PM
Common Elements of Industries that Successfully Recycle/Reuse Spent Refractory Material: *James P. Bennett*[1]; Kyei-Sing Kwong[1]; [1]Albany Research Center-USDOE, 1450 Queen Ave. S.W., Albany, OR 97306 USA

The successful reuse/recycling of refractory materials after removal from industrial service is limited because of contamination, the low value of spent materials, the low cost of virgin raw materials, the lack of driving forces such as regulations or taxes, and low disposal costs. Most spent refractory is disposed in landfills. Concerns over future liability for waste materials, corporate images, and ISO 14000 have helped to encourage recycling at some companies. The recycling practices and applications for spent refractory materials at facilities successfully recycling used refractory materials will be discussed, with emphasis on similarities among them.

2:20 PM
Reclamation of Spent Lost-Wax Casting Molds: *Cheryl L. Dahlin*[1]; David C. Dahlin[1]; [1]US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA

The practicality of reclaiming lost-wax or investment-casting mold materials for reuse or recycling depends upon numerous technological, economic, energy, quality, social, and regulatory considerations, which can involve different issues than are faced by conventional sand-casting operations. Experimental reclamation tests employing mineral-processing techniques to beneficiate spent mold materials into reusable and/or recyclable components were done on materials from three lost-wax casting operations. Bench scale beneficiation tests using conventional minerals-processing technology were unsuccessful in producing reusable investment mold components at acceptable grade and recovery. Intimate association of phases makes liberation difficult and requires expensive leaching to produce reusable slurry materials. However, this investigation suggests that mineral beneficiation can separate spent mold components into potentially marketable concentrates. Concentrates of facecoat materials, structural iron, casting metal splash, and ceramic refractory were successfully produced by the experimental processes. A preliminary conceptual process flowsheet was developed based on the findings of the investigation.

2:40 PM
Treatment of Electronic Scrap: *Derek J. Fray*[1]; Robert W. Gibson[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The quantity of electronic scrap will continue to expand in the foreseeable future and there may be a need to be able to recycle used printed circuit boards, together with the components. At the present time, the two most common methods of processing electronic scrap are landfilling of feeding the material into furnaces and recovering the majority of the metals. In both these methods, the actual value of the components is lost. This paper reports a method for the selective dissolution of the solder that allows the components to be recovered which can then be sorted by value, usefulness or toxicity. The leachant can then be electrolysed to recover the solder, the boards shredded and copper and precious metals recovered. Experimental results will be presented confirming the validity of this approach.

3:00 PM Discussion

3:20 PM Break

3:40 PM
Spent Pot Lining (SPL)Treatment and Fluoride Recycling Process: *Ken Mansfield*[1]; [1]Portland Aluminium, Spent Pot Lining, PB 1, Portland, Victoria 3305 Australia

Spent Pot Lining (SPL) is a hazardous waste product from the smelting of aluminium due to itís fluoride and cyanide content. Disposal of SPL is a major problem for the aluminium industry due to disposal by landfill increasingly being banned. Between 1992-94 Portland Aluminium, Alcoa and Ausmelt conducted trials in Ausmeltís demonstration furnace to treat SPL. From these trials, and work with CSIRO to produce aluminium fluoride from the furnace offgases, Portland Aluminium and Alcoa authorised AUD$24,000,000 in 1995 to construct an SPL treatment facility. Output products from the process are aluminium fluoride, which has been successfully trialed in the aluminium smelting process instead of imported material, and a vitreous granulated slag having leachability qualities acceptable to the EPA for specific end uses. The ìSPL Treatment and Fluoride Recycling Processî at Portland, Australia is successfully treating SPL and converting it into useful products.

4:00 PM Cancelled
Oxidative Ageing of Chromium(III) Bearing Land Disposed Slags and Possible Prevention: Kriveshini Pillay

4:20 PM
Selective Flocculation of Western Phosphate Tailings: *Cathy A. Summers*[1]; David C. Dahlin[1]; Keith Prisbrey[2]; [1]US Department of Energy, Albany Research Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA; [2]University of Idaho, Dept. of Metlgcl. & Mining Eng., Moscow, ID 83844-3024 USA

Western phosphate cyclosizer overflow tailings were processed by selective flocculation to recover an upgraded phosphate product. Feed slurries contained 5 to 9 pct solids by weight and had P_2O_5 grades between 14 and 16 pct; in bench-scale tests, they were treated with dispersant and flocculant(s) at slightly elevated pH to selectively flocculate the silicate minerals. Under the best conditions, dispersed products contained 25 to 26 pct P_2O_5 and recovered from 19 to 37 pct of the total P_2O_5. The pilot-scale demonstration used on-line analysis of P_2O_5 grade to close the feedback control loop for reagent additions and confirmed bench-scale results.

High Performance Metallic Materials for Cost Sensitive Applications: Metal Matrix Composites

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

Wednesday PM	Room: 213
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Lu Li, National University of Singapore 119260 Singapore

2:00 PM
Novel Al-RE High Temperature Materials for Aerospace Applications: *Shihong Gary Song*[1]; [1]United Technologies Research Center, Matls. & Proc., 411 Silver Ln., E. Hartford, CT 06108 USA

Most conventional aluminum alloys suffer from long-term exposure to elevated temperatures and therefore are rarely used above 175∞C due to a sharp decrease in strength with increasing temperature. In practice, titanium and nickel based superalloys are routinely utilized for structural applications in the temperature regime of 175 ~350∞C. They are, however, considered overkill in many cases and inevitably lead to high costs and weight penalties. Aluminum alloys or composites can be used in place of titanium and superalloys, provided that the strength of the former is enhanced in the temperature regime. To this end a strengthening mechanism other than precipitation has to be employed, which should be temperature insensitive. In this light, new and cost-effective aluminum materials utilizing dispersion strengthening are being developed at the United Technologies Research Center, which have demonstrated promising properties for elevated temperature applications.

2:20 PM
In-Situ Synthesis of Al/TiC Master Alloy Grain Refiners by Different Methods: *B. Q. Zhang*[1]; L. Lu[1]; M. O. Lai[1]; H. S. Fang[1]; [1]National University of Singapore, Dept. of Mechl. Eng., 10 Kent Ridge Crescent, 119260 Singapore

Al/TiC master alloy is a potential grain refiner for aluminium and aluminium alloys. However, the difficulty in the synthesis of TiC particles and thus the relatively higher production cost of Al/TiC have led to a great obstacle for its practical application in the aluminium industry. A new method has been developed for the in-situ synthesis of TiC and the preparation of Al/TiC master alloys. In the present paper, Al/TiC master alloys were prepared by the conventional method and the newly developed method respectively. By using chemical analysis, X-ray diffraction phase analysis (XRD) and scanning electronic microscope (SEM), it was found that, in the conventional method, TiC particles synthesized at two temperatures of 850∫C and 1250∞C accompanied by serious oxidation of aluminum melt, which introduces large number of oxide inclusions in the master alloys. While in the new method, the synthesis process was much accelerated and plenty of TiC were synthesized in a shorter period at a low temperature of 850∫C. The synthesis mechanism was discussed in details in this paper.

2:40 PM Cancelled
Fabrication and Characterization of AluminumñFly Ash Composites: *Ruby Zhang*

2:40 PM
Microstructures and Mechanical Properties of Pure Al Matrix Composites Reinforced with Al-Cu-Fe Quasicrystal Particles: *Fei Tang*[1]; Iver E. Anderson[1]; Bulent Biner[1]; [1]Ames Laboratory, Ames, IA 50011 USA

Model composite materials were produced by using a powder metallurgy technique from either commercial purity or high purity Al matrix powders, reinforced with 30 vol.% Al-Cu-Fe quasicrystal particles, where all powders had dia. < 10µm. The tensile and yield strength of this model composite material was improved 111% and 220%, respectively, compared to the pure Al matrix values for each matrix powder. Elastic modulus (ultrasonic measurements) of the both composites is very close to the theoretical upper bound value from the rule of mixtures. The modulus and microstructure results on fractured specimens are consistent with good interface bonding between the quasicrystals and both Al matrix types. Use of high purity Al matrix powder from a gas atomization reaction synthesis (GARS) technique resulted in improved sintering, as shown by Auger interdiffusion layer measurements, and enhanced tensile ductility. A load transfer mechanism appears to be the main strengthening mechanism for these Al/QXL composites because the CTE of Al and Al-Cu-Fe quasicrystal are relatively low. Project funding from DOE-BES under contract number W-7405-Eng-82.

3:00 PM
Synthesis of TixAly-Al2O3-TiC Composites from TiO2, Al and C Powders: *Z. H. Cai*[1]; Deliang Zhang[1]; [1]University of Waikato, Dept. of Matls. & Proc. Eng., PB 3105, Hamilton New Zealand

Bulk TixAly-Al2O3-TiC composite materials have been successfully synthesized by using a combination of high energy mechanical milling, chemical reactions among TiO2 and C and Al and sintering. It has been demonstrated that this process is very effective in producing composites with Al3Ti, TiAl and Ti3Al matrices and different volume fractions of Al2O3 and TiC reinforcement particles. The reinforce particle size and morphology of the reinforcement particles are significantly influenced by the sintering conditions, especially the sintering temperature. It has been found that during sintering the coarsening of TiC particles is significantly slower than that of Al2O3 particles, resulting in a small TiC particle size in the final microstructure of the composites. The small TiC particle size is highly favorable in improving the strength and fracture toughness of the composites. This paper will present and discuss the results of this study which includes phase formations as a result of different chemical reactions, microstructural development of the materials during sintering, and the effect of microstructure on the strength and fracture toughness of the composite materials.

3:20 PM Break

3:40 PM
Amorphous and Nanostructured Ti-Based Alloys: *Adrian Jianu*[1]; Eberhard Burkel[1]; [1]Rostock University, Phys. Dept., Aug.-Bebel-Str. 55, Rostock D-18051 Germany

The formation and stability of amorphous and nanocrystalline phases in multicomponent Ti-based alloys and composites were studied using high-resolution X-ray diffraction experiments, electron microscopy, differential scanning calorimetry and in situ temperature/pressure synchrotron radiation diffraction experiments. The substitution of titanium with silver significantly enhances the formation of amorphous or nanostructured phases during rapid solidification. Addition of small amounts of Ce in Ti53Zr27Ni20 alloy composition leads to an enhancement of the nucleation rate of the icosahedral phase. When amounts of Ce are added, the icosahedral phase becomes disordered tending to amorphisation and to the formation of a nanocrystalline CeNi intermetallic compound. The addition of Sc in Ti-based alloys generated a mixture of two nanograin phases with C14, respectively quasicrystalline icosahedral structure. Alternative lower-cost processes have been investigated like non-equilibrium synthesis (rapid solidification, mechanical alloying and field assisted sintering) in order to establish the technology and know-how for new Ti-based material system processing.

4:00 PM
Reaction Synthesis of Ti/TiC Particulate Reinforced Composite by Powder Metallurgy Processing: *Hyungsik Chung*[1]; Yong-Hyun Kim[1]; Yong-Jin Kim[2]; [1]Ajou University, Molecular Sci. & Eng. Dept., San 5, Wonchun-dong, Paldal-gu, Suwon, Kyonggi Province 442-749 Korea; [2]Korea Institute of Machinery and Materials, Matls. Eng., 66 Sangnam-dong, Changwon, Gyungnam Province 641-010 Korea

TiC particulates were synthesized in a porous titanium compact by reacting a blended elemental Ti-alloy powder compact with a hydrocarbon gas such as methane or propane and sintering in a vacuum. Titanium carbide was formed as a thin layer around each particle surface during the reaction. Various transient carbides and hydride were also formed during the reaction but disappeared completely by a high temperature vacuum sintering, leaving only the titanium carbide globules. The volume percent of the carbides could be controlled precisely by adjusting the reaction temperature and time while the size and morphology of the carbides were mostly affected by the sintering temperature. Hipping or hot forging was employed to obtain fully densified composite after the sintering. The resulting structure shows uniformly distributed carbide particles, well adhered to the matrix. Tensile yield strength over 1000 MPa, elastic modulus over 130 GPa and 2% elongation were obtained with 10 vol.% TiC reinforcement at room temperature.

4:20 PM
Low Density Ti-Mg-B Alloys: *Mohsin Qureshi*[1]; Sunil 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

Low density titanium alloys were synthesized by mechanical alloying with magnesium and boron. Boron was used because of its low density coupled with its ability to readily form boride with titanium. The microstructure evolution due to the milling process was investigated using optical microscopy, X-ray diffraction (XRD) and differential thermal analyzer (DTA). Ti-Mg-B powder mixture was found to undergo amorphization during mechanical alloying and was partially transformed into TiB2 during subsequent hot isostatic pressing. The resultant material was a composite material with the TiB2 phase dispersed in a Ti-Mg matrix.

4:40 PM
Synthesis of a Ti5Si3-Mo Alloy: *Fusheng Sun*[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

A Ti5Si3-2Mo alloy was synthesized from blended elemental powders of Ti, Si and Mo by mechanical alloying. An incubation time exists for the formation of the Ti5Si3 alloys, and addition of Mo decreases the incubation time of theTi5Si3-2Mo alloy. XRD results show that the Mo was solid solutioned in the Ti5Si3 phase, and the c/a ratio of the Ti5Si3 phase decreases with the addition of Mo. After mechanical alloying for 8 hours, a nanometer alloy of Ti5Si3-2Mo was synthesized with a grain size of about 5nm. Phase transformations occurring in the system during mechanical alloying and heating were also studied with the use of DTA and XRD. During mechanical alloying, the grain size of alpha-Ti was refined, and the Si was solid solutioned in alpha-Ti. When an amount of stored energy (in the form of interfaces) was introduced by MA, the reaction of Ti5Si3 phase occurs by a form of self-propagating high-temperature synthesis (SHS).

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

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

Wednesday PM	Room: 617
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Aris Christou, University of Maryland, Matls. Sci. & Eng., Bldg. 090, College Park, MD 20742-2115 USA; K. L. Murty, DMR, National Science Foundation, Metals Rsrch., 4201 Wilson Blvd., Arlington, VA 22230 USA

2:00 PM Invited
Gallium Nitride Epitaxy on (0001) Sapphire: *Subhash Mahajan*[1]; [1]Arizona State University, Cheml. & Matls. Eng., PO Box 876006, 1711 S. Rural Rd., Tempe, AZ 85287-6006 USA

Two-step epitaxy consisting of low temperature GaN nucleation layers (NLs) and high temperature (HT) GaN overgrowths, deposited on (0001) sapphire by metalorganic chemical vapor deposition, has been examined by transmission electron microscopy and atomic force microscopy. Results indicate that NLs consist of faceted crystalline islands that exhibit a spread in rotation about the (0001) axis. This structure undergoes a metamorphosis on annealing and evolves into rounded islands. The lateral growth during HT deposition of GaN occurs by the attachment of atomic species to steps associated with the rounded islands, resulting in faceted, flat top islands. It appears that growth occurs preferentially in certain regions which develop into ìgrowth patches.î These patches then grow vertically and laterally over the underlying substructure of NLs. The coalescence of these patches produces a continuous GaN layer. The origins of threading dislocations (TDs) in GaN layers has also been investigated. Results show that TDs do not form during the coalescence of islands as was assumed previously. Instead, three possible sources of TDs have been identified: (1) defects in NLs, (2) steps on island surfaces, and (3) point defects. Defects in the NLs constitute the major source and develop into TDs by glide and climb. Furthermore, TDs can form during the HT island growth stage because of sources (2) and (3). It has also been suggested that the mosaic structure observed in fully grown GaN layers is due to elastic interactions between TDs. As a result, TDs undergo glide and climb to form subgrain boundaries.

2:25 PM Invited
Epitaxial Issues and Growth Morphologies of InAlAs/InGaAs MQWs and Heterostructures on Non-(100) Index Substrates: *Aris Christou*[1]; [1]University of Maryland, Matls. Sci. & Eng., Bldg. 090, College Park, MD 20742-2115 USA

Substrate orientation: (100), (11)A, (110)B, (111)A, (111)B and (112) significantly affects phase decomposition and ordering. Through TEM, AFM and photoreflectance investigations, the effect of MBE growth on the above surfaces has been determined. Significant features related to phase decomposition due to (110)B, (111)B and (112) surfaces has been identified. Hexagonal defects in samples grown on (111) and (112) InP surfaces have been identified and correlated with optical measurements.

2:50 PM Invited
Nanoscale Phenomena of Synthetic Functional Oxide Heterostructures: Role of Interfaces: *R. Ramesh*[1]; [1]University of Maryland, Matls. Sci. & Eng., College Park, MD 20904 USA

Over the past six years, we have focused considerable effort on understanding the growth and characterization of functional oxide thin films and heterostructures, specifically ferroelectric, dielectric and magnetic perovskites. Using both epitaxial and polycrystalline materials on a variety of substrates as test vehicles, we have been carrying out systematic studies on the effect of composition, point defect chemistry, strain and processing variables on the microstructure and physical properties. A novel aspect of our work is the combined use of focused ion beam milling and scanning force microscopy techniques to understand the influence of film microstructure on the relevant properties at the nanoscale. This presentation will address two problems and approaches to understand and solve them: (i) the materials science of thin conducting barrier layers that enable the integration of the complex oxides on a silicon surface; (ii) domains in epitaxial films and mapping of their dynamics using scanned force microscopy. In this presentation, I will describe some possible areas where fundamental measurements in conjunction with theoretical studies and modeling will enable a better understanding of the complex phenomena involved in these materials, especially the role of structural, chemical and functional interfaces (such as domain walls). The work at Maryland is supported by the NSF-MRSEC.

3:15 PM Invited
The Role of Silicide Interfaces in Silicon Technology: *Francois Max díHeurle*[1]; [1]IBM, Rsrch. Dept., PO 218, Yorktown Heights, NY 10598 USA

Silicon devices require metal contacts to make connections to the outside world. The silicides used for this application must satisfy a number of criteria relating to how their metallurgical and electrical properties modify the interface characteristics. The silicides used for this purpose usually result from metal-silicon reactions which can be significantly affected by the nature of the interface, epitaxial or not. Epitaxy with silicon stabilizes phases that do not exist under equilibrium conditions. In TiSi2, epitaxy with alloyed phases, enhances the formation of the desired C54 phase. Silicide grain boundaries constitute fast diffusion paths for metal or silicon atoms during the reactions themselves, and later on can be fast diffusion paths for dopant atoms. Extremely high density of stacking faults have been observed in some silicides with a structure and structural transformations akin to cobalt.

3:40 PM Invited
The Atomic-Scale Structure of Surfaces and Interfaces in III-V Semiconductor Devices: *Lloyd J. Whitman*[1]; [1]Naval Research Laboratory, Code 6177, Washington, DC 20375-5342 USA

NRL has a long-standing effort to develop novel optoelectronic and high-speed devices based on the ì6.1 ≈î family of semiconductors-InAs, GaSb, and AlSb. Many of these applications are based on heterostructures where the individual components are only several atomic layers thick, presenting a significant challenge to MBE growth technology. One way we are attacking this problem is by characterizing the surfaces and interfaces within the devices on the atomic scale using scanning tunneling microscopy. For example, we grow a number of samples differently, and then compare their material, optical, and electrical properties. Alternatively, we take a bottom-up approach, working to understand and optimize the surfaces and interfaces first and then apply this knowledge to a device. I will review our efforts to reach a fundamental understanding of all the competing physical and chemical processes during growth, and thereby develop knowledge-based strategies for optimizing the device fabrication.

4:05 PM Invited
Silicon-Germanium-Carbon Self Assembled Quantum Dot Growth and Applications in Electronic Memory Devices: *Sanjay Banerjee*[1]; [1]University of Texas, Austin, TX 78712 USA

We will discuss the growth of strain-engineered Si-Ge-C self-assembled quantum dots (SAQDs) via ultra-high vacuum chemical vapor deposition (UHVCVD) under varying conditions on various substrates including Si, SiO2 gate oxides, oxynitrides, and CVD high-K materials such as ZrO2, HfO2 and their silicates. There are interesting surface chemistry and physics issues in quantum dot growth under such circumstances involving the interplay between reactant precursor partial pressures, growth temperature, surface free energies, and strain energy in Si-Ge-C quantum dots. We will discuss a study of nucleation and grain growth by CVD in the binary/ternary alloys Si-Ge-C on the various types of gate dielectrics mentioned above. We will show how the compressive strain in Si-Ge can be compensated by the tensile strain using C, and how that impacts dot formation. The role of B and P doping will also be discussed. The use of a Scanning Tunneling Microscope to seed such quantum dot growth will also be described. We will also discuss our work on quantum dot flash memories using these Si and Ge nano-crystals as floating gates.

4:30 PM Invited
Diamond Surfaces and Interfaces: *James E. Butler*[1]; [1]Naval Research Laboratory, Chem. Div., Code 6174, Washington, DC 20375 USA

Synthetic diamond materials grown by chemical vapor deposition (CVD) impact a wide range of advanced technologies, ranging from industrial laser windows to high voltage electronics and power distribution. The chemistry of diamond surfaces is integral to the growth by CVD, surface electrical properties, and bonding to other materials. I will provide a summary of our current understanding of diamond surfaces and interfaces.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Fundamentals, Phases, Wetting and Surface Tension

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

Wednesday PM	Room: 612
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Martin Weiser, Honeywell Electronics Materials, Spokane, WA 99216 USA; Michael Pfeifer, Motorola, 4000 Commercial Ave., North Brook, IL 60062 USA

2:00 PM Invited
A Study on Applying the Sn-3.9Ag-0.6Cu Solder to Electronic Packages with Ni Metallization: *C. E. Ho*[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 320 Taiwan

The 95.5Sn3.9Ag0.6Cu (wt.%, Sn3.9Ag0.6Cu) alloy is considered to be one of the most promising replacement for the Sn37Pb solder. The soldering pads in many electronic packages, such as BGA substrates or C4 devices, use Ni layer as a diffusion barrier. However, very few studies on the reaction between Sn3.9Ag0.6Cu solder and Ni substrate had been reported. The objective of this paper is to study the interactions between Sn3.9Ag0.6Cu solder and Ni during reflow soldering. In this study, the reaction kinetics of Sn3.9Ag0.6Cu/Ni at 225-275C was investigated. The effect of soldering temperature on the reactions was also studied in details.

2:25 PM
Surface Tension Measurements of the Eutectic Alloy (Ag-Sn 96.2 at.%) with Cu Additions: *Zbigniew Stanislaw Moser*[1]; Wladyslaw Gasior[1]; Janusz Pstrus[1]; Stanislaw Ksiezarek[2]; [1]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., Reymonta St. 25, Krakow 30-059 Poland; [2]Institute of Non-Ferrous Metals, Sowinskiego St. 25, Gliwice 44-101 Poland

Surface tension may be obtained from experiments or from modeling by Butlerís method based on the optimized thermodynamic parameters. In our previous studies we have measured surface tensions for the entire range of concentrations and at the extensive temperature range for the binary liquid alloys: Ag-Sn,Ag-Bi,Bi-Sn,In-Sn and for the ternary additions of Zn, In and Bi to the binary eutectic (Ag-Sn 96.2 at.%). In this study we have measured surface tensions of (Ag-Sn 96.2 at.%) with following Cu additions: 0.5, 2, 3.75, and 6.5 at.% at the temperature range 227 to 927C. It was shown that the Cu additions increases surface tensions in the relation to the binary eutectic Ag-Sn. The same trend is observed by modeling of the surface tensions from optimized thermodynamic parameters reported in the literature. The 4th element alloying to near-eutectic Sn-Ag-Cu will be analyzed to decrease the surface tension and the melting temperature.

2:45 PM
Solder Balling of Lead-Free Solder Pastes: *Minna Arra*[1]; Dongkai Shangguan[2]; Eero Ristolainen[3]; Toivo Lepistˆ[4]; [1]Flextronics, Finlaysoninkuja 21 A, Tampere 33500 Finland; [2]Flextronics, 2090

Fortune Dr., San Jose, CA 95131 USA; [3]Tampere University of Technology, Elect., PO Box 692, Tampere 33101 Finland; [4]Tampere University of Technology, Matls. Sci., PO Box 589, Tampere 33101 Finland

Solder balling in reflow process for some of the Sn/Ag/Cu solder pastes was studied in this work. Three different solder pastes, several different reflow profiles and conditions and two stencil thicknesses were used in the investigation. During the process screening phase, the reflow profile was varied. Results show that flux chemistry and reflow atmosphere play the major role in solder balling. With all the pastes, the number of solder balls dropped close to zero when nitrogen atmosphere was introduced. Another finding was the influence of the stencil thickness on solder balling. With a thinner stencil, the number of solder balls increased significantly for two of the pastes. In the last phase, the effect of the solder paste particle size on solder balling was studied. One flux chemistry was chosen and the solder particle size was varied between type 3 and type 4. The results show that with type 4 ~40-140 % more solder balls are formed compared to type 3 paste.

3:05 PM

An Experimental Investigation of Solidification Response of Nanoparticle-Reinforced Tin-Lead Solder: *Guo-Xiang Wang*[1]; Dechao Lin[1]; Song Liu[1]; T. S. Srivatsan[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

A series of experiments have demonstrated that nanopowders can be effectively used as a viable means for increase the strength of eutectic tin-lead solder, even for low percentages of the reinforcing nanopowders. This presentation will highlight experimental observations on solidification response of tin-lead solder blended with nanopowders. The solidification kinetics of the solder mixture, tin-lead powder mixed with either copper or titanium-dioxide nanopowders, was conducted on both copper substrate and aluminum substrate under controlled rates of cooling. It was generally observed that addition of nanopowders resulted in a refinement in the eutectic spacing of the solidified end product thus providing ample evidence for its influence in altering the kinetics of both nucleation and growth. The presentation will highlight a nucleation and growth theory that was used to rationalize microstructural development. Strength of the solidified end product was quantified through microhardness tests. Results reveal an increase in hardness of the composite solder, with addition of copper nanopowders having a significant influence than titanium-dioxide nanopowders.

3:25 PM Break

3:40 PM Invited

Phase Equilibria of the Cu-Ni-Sn System: Experiments and Modeling: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL USA

The knowledge of the Cu-Ni-Sn phase equilibria is very important to understand the evolution interfacial microstructure of solder joints that employ Cu/Ni metallization schemes. In such cases the diffusion path or the interfacial microstructure can be strongly time dependent. To address these and other basic issues relevant to soldering, we have performed selected experiments to determine the phase equilibria of the Cu-Ni-Sn system that complement the existing results. To minimize the number of experiments, a comprehensive thermodynamic modeling of the ternary system is carried out. This allows the calculation of Cu-Ni-Sn phase diagrams at any temperature. Both experimental and calculated results in the temperature range relevant to soldering will be presented.

4:05 PM

Thermodynamics of the In-Sn-Zn System: *Jean-Pierre Bros*[1]; [1]Universite de Provence, CNRS UMR 6595, IUSTI, Technopoloe Chateau-Gombert, 5 rue Enrico Fermi, Marseille 13453 France

Lead+tin solder alloys are widely used in electronics industry because of their physical properties (low melting point, good corrosion resistance, correct electrical conductivity,. . .). But, because of the environmental issues related to lead, its use is restricted in many applications. Accordingly, much research is developing on new lead-free solders and tin-based multicomponent alloys including silver, zinc, indium, bismuth, antimony and/or copper appear to be the most promising substitutes. The melting point or the melting range of these new materials will be one of the most important requirements. The present paper reports such a thermodynamic approach: The enthalpy of formation and the phase diagram of the In-Sn-Zn ternary liquid system were measured over an extended composition range. This ternary enthalpy of formation was compared to that estimated from the [In-Zn], [Sn-Zn] and [In-Sn] binary liquid systems. The location of the liquidus surface and of the ternary eutectic plan have been precise. This new information, in good agreement with some results already published, allows the In-Sn-Zn phase diagram to be proposed.

4:25 PM

Experimental Determination and Thermodynamic Calculation of the Phase Equilibria and Surface Tension in the Sn-Ag-In System: *Xing Jun Liu*[1]; Yasuo Inohana[1]; Ikuo Ohnuma[1]; *Ryosuke Kainuma*[1]; Kiyohito Ishida[1]; *Zbigniew Moser*[2]; Wladyslaw Gasior[2]; Janusz Pstrus[2]; [1]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Sendai, Miyagi 980-8579 Japan; [2]Polish Academy of Science, Inst. of Metall. & Matls. Sci., Reymota St. 25, Krakow 30-059 Poland

The phase equilibria and liquidus surface in the Sn-Ag-In system are important for the design of the Pb-free solder, however, the phase equilibria in this system have not been well established. In the present paper the phase equilibria of the Sn-Ag-In system were investigated by means of differential scanning calorimetry (DSC) and metallography. The isothermal sections at 180-600C, as well as some vertical sections were determined. Thermodynamic assessment of the Cu-In-Sn system was also carried out based on experimental data of activity and phase equilibria using the CALPHAD method, in which the Gibbs energies of the liquid, fcc and bcc phases are described by the subregular solution model and that of compounds are represented by the sublattice model. The thermodynamic parameters for describing the phase equilibria were optimized, and agreement between the calculated and experimental results was obtained. The maximum bubble pressure method has been used to measure the surface tensions and densities of the In-Sn liquid alloys (25, 51.7 and 75 at. % In) at temperatures 227 to 927C. Similarly, there were investigated ternary alloys, adding 5 and 10 at.%In to the eutectic alloy (Ag-Sn 96.2 at.%) at the same temperature range as for the In-Sn alloys. Due to the similarity of both surface tension and density of pure In and Sn experimental data are scattered. In such cases, modeling of surface tension from optimized thermodynamic parameters by Butlerís model is superior over experimental data, as shown in this contribution.

4:45 PM

Thermodynamic Investigations of Possible New Solder Alloys: *Adolf Mikula*[1]; [1]Institute of Inorganic Chemistry, Waehringerstrasse 42, Vienna A-1090 Austria

Lead-tin solders are commonly used in electronic packaging due to their unique combination of electrical, chemical, physical, thermal and mechanical properties. Since lead and lead containing alloys cause great environmental concern and health hazards it is necessary to replace lead in solder materials. To get the same or better properties it is therefore necessary to investigate some multicomponent systems. Some of these materials are ternary tin or indium alloys. Not much information about thermodynamic properties is known. We measured the thermodynamic properties of several ternary alloys like Au-Sn-Zn, Ag-Sn-Zn, Al-Sn-Zn, Cu-In-Zn and In-Sn-Zn. The free energy and the heat of formation were determined with an emf method and with calorimetric measurements over the whole composition range. These thermodynamic data were used to improve the phase diagram calculations and will be helpful for some theoretical models to improve the calculation of physical or mechanical properties.

5:05 PM

Strong Effect of Cu Concentration on the Reactions between SnCu Lead-Free Solders and Ni: W. Chen[1]; C. E. Ho[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 320 Taiwan

The eutectic Sn-0.7Cu solder is considered a very promising lead-free replacement for the Sn-37Pb solder, especially for wave soldering applications. For industrial uses, a ±0.2% accuracy in composition is generally accepted. In other words, for the Sn-0.7Cu solder, the Cu concentration can range from 0.5 to 0.9 weight percentage. However, our recent study revealed that the Cu concentration had a very strong effect on the reactions between Sn-Cu solders and Ni. One of the main reaction product $(Cu_xNi_{1-x})_6Sn_5$ showed a very rich morphology, depending on the exact Cu concentration. The morphology and distribution of $(Cu_xNi_{1-x})_6Sn_5$ would in turn have a strong influence on the strength of solder joints. This study showed that the Cu concentration must be controlled in a much tighter range than ±0.2% in applications involving Ni-bearing contacts and surface finishes.

Magnesium Technology 2002: Magnesium Alloy Processing and R&D Strategies

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

Wednesday PM	Room: 606
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; Neale Neelameggham, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA

2:00 PM Cancelled
Semi Solid Forming of Magnesium Alloys: An Innovative Route to Produce Advanced Components: Dierk C. Hartmann

2:20 PM
Evaluation of a New Concept for Semi-Solid Magnesium Billet Forming: *Basant L. Tiwari*[1]; Raja K. Mishra[1]; [1]General Motors, R&D Ctr., Matls. & Proc. Lab., MC 480-106-212, Warren, MI 48090-9055 USA

A new concept of using ternary eutectic for semi-solid magnesium billet forming was evaluated. Magnesium alloy ZC63 showed that the presence of only 16% liquid facilitated metal forming at low pressures and temperatures in the semi-solid state. It was also shown that the amount and composition of the liquid rich in the alloying elements did not change significantly with temperature. This feature will alleviate the problems caused by excessive oxidation of magnesium during reheating and handling of the billets. The new concept also provides a basis for designing new alloys exclusively suited for semi-solid billet forming.

2:40 PM
Tensile Coherency in Semi-Solid AZ91 Alloy: *John F. Grandfield*[1]; John Taylor[2]; Cameron Davidson[1]; [1]CRC for Alloy and Solidification Technology (CAST), CSIRO Mfg. Sci. & Tech., Albert & Raglan Sts., Preston, Victoria 3072 Australia; [2]The University of Queensland, Dept. of Mining, Minls. & Matls. Eng., St. Lucia, Queensland 4072 Australia

Hot tearing during casting is related to tensile loads applied to the semi-solid region. Recent attempts to horizontal direct chill cast magnesium alloy AZ91 resulted in typical centre-line hot tears. The tensile strength of AZ91 in the semi-solid region was therefore investigated. Results indicate that the tensile strength of the mush at high fraction solid is due to the strength of the bonded solid grains while at low fraction solid the tensile strength is related to the surface tension of the liquid film between the grains. Tensile coherency is estimated to occur at 85% fraction solid, at which point the solid grains become bonded.

3:00 PM
Advances in Thixomolding Magnesium: *Steve LeBeau*[1]; Jason Fulcher[2]; Oscar Roberto[3]; Robert Altham[4]; Randy Beals[5]; [1]Thixomat, Ann Arbor, MI USA; [2]DaimlerChrysler Corporation Auburn Hills-Michigan, MI USA; [3]Henkel Surface Technologies, Madison Heights, MI USA; [4]Keronite, Ltd. Advanced Surface Technology, Great Abington, Cambridge UK; [5]DaimlerChrysler Corporation, Cast Metals Eng. & Prototyping, 800 Chrysler Dr., Auburn Hills, MI 48326 USA

Thixomolding is a relatively new process in which the metallic slurry is injected into a die cavity at semi-solid temperatures to form near net-shape products from the solid feed stock. As part of on-going research regarding the Thixomolding process, this study concentrated on Thixomolded magnesium alloys AZ91D and AM60B. The test samples where made with high and low volume percent solid for each alloy. Each sample was benchmarked using an extensive test matrix that includes different material characteristics (tensile, hardness, microstructure, metallographic image analysis, x-ray, surface roughness, warp, linear thermal expansion, notched bar impact), joining (welding, riveting, adhesives) and finishing examinations (corrosion, pretreatment, e-coating, anodizing and Keronite, etc.) The database will be used to create a working model using the rule-of-mixture approach for the various properties. Future work will include examination of newly developed magnesium alloys for high temperature applications.

3:20 PM
Blended Magnesium Alloys Produced by the ThixomoldingÆ Process: *T. K. Nandy*[1]; J. W. Jones[1]; T. M. Pollock[1]; D. M. Walukas[2]; R. F. Decker[2]; [1]University of Michigan, Ann Arbor, MI USA; [2]Thixomat, Inc., Ann Arbor, MI USA

Blended alloys consisting of mixtures of varying fractions of AM60B and AZ91D have been produced using the ThixomoldingÆ technique. Alloys investigated included 100%AM60B, 67%AM60B + 33%AZ91D, 33%AM60B + 67%AZ91D and 100%AZ91D. The blended alloys were fabricated by first mechanically mixing chips of the two base alloys and ThixomoldingÆ the blended mixtures. Molding parameters were adjusted in order to investigate the structure and properties of each of the four alloys under conditions of molding with low fractions of solid during the process (4-9%) as well as high fractions of solid (15-22%). At both low and high fractions of solid, the yield strengths and tensile elongations of the blended alloys were consistent with a Rule of Mixtures prediction. Microstructures have been examined in detail by optical and scanning electron microscopy. Additionally, energy and wavelength dispersive spectrometry have been utilized to examine chemical gradients in the mixed microstructures.

3:40 PM Break

4:00 PM
A Comparison of Aging Kinetics between a Die Cast and a Thixocast Magnesium Alloy: *Emanuela Cerri*[1]; Salvatore Barbagallo[1]; Marcello Cabibbo[2]; Enrico Evangelista[2]; [1]INFM-University of Lecce, Dept. Ingegneria dellíInnovazione, via per Arnesano, Lecce 73100 Italy; [2]INFM-University of Ancona, Dept. of Mech., via Brecce Bianche, Ancona 60131 Italy

The aging kinetics of two Mg-Al alloys were investigated by different techniques after various heat treatments. One alloy was produced by die-casting and the other by thixocasting. The material furnished in the as-thixocast state consists of large α-globules separated by quasi-eutectic phase (α+β). The cast structure is formed by a dispersion of Mg-α grains and the eutectic (α+β) where β is the $Mg_{17}Al_{12}$. Thermal analysis was performed on the two AZ91 alloys to define the temperature formation of the phases. Afterwards, the solution kinetics and the aging processes were followed by hardness, electrical conductivity and microscopy. The kinetics of aging are faster at the higher temperature due to the enhanced diffusion of atoms in the matrix, so the hardness peaks at 210-220∞C occur after 3-4 h while at 170∞C, the peaks are not reached in the time range considered. Differences arise in aging kinetics of the two magnesium alloys.

4:20 PM
Rapidly Solidified Powder Metallurgy Mg97Zn1Y2 Alloys with Tensile Yield Strength of 610 MPa and Elongation of 5%: *Yoshihito Kawamura*[1]; Akihisa Inoue[2]; [1]Kumamoto University, Dept. Mechl. Eng. & Matls. Sci., 2-39-1 Kurokami, Kumamoto 860-8555 Japan; [2]Tohoku University, Inst. for Matls. Rsrch., Sendai 980-8577 Japan

The RS powder metallurgy (P/M) magnesium alloys have been investigated for conventional alloys, resulting in no remarkable improvement in strength. Moreover, the RS P/M processing has been applied to amorphous alloys, in which the nanocrystalline P/M magnesium alloys were synthesized by the crystallization of the amorphous phase. The nanocrystalline RS P/M alloys exhibit excellent tensile strength, while the ductility is very poor for practical use. In order to develop high strength RS P/M magnesium alloys with enough ductility for practical use, it is necessary to investigate new alloy compositions suited to the RS processing. We have found that Mg-Zn-Y alloys were the most promising system for production of RS P/M alloys with high strength and good ductility, as consequence of the preliminary investigations of the alloy compositions in Mg-X1-X2 (X=Li, Al, Zn, Ca, Sn and Y) alloys using the melt-spun ribbons. Nanocrystalline magnesium alloys having high tensile strength, high elevated-temperature tensile strength, high-strain-rate superplasticity and high thermal stability have been developed in Mg97Zn1Y2 alloy by rapidly solidified powder metallurgy (RS P/M) processing. The tensile yield strength and elongation that were dependent on the consolidation temperature were in the ranges of 570 to 606 MPa and 5 to 15%, respectively. Youngís modulus of the RS P/M alloy was 45 GPa. The specific tensile yield strength was four times as high as that of a commercial AZ91-T6 alloy, and was higher than those of conventional titanium (Ti-6Al-4V) and aluminum (7075-T6) alloys. The RS P/M alloys exhibited excellent elevated-temperature yield strength that was 500 MPa at 423 K. The RS P/M alloy also exhibited high-strain-rate superplasticity at a wide strain-rate range from 1x10-2 to 1x100 s-1 and at a low temperature of 623K. It is expected that the Mg97Zn1Y2 RS P/M alloy can be applied in some fields that requires simultaneously the high specific strength at ambient and elevated temperatures and high workability.

4:40 PM
Australian R&D on Magnesium Alloys: *Gordon Dunlop*[1]; [1]CAST, UDP No 055, Univ. of Queensland, St. Lucia, Queensland 4072 Australia

The current expansion of useage of magnesium alloys in automotive and electronics applications has led to a significant upsurge in the amount of R&D that is being undertaken on these alloys. Much of this R&D is addressing important challenges for the successful development of magnesium applications, such as: improved recycling technology; improved die casting technology; design expertise; property data bases; replacement of SF6 cover gas; improved high temperature alloys; coatings and corrosion resistance; joining technologies; wrought alloys and processes. CAST is an Australian cooperative research centre focussed on light metals. The centre involves research organisations, universities, manufacturers and light metals producers as its members. The presentation will focus upon work that is being undertaken in Australia and elsewhere in the world to develop a better understanding of magnesium alloys and improved technologies for their processing and application.

5:00 PM
Magnesium Die Casting Alloy Design: *Ketil Pettersen*[1]; Per Bakke[1]; Darryl Albright[2]; [1]Norsk Hydro ASA, Light Metals Rsrch. Ctr., PO Box 2560, N-3907, Porsgrunn Norway; [2]Magnesium Market Development, Hydro Light Metals, 39209 W. Six Mile Rd., Ste. 200, Livonia, MI 48152 USA

The properties of an alloy are closely linked to the compositional and processing parameters. Magnesium applications demand different critical properties, such as creep resistance for automotive drive train components, ductility and energy absorption in safety parts, and high yield strength in structural parts. Consequently, alloy design must be targeted to achieve specific microstructural features for the various application areas. In the alloy development process, thermodynamics is an important tool in identifying promising candidate compositions. However, the phase morphology and material grain size are not easily predicted. The experimental work of casting test samples and evaluating them in the laboratory is thus an inevitable part of building new knowledge about the relationships between composition, microstructure and properties. This paper presents some fundamental considerations for designing magnesium alloy compositions, as well as results from investigations of selected die cast alloy samples.

5:20 PM
Research Strategy for AM60B Magnesium Steering Wheel: *Shae K. Kim*[1]; Hyung-Jo Yoo[2]; Young-Jig Kim[1]; [1]Sungkyunkwan University, Sch. of Metall. & Matl. Eng., 300 Chunchun-dong, Jangan-gu, Suwon, Gyeonggi-do 440-746 Korea; [2]Donghwa Enterprize Company, Ltd.

Driven largely by the desire to reduce the weight of products, the automotive industry in Korea is predicting significant growth in the use of magnesium alloys. As one of these efforts to adopt magnesium alloys in car components, the paper describes the research strategy for high pressure die castings of AM60B magnesium steering wheel. Design and die casting process optimization for AM60B magnesium steering wheel will be explained compared to other magnesium steering wheels. Design/Structure/Property relationship will be also presented.

Materials Issues Related to the Collapse of the World Trade Center

Program Organizer: Toni Marechaux, National Research Council, National Materials Advisory Board, 2101 Constitution Ave. NW, Washington, DC 20418 USA

Wednesday PM — Room: 611
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Toni Marechaux, National Research Council, National Materials Advisory Board, 2101 Constitution Ave. NW, Washington, DC 20418 USA

2:00 PM Opening Remarks

2:10 PM Invited
Why Did the World Trade Center Collapse? Science, Engineering, and Speculation: *Christopher Musso*[1]; Thomas W. Eagar[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA

There have been numerous reports detailing the cause of the World Trade Center collapse on September 11, 2001. Most have provided qualitative explanations; however, simple quantitative analyses show that some common conclusions are incorrect. For example, steel could not have melted in the Tower fires and there was more stuctural damage than simple softening of the steel at elevated temperatures. Some guidelines for improvements in future structures are also presented.

2:50 PM Invited
New Materials for Building Structures?: *John Hooper*[1]; [1]Skilling Ward Magnusson Barkshire, 1301 Fifth Ave., #3200, Seattle, WA 98101-2699 USA

Although new materials are being introduced into the building structures, their introduction is slowed by many factors including costs and the code approval process. Our ìnew materialsî, generally, relate to finding new ways to use familiar materials. Advances in analysis tools and methodologies has allowed this opportunity to extend the envelope and geometric application of traditional materials such as steel, timber and concrete as well as developing new concepts for nonstructural items such as curtain walls and stairs. Applications of these advances will be presented with additional discussion regarding the next generation provided. Research is underway that, over time, will allow for carbon, kevlar, mylar and other composites to enter building structure mainstream. Some examples of these opportunities will be presented and estimated time table regarding their use given.

3:30 PM Break

3:40 PM Invited
A Structural Engineerís Dream for Ideal Post 9/11 Construction Materials: Loring A. Wyllie[1]; [1]Degenkolb Engineers, 225 Bush St., Suite 100, San Francisco, CA 94104-4207 USA

A Structural Engineer uses existing construction materials to design many large complex structures to provide safety to its occupants. Connections must be adequate and designs consider some extraordinary conditions such as strong winds and earthquakes. With the recent wave of terrorist attacks there is a desire to toughen our structures to improve their performance under extreme and unexpected loadings. While some improved structural details will help, better materials would significantly improve performance for these unexpected conditions. The talk will focus on some of the material properties that Structural Engineers would consider ideal for survival under extreme conditions. Normally, material changes are relatively minor and focus on higher strength, improved chemistry to facilitate connections or improved ductility. Rather than trying to make minor changes to existing materials, the talk will step back and dream of new materials that would significantly improve our built environment for the unexpected loadings and conditions which may occur.

4:20 PM Invited
Virtual Materials and Service Life Design: *Geoffrey Frohnsdorff*[1]; Ken P. Chong[2] [1]National Institute of Standards and Technology, Guest Researchers, Building and Fire Research Lab., Rm. B368, Bldg. 226, Gaithersburg, MD 20899 USA; [2](On leave from the National Science Foundation)

The designer of a building, or other constructed asset, must exercise many judgments in ensuring that the intended life will be achieved without large unforeseen expenses. The generally-satisfactory performance of buildings attests to the generally-good judgment of designers. However, enough durability problems occur to suggest that more needs to be done to help ensure achievement of the intended service life at a reasonable life-cycle cost. Possibilities for increasing the likelihood of satisfactory long-term performance are: 1) establishing service life design standards, 2) providing reliable and realistic accelerated tests and models for predicting long-term behavior of materials and components, and 3) providing convenient access to reliable, easily-usable knowledge about the long-term performance of materials and components.

Materials Processing Fundamentals - IV

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

Wednesday PM — Room: 614
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Alvaro Giron, Alcoa Technical Center, Ingot & Solidification Platform, 100 Technical Dr., Alcoa Center, PA 15069 USA; Adam Powell, Massachusetts Institute of Technology, 17 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4301 USA

2:00 PM
Applications of High Frequency Induction Heating for Metallurgical Simulation and Thermal Analysis of Industrial Light Metals Casting Processes: *W. Kasprzak*[1]; W. T. Kierkus[1]; J. H. Sokolowski[1]; M. Kasprzak[2]; [1]NSERC/Ford-Nemak/University of Windsor, Mech., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Silesian University of Technology, B. Krzywoustego St. No. 2, Gliwice 44-100 Poland

Modern light metals processing technologies are very complex and involve a diverse set of variables that must be quantitatively evaluated. The most effective way to design new casting processes that deliver specified metallurgical and mechanical properties is to develop a method of simulating the real time process within a test sample of sufficient size. The results can then be analyzed and required optimizations can be performed. This paper presents a novel method of simulating the industrial casting processes in a laboratory environment using experimental apparatus based on the principle of high frequency induction heating. The applicability and accuracy of the proposed analytical method is demonstrated, as an example, by simulation of the solidification and heat treatment of a test sample composed of two dissimilar materials. The simulation can be used to develop a stress model for an automotive component made from an aluminum based alloy with iron cylinder liner inserts.

2:30 PM
Unsteady Mold Flow Analysis in Continuous and Direct Chill Casting Processes using Large Eddy Simulation: *Anirudh Mukhopadhyay*[1]; [1]Fluent, Inc., 10 Cavendish Ct., Lebanon, NH USA

Traditional design process of the Submerged Entry Nozzles (SEN) in Continuous Casting (CC) of steel and Direct Chill (DC) casting of aluminum often involves scaled water modeling to identify time average and unsteady flow features. Using Computational Fluid Dynamics along with the advanced turbulence model, Large Eddy Simulation (LES), transient mold flow analysis has been carried out. Results are compared with the experimental results of Xu et al (1998)*. These simulations showcase both time-averaged and transient flow features along with the effects of variation of port angles. Use of LES makes the quantitative assessment of turbulence more practical with transient and anisotropic large-scale vortical structures within the molds. This study illustrates the utility of CFD in optimizing the analysis effort by reducing the extent of water model experiments and plant trails. *D. Xu, W. K. Jones Jr., J. W. Evans and D. P. Cook, 1998, Applied Mathematical Modeling, vol. 22, pp. 883-893.

3:00 PM
Kinetics of Alumina Dissolution in Molten Slags: *Weol D. Cho*[1]; Peter Fan[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Fast dissolution of nonmetallic inclusions such as alumina particles in molten slags has been required in secondary steelmaking)ladle and tundish)to minimize the amount and the size of nonmetallic inclusions in steel products. In the present work, the dissolution behavior of alumina particles in molten CaO-Al2O3 and CaO-Al2O3-SiO2 slags at the temperatures between 1450 and 1550°C have been investigated in terms of kinetics and mechanism. Alumina particles were added directly into the slags and the variation of the particle size with time was determined using optical and electron microscopies. The conventional types of slag for ladle and tundish were included in the dissolution study. The effects of Al2O3, SiO2, CaF2 and MgO contents on the dissolution rate of alumina particles have also been determined. The diffusion boundary layer between alumina particle and bulk slag phase has been observed and analyzed using SEM and EDS. Based on the analysis of kinetic data and the boundary layer, the dissolution mechanism has been discussed.

3:30 PM Break

4:00 PM
Kinetics Aspects of Zinc Basic Carbonate Decomposition: *N. Kanari*[1]; D. Mishra[1]; I. Gaballah[1]; B. DuprÈ[2]; [1]Mineral Processing and Environmental Engineering Team, LEM, CNRS UMR 7569, ENSG, INPL, BP 40, 54501 Vandúuvre France; [2]Laboratoire de Chimie du Solide MinÈral, UHP Nancy I, UMR 7555, BP 239, 54506, Vandúuvre France

The thermal decomposition studies of two zinc basic carbonate samples were carried out up to 400∞C by thermogravimetric analysis, boat experiments and differential scanning calorimetry measurements. The initial samples and the solid decomposition products were analyzed by scanning electron microscopy, x-ray diffraction and laser particle size analyzer. The thermal decomposition of both samples started at about 150∞C and the rate of decomposition became significant at temperatures higher than 200∞C. The apparent activation energies ëEaí in the temperature range 150-240∞C for sample decomposition were between 132 and 153 kJ/mole depending on the sample origin. The XRD analyses of the intermediately decomposed samples and the DSC results up to 400∞C suggested that the decomposition reaction most probably occur in a single step.

Materials & Processes for Submicron Technologies - II: Microstructures, Properties & Characterization

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

Wednesday PM — Room: 209
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Ashok Kumar, University of South Florida, Ctr. for Microelect. Rsrch.; Sailesh M. Merchant, Agere Systems, VLSI Proc. Dvlp.

2:00 PM
Electrical and Compositional Properties of TaSi2 Films: *Vishal Mehta*[1]; Anthony T. Fiory[1]; [1]New Jersey Institute of Technology, Dept. of Phys., Newark, NJ 07102 USA

Electrical and compositional properties of sputter deposited TaSi2 films, as function of film thickness, is presented in this study. In particular, the effects of annealing of the TaSi2 fillms in steam ambients has been investigated. It is found that the films are very stable and at least within the limits of X-ray diffraction, no observable oxidation of the films has been detected. A comparison of the present study is made with the literature. The evolution of the structure of the films with film thickness is also investigated.

2:20 PM Invited
Process Parameter Impacts on the Properties of Cu Interconnects: *Jon Reid*[1]; John Sukamto[1]; [1]Novellus Systems

The transition from etched Al to electroplated Damascene interconnects is now well underway for the 0.13 micron device generation. The electrodeposition chemistries and process conditions required to achieve uniform within die plated thickness, complete filling of smaller high aspect ratio features, and low metal defect densities have evolved considerably from the chemistries and conditions initially introduced for IC applications. In many cases, the changes in accelerating, suppressing, and leveling additives, electrolyte component concentrations, and electroplating current profiles required to meet criteria such as void free filling have also influenced the metallurgy of the copper deposit. Important interconnect behaviors such as electromigration stability and via chain yield thermal stability are known to depend strongly on barrier properties and initial filling quality. The importance of deposit metallurgy to these behavior and a clear understanding of the electrodeposited metallurgy itself has not been well established. In this paper, deposit properties such as resistivity, stress, purity, void formation, and grain structure are studied at various stages of annealing as a function of deposition current, additive concentration and type, and electrolyte concentrations.

2:50 PM
Plastic Deformation of Electrodeposited Cu Foil at Low Homologous Temperatures: *Hans Conrad*[1]; Di Yang[1]; [1]North Carolina State University, Matls. Sci. & Eng., PO Box 7907, Raleigh, NC 27695-7907 USA

The plastic deformation kinetics of electrodeposited (EP) 18mm Cu (grain size vapor deposited (VP) Cu foil (d=0.5mm) tested at 77-473K. The apparent activation volume v=kTlne/s for both materials exhibited a minimum at ~350K, while the flow stress decreased monotonically. The values of v for the EP Cu foil at e[a] 0.05 were smaller than those for the EP foil determined at e=0.008. The rate-controlling mechanism in both materials at T< 350K appears to be grain boundary shear induced by dislocation pile-ups, that at T>350K may be either cross slip or dislocation climb controlled by dislocation pipe diffusion.

3:10 PM
Effects of Mechanical and Tribological Properties of Low-K Materials on Chemical Mechanical Polishing: Arun Kumar Sikder[1]; Frank Giglio[1]; John Wood[1]; *Ashok Kumar*[1]; Mark Anthony[1]; [1]University of South Florida, Ctr. for Microelect. Rsrch., Col. of Eng., 4202 E. Fowler Ave., Tampa, FL 33620 USA

In the 50 nm node, the International Roadmap for Semiconductors (ITRS) predicts a need for dielectrics with a bulk K value of <1.3 and effective K value <1.5 for the dielectric stack. Mechanical characterization of low-K materials has shown that lower K typically also means lower elastic modulus and hardness. Also the major challenges involves in chemical mechanical polishing (CMP) of these films due to the reduced modulus and cohesive strength of many low-K materials. In this study, we investigate the mechanical and tribological properties of low-K dielectric materials. The films surface properties along with the mechanical properties affect the within-wafer-nonuniformity, removal rate and roughness. The friction coefficient and acoustic emission signal was monitored during CMP process. The film surfaces were characterized using both SEM and atomic force microscopy. Effect of friction co-efficient, hardness and modulus on the polishing behavior was investigated. The validity of Prestonís equation was also discussed.

3:30 PM
Effects of Pad Characteristics on Material Removal Rate in CMP: Bo Yan[2]; *Ashraf Bastawros*[2]; *Abhijit Chandra*[1]; [1]Iowa State University, Mechl. Eng., 3038 Black Eng. Bldg., Ames, IA 50011 USA; [2]Iowa State University, AEEM Dept., Ames, IA 50011 USA

The role of the porous pad in controlling the material removal rate (MRR) during the chemical mechanical planarization (CMP) process has been studied numerically. The numerical results are used to develop a phenomenological model that correlates the forces on each individual polishing particles to the applied nominal pressure. The model provides physical explanation of experimentally observed domains of pressure dependent material removal rates, wherein the pad deformation controls the load sharing between active cutting particles and direct wafer-pad contact. The predicted correlations between MRR and slurry characteristics (i.e., particle size, concentration) are in agreement with experimentally measured trends.

3:50 PM Break

4:10 PM Invited
Characterization and Optimization of Copper CMP Planarization: *Thomas Laursen*[1]; Tezer Battal[1]; Ben Palmer[1]; Malcolm Grief[1]; [1]SpeedFam-IPEC

Abstract unavailable

4:40 PM
Experimental and Numerical Analyses on Mechanical Stress Evolution in Metal Interconnects for Various Line Aspect Ratios and Passivation Dielectrics: *Young Bae Park*[1]; In Soo Jeon[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

Mechanical stress in metal interconnect line is very important in large scale device integration because the metal line is mostly under a tri-axial tensile stress state that is favorable for void formation which is a major concern for the reliability of multi-level metallization. Both direct X-ray diffraction analysis and finite element analysis were used to investigate the effects of various passivation dielectrics and metal line aspect ratios on the mechanical stress-state in Al-Cu interconnect lines. Passivation dielectrics used in this study were PETEOS (Plasma Enhanced Tetra Ethyl Ortho Silicate), FOX (Flowable Oxide), HDP FSG (High Density Plasma Fluorinated Silicate Glass), and SiN (Silicon Nitride). A series of parallel interconnect lines with various aspect ratios (or, various line widths of 0.25 ~ 10µm at a constant thickness) and a constant pattern density of 50% were used for stress analysis. Plane-strain, elasto-plastic finite element analyses were carried out to calculate thermal stress by using Youngís modulus of each constituent layer measured with nanoindentation method. Finally, average thermal stresses from finite element analysis were compared to experimentally measured estimates by considering not only residual stresses and mechanical properties of passivation dielectrics but also their characteristic deposition shapes.

5:00 PM
Improvement of Form Accuracy in Hybrid Machining of Microstructure: *Lim Han Seok*[1]; A. Senthilkumar[1]; M. Rahman[1]; [1]National University of Singapore, Dept. of Mechl. Eng., 10, Kent Ridge Crescent 119620 Singapore

Micro machining is gaining popularity due to the recent advancements in Micro Electro Mechanical Systems. Using conventional micro machining it is relatively difficult to produce moving components in the order of microns. Therefore an attempt is made in this paper to build microstructures using a combination of turning and electrochemical machining. Several sets of experiments were performed to study the characteristics of the hybrid machining process. From the experiments it was observed that higher form accuracy could be obtained by integrating the on machine fabrication of machining tool and subsequently using the tool for machining the workpiece. The main cause of the form error is due to the deflection of the shaft while machining and hence an attempt was made to develop the deflection sensor. The deflection error was observed using the developed sensor and it was compensated using the feedback mechanism thus considerably improving the form accuracy of the component. The procedure involved and the results obtained are discussed in detail in this paper.

5:15 PM
A Study on the Grinding of Glass using Electrolytic Inprocess Dressing: *A. Senthilkumar*[1]; M. Rahman[1]; Lim Han Seok[1]; K. Fathima[1]; [1]National University of Singapore, Dept. of Mechl. Eng., 10, Kent Ridge Crescent 119620 Singapore

Grinding of brittle materials like glass is gaining importance due to the rapid development in the areas such as machining of storage devices, micro lenses and optical communication devices. Grinding of such glasses are difficult due to their brittle nature and the grinding wheel worn out easily due to the hardness of the material being machined. Therefore an electrolytic in process dressing (ELID) of the grinding wheel is used to dress the wheel continuously so as to make the fresh diamond grit to protrude and grind the glass under ductile mode machining in order to yield surface roughness in the order of nanometers. In this paper a fundamental study on the mechanism of ELID grinding technique is discussed in detail. Several sets of experiments were performed to determine the optimal grinding conditions. From the experiments it was observed that surface roughness could be improved if the current duty ratio to dress the grinding wheel is increased. The force patterns and the changes in the profile of the grinding wheel are presented and discussed in detail.

Modeling of Multi-Scale Phenomena in Materials Processing: Process 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

Wednesday PM	Room: 304
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chair: Adrian S. Sabau, Oak Ridge National Laboratory, Metals & Cer., Bethel Valley Rd., Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

2:00 PM

Simulation of Fine Particle Dynamics in Materials Processing Systems: *Aniruddha Mukhopadhyay*[1]; Abhijit Dutta[1]; Eric William Grald[1]; Evan Whitby[2]; Frank Stratmann[2]; Martin Wilck[2]; [1]Fluent, Inc., 10 Cavendish Ct., Lebanon, NH 03766 USA; [2]Chimera Technologies, Inc., 15051 Zodiac St. N.E., Forest Lake, MN 55025 USA

In many industrial processes particles are either a desired product, such as TiO2 powder, or an unwanted contaminant, such as large particles in chemical vapor deposition reactors. Successful development of such systems requires understanding and controlling processes governing particle formation, growth, transport, and deposition. These processes can be simulated with computational fluid dynamics (CFD) based particle dynamics models. Because no general-purpose, commercial software exists for such particle-dynamics simulations, Chimera Technologies and Fluent are developing the Fine Particle Model (FPM), which is a CFD-based Eulerian model that solves for coupled fluid flow, heat transfer, chemical reactions, and particle dynamics. Typical applications include nano-particle sprays, chemical reactors, pollution formation/transport, and particle inhalation/transport. In this talk we describe processes simulated with the FPM and show typical examples.

2:25 PM

Characterization of Embedded Fiber Optical Sensors: Jill Noel Johnsen[1]; Joanna R. Groza[1]; Fritz Prinz[2]; Xiaochun Li[2]; [1]University of California, Chem. Eng. & Matl. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Stanford University, Palo Alto, CA USA

The benefit of Laser Assisted Shape Deposition Manufacturing (LASDM) and other rapid prototyping processes is the ability to create physical models with little or no restriction from shape complexity. Silica fibers have been embedded into real parts to be used as in-situ sensors for stress and temperature measurements during their operation. The parts produced and characterized for this study consist of layers of stainless steel, nickel, and fibers. First, a layer of nickel was electrolytically deposited on a stainless steel support. Then fibers, which were sputter coated with a thin metal film, were placed on this layer of nickel and topped with another layer of electrolytically deposited nickel, followed by a stainless steel deposit by LASDM. The deposited stainless steel was created with a 20 mm/s velocity, a 2000 W laser power, and a 20 g/min deposition rate. Microstructural studies of each layer and interfaces were conducted using SEM and optical microscopy. The nickel grain structure was found to be very uniform around the fibers both before and after laser deposition and the fibers showed good adhesion to the matrix. In situ stress and temperature measurements were successfully performed on fiber embedded parts to test the validity of this approach.

2:50 PM

Evaluation of Field Activated Sintering of Silicon: *Martin Kramer*[1]; Joanna R. Groza[1]; [1]University of California, Matls. Sci. Dept., Davis, CA 95616 USA

A kinetic study on sintering pure, fine grained silicon powder by field activated sintering using the Sumitomo SPS sintering system has been carried out. Densities better than 97% could be achieved by tailoring temperature and pressure profiles. Densification kinetics as a function of different heating rates and uniaxial pressures have been determined to derive experimental values for activation energies. The results are discussed in terms of kinetics and the technical capabilities and potential of the sintering system addressing both problems of temperature distribution throughout the specimen and furnace setup which have been studied by finite element modeling and experiments.

3:15 PM Break

3:30 PM

Assessment of Diffusion between Nickel Base Superalloys: Rob Marsh[1]; *Carelyn E. Campbell*[2]; Ty W. Hansen[1]; Boyd A. Mueller[1]; William J. Boettinger[2]; [1]Howmet Research Corporation, Elect. Prod. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA; [2]National Institute of Standards, Metall. Div., Gaithersburg, MD 20899 USA

The availability of thermodynamic databases for up to 15 components developed through the Calphad approach has permitted the prediction of solidification path in commercial superalloys. The enthalpy and the derived density information predicted for these paths has been employed in the modeling of macroscopic heat transfer and fluid flow phenomena in real parts. An area that has received considerably less attention is the solid state diffusion processes which occur during heat treatment and coating operations. This presentation will report on experimental diffusion couple studies for a series of times between various nickel base superalloys such as Rene N4 and Rene N5 at temperatures in the single phase gammaí region. The couples were analyzed by microprobe to map the extent of diffusion. The experimental results are compared to numerical predictions of the code DICTRA using the available thermodynamic database and a mobility database assessed from diffusion data in the various binary and ternary subsystems.

3:55 PM

Brownian Dynamic Simulation of the Rheology of Dense Colloids: *Andrew Martin Mullis*[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Dense colloidal suspensions are structured fluids displaying complex rheological properties, including both shear thinning and shear thickening. One route to understanding such systems is Brownian Dynamics, a particle dynamics technique in which hydrodynamic interactions between the particles are ignored. Simulations containing thousands of particles are feasible, yielding statistical information about the level of structure in the fluid. Brownian Dynamics thus spans several length scales relating interparticle forces to the suspension microstructure and hence the rheology of the bulk slurry. We will give an overview of the technique illustrated by simulations of a dense (55% volume fraction solid) suspension of repulsively electrostabilized particles. Such suspension are observed to undergo a shear thickening transition with increasing applied shear, related to an order-disorder transition in the microstructure. This sequence is reproduced in the simulations with progressive loss of order being observed as the applied shear is increased.

4:20 PM

Multi-Scale Modeling of Packed Bed: *Subrata Pal*[1]; A. K. Lahiri[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore 560 012 India

The modeling studies of the counter current gas-solid reactors are usually done assuming all solids as continuum. In this approach the transport processes occurring in the individual particle are neglected. A multi-scale model of char burning in a moving bed has been carried out. The temperature profile in the char bed as well as in the char particle has been computed considering all the possible reactions. Cylindrical and spherical coordinate systems have been used for the reactor and the particle respectively.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - III

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

Wednesday PM — Room: 211
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Matls. Sci. & Eng., 442 Whitaker Lab., Bethlehem, PA 18015 USA; C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chung-li, Taiwan

---

2:00 PM Invited
Ripening-Controlled Solder Interfacial Reactions: *King-Ning Tu*[1]; [1]UCLA, Matls. Sci. & Eng., 6532 Boelter Hall, Los Angeles, CA 90095-1595 USA

Kinetic models of reactive phase formation with planar interfaces in solid state reactions are well established. Whether they are diffusion-controlled or interfacial-reaction-controlled models, no surface and interfacial energies are taken into account. In solid state aging of a solid solder (e.g., eutectic SnPb) on a solid conductor (e.g., Cu), the intermetallic compound (IMC) formed between them has a layer-type morphology. But in the wetting reaction between molten solder and a solid conductor, the IMC formed between them has a scallop-type morphology. The interface of the scallops is non-planar and the growth of an average size scallop has been found to show t 1/3 dependence, suggesting that it may be controlled by ripening. The growth rate of scallop-type IMC is four orders of magnitude faster than that of the layer-type. To develop a kinetic model of ripening-controlled interfacial reactions, we have confirmed that the scallop-type morphology is thermodynamically stable in the wetting reaction, because of minimization of interfacial and grain boundary energies. Among the kinetic processes in the growth of scallops, the growth itself or ripening becomes the slowest step because the scallops are 3-dimensional. We propose that the interfacial reaction is ripening-controlled.

2:25 PM Invited
Morphology and Growth Kinetics of Solder/Substrate Interfacial Reaction Products using Tin-Based Solders: *Michael R. Notis*[1]; Sarah L. Allen[1]; Ben K. Jackson[1]; Rick P. Vinci[1]; [1]Lehigh University, Matls. Sci. & Eng., 442 Whitaker Lab., Bethlehem, PA 18015 USA

The nature and kinetics of growth of interfacial reaction products that form during soldering using lead-free tin-based alloys on copper substrates is of significant concern for the manufacture of many commercial electronic component and package configurations. This paper will first review the literature and our own studies concerning the morphology and kinetics of growth of interfacial reaction products in liquid-solid and solid-solid tin/copper diffusion couples. These results will be compared with growth studies involving silver-tin binary and silver-copper-tin ternary solders on copper substrates. In the silver-containing solders, long needle-like growths of Ag3Sn appear to nucleate and grow into the solder from the interfacial region. We have used a deep-etching method to examine the nucleation and growth morphology of this Ag3Sn phase. Suggestions as to the reasons for rapid anisotropic phase growth will be made, and possible effects on mechanical properties will be discussed.

2:50 PM Invited
Strong Effect of Cu Concentration on the Reactions between SnAgCu Lead-Free Solders and Ni: C. E. Ho[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City Taiwan

The electronic industry recently reached a consensus that the 95.5Sn-3.9Ag-0.6Cu (wt.%, Sn-3.9Ag-0.6Cu) was the most promising lead-free candidate to replace the lead-bearing solders. In this study, the effect of a small perturbation in Cu concentration was studied. Specifically, two solders, Sn-3.9Ag-0.2Cu and Sn-3.9Ag-0.6Cu, were reacted with Ni at 250°C. It was found that the Cu concentration had a strong effect on the reaction. When Sn-3.9Ag-0.2Cu was reacted with Ni, a continuous $(Ni_{1-x}Cu_x)_3Sn_4$ intermetallic layer formed at the interface. When Sn-3.9Ag-0.6Cu was reacted with Ni, a continuous $(Cu_{1-y}Ni_y)_6Sn_5$ intermetallic layer formed at the interface. These two intermetallic compounds might produce solder joints with very different strength. The results of this study shows that the industry must impose a very strict control over the Cu concentration in order to produce the desired intermetallic compound that has the right mechanical properties.

3:15 PM Invited
Mechanisms of Channel Formation and its Effects on Kinetics of Intermetallic Layer Growth at Solder/UBM Interface: Jong-Hyun Lee[1]; Jong-Hwan Park[1]; *Yong-Seog Kim*[1]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Seoul 121-791 S. Korea

Mechanisms of channel formation at solder/UBM interface were investigated by studying orientation of scallop formed at the interface and penetration behavior solders into intermetallics such as Cu6Sn5 and Ni3Sn4. Orientation of scallops analyzed by EBSD technique indicated that the scallops grow in a preferred direction and their grain boundaries are of high angle in character. The penetration speed of solders into the intermetallic, especially the Cu6Sn5, was measured to be orders of magnitude faster than the growth rate of the intermetallics during reflow soldering. These results indicate that the channels at solder/UBM interface are thermodynamically stable throughout the reflow soldering process and formed by wetting of solders into the grain boundaries of intermetallics. The effects of channel presence on the growth kinetics of intermetallics at solder/UBM interface were analyzed theoretically and measured experimentally.

3:40 PM Break

4:00 PM Invited
Prediction of Primary Intermetallic Compound Formation during Interfacial Reaction between Sn-Based Solder and Ni Substrate: Won Kyoung Choi[2]; *Hyuck Mo Lee*[1]; Sang Won Jeong[1]; [1]KAIST, Dept. of Matl. Sci. & Eng., Kusung-Dong 373-1, Yusung-Gu, Taejon Korea; [2]IBM Watson Research Laboratory, PO Box 218, Yorktown Heights, NY 10598 USA

A modified methodology to predict the primary intermetallic compound (IMC, hereafter) phase that forms first during soldering at the interface between Sn-based solders and metal surfaces is suggested. By comparing the activation energy for nucleation of individual IMC phases, which is a function of the interfacial energy and the driving force for formation, the IMC phase that forms first is predicted as the one with the smallest activation energy. This methodology has been successfully applied to observations of IMC phases that form during liquid soldering on Cu and Ni plates.

4:25 PM Invited
Variation of Melting Temperature upon Particle Size Reduction: *Kwang-Lung Lin*[1]; [1]National Cheng Kung University, Matls. Sci. & Eng., 1 Ta-Hsuey Rd., Tainan 701 Taiwan

Solder paste applies solder powder for forming microelectronic interconnection. Pb-free solder development generally attempts to search for eutectic system in order to lower the reflow temperature. It however has been known that the melting temperature of a material can be lowered by particle size reduction. This present work performed an analysis on the behavior of melting temperature depression, basing on classical thermodynamic theory, for several metals. It is shown that there exists a particle diameter, termed as critical radius of fusion, at which the latent heat of fusion of a fixed mass equals the total surface energy of the particles. It was found that this radius is in the range of nano-size. The depression of melting temperature also becomes quite prominent in the range of nano-size. This suggests a possibility of performing solder reflow at lower temperature than the melting temperature of the bulk solder.

4:50 PM Invited
Two Phase Equilibrium in Electronic Materials and its Application in Advanced Devices: *Choong-Un Kim*[1]; ManJong Lee[1]; Nancy Michael[1]; Qing-Tang Jiang[2]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., Woolf Hall, Rm. 325C, Arlington, TX 76019-0031 USA; [2]International Sematech, Austin, TX 78741-6499 USA

The proximity to equilibrium is important to all material systems, including electronic devices. Equilibrium seeking behavior can be exploited to develop new classes of electronic materials or it can drive damage mechanisms. This paper will present two examples of materials designed to actively use two-phase equilibrium and one example of device damage resulting from interface instability. The first example is the use of solid-solid phase equilibrium to efficiently develop semiconductor quantum-dots. Semiconductor alloys with limited mutual solubility, for example PbTe-4HgTe, can be processed to induce precipitation in a controlled manner. The resulting nanocrystals are self-assembled and self-aligned. The second example is the use of low-temperature solder alloys in a solid-liquid equilibrium state as a thermal interface material. In the two-phase state, the alloy reduces thermal resistance far better than any existing contact material. Finally, the role of solid-gas equilibrium during electromigration failure of submicron Cu interconnects will be introduced.

Second International Symposium on Ultrafine Grained Materials: Mechanical Behavior

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

Wednesday PM — Room: 210
February 20, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Amiya K. Mukherjee, University of California, Dept. of Chem. Eng. & Matls. Sci., Davis, CA 95616 USA; Evan Ma, Johns Hopkins University, Dept. Matls. Sci. & Eng., 105 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

2:00 PM Keynote
Dislocation Activity in Nanocrystalline Model Metals: Richard C. Hugo[2]; Harriet H. Kung[2]; *Julia R. Weertman*[1]; [1]Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

The deformation behavior of nanocrystalline metals at very small grain sizes (<~20 nm) has been the subject of experiments, theories, and molecular dynamics simulations. It has been predicted that dislocation activity ceases and grain boundary sliding becomes dominant at the ultrafine grain sizes. Results of these studies will be reviewed and recent experiments described of in situ straining in the TEM of Ni nanocrystalline films to look for dislocation activity. This research was partially supported by the Department of Energyís Office of Basic Energy Research and by the LDRD program at the Los Alamos National Laboratory.

2:25 PM Invited
Current Views on the Fatigue Performance of Ultrafine-Grained Metals: *Hael Mughrabi*[1]; Heinz Werner Hoeppel[1]; [1]University Erlangen-Nuernberg, Inst. fuer Werkstoffwissenschaften, Martensstr. 5, Erlangen D-91058 Germany

The most important factors that control the cyclic deformation behaviour and fatigue life of ultrafine-grained (UFG) materials have become clearer in recent time. Interesting aspects of the earlier work were that the strength enhancement related to the strongly reduced grain size, paired with an enhanced strength and a reduced ductility, led to an improved fatigue resistance in W^hler (S-N) plots but to reduced fatigue lives in Manson-Coffin plots. In addition, the mode of testing (stress or strain control) plays a role. The microstructural origin of fatigue damage was identified as macroscopic shear banding in combination with dynamic grain coarsening of the highly meta- or unstable deformed microstructure. Dynamic grain coarsening can obviously occur in UFG materials at homologous temperatures as low as 0.2. Application of a suitable annealing treatment has been shown to improve the stability of the UFG microstructure and to lead to an enhancement of the fatigue performance in the entire range in S-N and in Manson-Coffin plots. These aspects of fatigue of UFG materials are reviewed with reference to the potential of enhancing the fatigue performance of commercial engineering materials.

2:45 PM
Corrosion Fatigue of Ultra-Fine Grain Copper Fabricated by Severe Plastic Deformation: *Hiroyuki Miyamoto*[1]; Takashi Yamasaki[1]; Takuro Mimaki[1]; Alexei Vinogradov[2]; Satoshi Hashimoto[2]; [1]Doshisha University, Mech. Eng., 1-3 Miyakodani Tatara, Kyotanabe, Kyoto 610-0321 Japan; [2]Osaka City University, Dept. Intelligent Matls. Eng., Osaka 558-8585 Japan

Corrosion fatigue is investigated of ultra-fine grain (UFG) copper fabricated by severe plastic deformation through equal-channel angular pressing (ECAP). The corrosion fatigue behaviour in 1M NaNO2 aqueous solution is compared with results of testing in air under low-cyclic regimes with constant plastic strain amplitudes Depl/2 ranged from 2x10-3 to 5x10-2. The significant enhancement of fatigue life in the UFG state is achieved in comparison with polycrystalline copper of ordinary grain size. It is shown that a higher overall resistance against corrosion fatigue can be obtained after grain reduction via ECAP. Detailed structural and morphological investigations are performed on different stages of cycling. While transgranular crack nucleation occurs in ordinary polycrystals, the intergarnular fracture occurs in the UFG material. It is shown that cyclic softening which occurs both in air and in aggressive environment is associated with abnormal grain growth. Formation of a dislocation structure characteristic for persistent slip bands (PSBs) is observed evidently in the grains coarsened during fatigue. The PSB-grain boundary interaction is supposed to facilitate strongly the intergranular fatigue fracture which is particularly pronounced in corrosion fatigue studies where the grain boundaries are preferentially attacked by aggressive spices. The findings of the present investigations are discussed in light of former fatigue experiments performed on ultra-fine grained copper and results of general corrosion stress-corrosion tests. The mechanism of intergranular corrosion fatigue damage in the UFG copper is proposed in terms of corrosion-deformation interactions.

3:00 PM
Texture and Elastic Strain Development during Mechanical Loading of Equal Angle Channel Processed Nickel and Aluminum: *Bjorn Clausen*[1]; Sven Vogel[2]; Ersan ‹st¸ndag[1]; Cheng Xu[3]; Donald W. Brown[2]; Mark A.M. Bourke[2]; Terence G. Langdon[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. 8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA; [3]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., 854 W 36th Place, Los Angeles, CA 90089-1453 USA

Severe plastic deformation (SPD) methods allow producing nanostructured metals and alloys. SPD processed metals and alloys can have very high yield strength while high ductility is maintained, a unique and extremely attractive combination of properties. Among other SPD methods, equal-channel angular processing/pressing (ECAP) is an efficient method of refining grain size of metals or composites to the order of 100 nm via severe plastic deformation. We will present results of neutron diffraction measurements investigating texture, grain size and residual strains as a function of ECAP cycles. By the very nature of neutron diffraction experiments, these results are averaged over the bulk of the sample. Additionally, the in-situ measured hkl-dependent response on uniaxial loading of ECAPíed samples will be shown. The neutron diffraction results will be compared with findings from a self-consistent modeling approach, which will be applied to ultrafine-grained materials for the first time.

3:15 PM
Dry Sliding Wear Behavior of Ultrafine Grained Commercial Purity Aluminum and Low Carbon Steel Produced by Severe Plastic Deformation Techniques: *Yong-Suk Kim*[1]; Taio Lee[1]; Kyung-Tae Park[2]; Woo-Jin Kim[3]; Dong Hyuk Shin[4]; [1]Kookmin University, Sch. of Metlgcl. & Matls. Eng., 861-1 Chonung-dong Songbuk-ku, Seoul 136-702 S. Korea; [2]Hanbat National University, Div. of Adv. Matls. Sci. & Eng., Taejon 300-717 S. Korea; [3]Hong-Ik University, Dept. of Metall. & Matls. Sci., Seoul 121-791 S. Korea; [4]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Kyunggi-Do 425-791 S. Korea

Dry sliding wear behavior of ultrafine grained commercial purity aluminum (1100) and low carbon steel (Fe-0.15%C-0.25%Si-1.1%Mn) was investigated. The ultra fine grains of the aluminum and the steel were obtained by an accumulative roll bonding (ARB) process and equal channel angular processing (ECAP), respectively. Pin-on-disk wear tests of the ultrafine grained specimens were carried out under various applied load and sliding speed conditions using hardened bearing steel as a counterpart material. Contrary to the increase of the hardness and yield strength, wear resistance of the ultra-fine grained aluminum and steel specimens was lower than the large grained as-received ones and the resistance decreased with the increase of the accumulative plastic strain. Worn surfaces and cross-sections of the worn specimens were examined with SEM and TEM to investigate the wear mechanism and to explain the wear behavior of the ultrafine-grained aluminum and steel specimens with the microstructural change at the wearing surface.

3:30 PM
Properties and Microstructure of Alumina-Niobium Nanocomposites made by Novel Processing Methods: *Joshua D. Kuntz*[1]; Julin Wan[1]; Goudong Zhan[1]; Amiya K. Mukherjee[1]; [1]University of California, Dept. of Chem. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Alumina-niobium nanocomposites have been fabricated using high-energy ball-milling and electric field assisted sintering (EFAS) or high pressure sintering (HPS). The 10 volume percent niobium nanocomposites have a fracture toughness of ~ 6 MPa√m with only a marginal decrease in hardness. This is nearly twice as tough as a pressureless sintered composite of the same composition reported in work by Garcia et al [J. of Am. Cer. Soc. 1998; 81:429]. This increase in toughness can be attributed to the novel microstructure in the nanocomposites. The present study shows a metallic phase distribution of ~20 nm particles along with a continuous 3-4 nm layer at the boundaries between alumina grains. This microstructure should lead to toughening by increasing ductility at the crack tip instead of the traditional ligament bridging in the crack wake which is typical of metallic-phase toughened ceramics. This material is based upon work supported by, or in part by, the US Army Research Laboratory and the US Army Research Office under contract/grant number G-DAAD19-00-1-0185.

3:45 PM Break

3:55 PM Invited
Strain Hardening, Strain Rate Sensitivity, and Plastic Instabilities in Nanocrystalline and Ultrafine-Grained Metals: *Evan Ma*[1]; D. Jia[1]; Y. M. Wang[1]; K. T. Ramesh[1]; [1]Johns Hopkins University, Ctr. for Adv. Metals & Cer., 3400 N. Charles St., Baltimore, MD 21218 USA

Fe and Ti with grain sizes in the nanophase to micrometer range have been prepared by severe plastic deformation, either through mechanical milling of powders [1] or ECAP [2]. The deformation behavior of the bulk samples prepared through these routes have been studied using quasi-static as well as high strain rate tests. Our focus is to monitor, as a function of plastic strain, the strain hardening behavior and the strain rate dependence of the flow stress, as well as their effects on the deformation/failure mode, especially uniform deformation versus localization and the tensile necking instability. Our experiments indicate that the nanocrystalline (nc) and ultrafine-grained (UFG) metals appear to lose the mechanisms for defect accumulation inside the grains and hence the capacity for strain hardening. Meanwhile, they tend to exhibit a low strain rate sensitivity (m). As a result, the tendency for plastic instabilities and catastrophic failure is enhanced: (adiabatic) shear banding is promoted in lieu of uniform deformation, and in tension necking instability sets in early [2]. For consolidated Fe, shear banding becomes the dominant mode of deformation from the onset of the plastic deformation [1]. Due to the vulnerability to the localized deformation/failure modes, high-strength nc and UFG materials are very limited in uniform deformation and elongation to failure in tension, or under high-rate conditions, even when they are ductile at conventional grain sizes or in compression tests. Some preliminary results will also be reported on the development of some bcc nc or UFG alloys for military kinetic energy penetrators, where the shear banding mode and self-sharpening capability during high rate deformation are desired. [1] Scripta mater. 42, 73 (2000); Acta mater. in review. [2] Appl. Phys. Lett. July 23 issue (2001).

4:15 PM
Mechanical Properties of Ultrafine Grained Aluminum and Ultra Low Carbon Steel Produced by ARB Process: *Nobuhiro Tsuji*[1]; Yuichiro Koizumi[1]; Yoritoshi Minamino[1]; Yoshihiro Saito[2]; Yoshinori Ito[3]; [1]Osaka University, Dept. of Adaptive Machine Sys., 2-1Yamadaoka, Suita, Osaka 565-0871 Japan; [2]Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [3]Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Mechanical properties of aluminum and ultra low carbon steel sheets with various ultrafine grain sizes above 200nm were clarified. 1100 aluminum sheet and IF steel sheet were highly strained up to an equivalent strain of 6.4 by ARB (accumulative roll-bonding) process. The ARB processed sheets were filled with pancake shaped ultrafine grains whose mean thickness is about 200nm. Annealing at various temperatures produced various grain sizes ranging from 200nm to 10 micrometers. The strength of ultrafine grained materials held Hall-Petch relationship with the grain size. However, the Hall-Petch slope of tensile strength was much smaller than that of yield strength, which suggests the decrease in work-hardening with decreasing grain size. Uniform elongation suddenly dropped around the grain size of 1 micro-meter, where tensile strength nearly meets yield strength. The sudden decrease in uniform elongation could be explained by plastic instability. Superior toughness of ultrafine grained steel will be also reported.

4:30 PM
An Evaluation of Homogeneity in Ultrafine-Grained Aluminum Prepared by Equal-Channel Angular Pressing: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-channel angular pressing (ECAP) is a processing technique capable of producing materials with submicrometer grain sizes. Microhardness tests were conducted to investigate the homogeneity of samples of pure aluminum subjected to ECAP via different processing routes. The results show that the samples achieve homogeneity throughout most of their volume after a single pressing and the homogeneity is further improved with increasing numbers of passes through the ECAP die.

4:45 PM
Mechanical Properties of Nanostructured Low-Carbon Steels Produced by Conventional Cold-Rolling and Annealing of Martensite Starting Microstructure: *Rintaro Ueji*[1]; Nobuhiro Tsuji[1]; Yuichiro Koizumi[1]; Yoritoshi Minamino[1]; Yoshihiro Saito[2]; [1]Osaka University, Dept. of Adaptive Machine Sys., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [2]Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

The present authors have developed simple thermomechanical processing without intense straining to obtain ultrafine grained bulky steel sheets with both high strength and adequate ductility. Plain low carbon steels (JIS-SS400 and SM490) were austenitized and water-quenched to obtain martensite as starting microstructure. As-quenched sheets were conventionally cold-rolled to a strain of 0.8, and subsequently annealed at temperatures of 673K to 973K. The SS400 sheets annealed at around 823K exhibited the multiphase microstructure composed of ultrafine grained ferrite with grain size of 50-300nm, fine cementite uniformly precipitated, and tempered martensite. Owing to this multiphase structure, the nanostructured sheets had both high strength (710MPa 0.2% offset stress and 870MPa tensile strength) and adequate ductility (9% uniform elongation and 20% total elongation). The nanostructured material impact tested at 103K showed ductile fracture even at this low temperature and the fracture surface revealed very fine dimples.

5:00 PM
Microstructure and Properties of 7475 Aluminum Alloy after Equal-Channel Angular Pressing: *Jingtao Wang*[1]; Cheng Xu[2]; Zheng Zhang[1]; Xicheng Zhao[1]; Lizhong Wang[1]; Terence G. Langdon[2]; [1]Xiían University of Architecture & Technology, Metlgcl. Eng., Yanta Rd., No. 13, Xiían 710055 China; [2]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-Channel Angular pressing (ECAP) was successfully applied to a 7475 aluminum alloy at temperatures from room temperature to 500°C up to an equivalent true strain of ~12 using routes C or Bc. The grain size was refined to within the range from ~0.3 to 1.9 micrometers by ECAP at these different temperatures. There appears to be a grain refinement limit which is dependent on the ECAP temperature. This paper reports investigations on microstructural characterization and tensile properties of the processed aluminum alloy.

5:15 PM Panel Discussion
Terence G. Langdon, Terry C. Lowe, Ruslan Z. Valiev, Michael J. Zehetbaue

Surface Engineering: Science & Technology II: Advances in Coating Technologies

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

Wednesday PM	Room: 203
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Kiyoshi Yatsui, Nagaoka University of Technology, Nagaoka, Nigata 940-2188 Japan; Y. F. Lu, National University of Singapore 119260 Singapore

2:00 PM
Tribolgy of Oxide Based Matrix Composite Coatings with Lubricant Inclusions: *J. S. Zabinski*[1]; T. Fitz[1]; J. E. Bultman[1]; J. J. Hu[1]; A. A. Voevodin[1]; [1]Air Force Research Laboratory, Wright Patterson AFB, OH 45433-7750 USA

Oxides offer thermal stability and oxidation resistance, but typically exhibit high friction, are brittle, and may create abrasive wear debris. Their limitations can be overcome by controlling microstructure and by creating solid lubricant inclusions in composite structures. Yittria stabilized zirconia and alumina were selected as matrix materials and their microstructure was adjusted to maximize toughness through control of lubricant chemistry and particle size. The lubricants studied were gold, molybdenum disulfide, diamond-like carbon, and cesium oxythiomolybdate. They were selected as inclusions in the oxide matrices to permit low friction and wear resistance across vacuum, moist air, and high temperature environments. Pulsed laser deposition combined with simultaneous magnetron sputtering was used to create composites with 2-10 nm lubricant inclusions. The coatings were tough and exhibited low friction in vacuum, dry nitrogen, and moist air to 650°C. The plasma chemistry and physics required for growth of nanocomposites were identified and the tribology of the coatings is discussed in terms of their chemistry, microstructure, and mechanical properties.

2:25 PM Cancelled
Results of Tensile Cracking Experiments with WC/DLC: *Nekkanty Srikant*

2:40 PM
Development of Cermet Thin Film Coatings: *Brajendra Mishra*[1]; Jinhui Zhou[1]; Frank Kustas[2]; [1]Colorado School of Mines, Metlgcl. Eng., 920 15th St., Golden, CO 80401 USA; [2]Engineered Coatings, Inc., 15422 Winterleaf Ct., Parker, CO 80134 USA

Co-deposited thin films of TiC/W, B4C/Mo and TiC-TiB2/W have been fabricated using magnetron sputtering, under conditions of varied working gas pressures, substrate bias and metal concentrations to develop high fracture toughness in combination with high hardness, good adhesion and enhanced wear-resistance. Coatings have been characterized using scratch adhesion, indentation, and sliding wear tests. Films have exhibited very good adhesion with critical scratch adhesion values from 50-70N, with slightly higher values measured for metal-containing films with an adhesion layer. Observations from Rockwell C Brale indentations to compare relative toughness, suggest that adding a moderate amount (from 20 to 40 atomic percent) of metal to the ceramic coatings reduced the damage diameter around the indentations, and resulted in a less-severe cracking mode (fine radial cracking observed for metal-containing films). This suggests that cermet coatings have higher coating/substrate interfacial toughness than pure ceramic coatings. Nanoindentation of the cermet coatings resulted in hardness values in the range of 20-30 GPa, with higher values for coatings with higher metal content. Reciprocating sliding wear tests were performed on the coatings using a tungsten carbide-cobalt (WC-Co) ball counterface. At a lower initial stress of 1.33 GPa, addition of metal to the carbide matrices resulted in higher wear factors, whereas at a higher initial stress of 1.91 GPa, metal-containing ceramic films had lower wear factors than for the pure ceramic coatings. This behavior suggests a load (or stress) sensitive wear performance. The data will be presented to demonstrate the advantages of metal incorporation in ceramic matrix thin films in applications requiring high tribological performance.

2:55 PM
Tribological and Structural Studies of Diamond-Like-Carbon and Fluorocarbon Thin Films: *Jason W. Goldsmith*[1]; Brajendra Mishra[1]; Eli Sutter[1]; John J. Moore[1]; Mark Crowder[2]; Robert Turner[2]; [1]Colorado School of Mines, ACSEL/MME, 1500 Illinois St., Golden, CO 80401 USA; [2]Maxtor Corporation, 2452 Clover Basin Dr., Longmont, CO 80503 USA

Fluorinated diamond-like carbon thin films have physical properties that may prove beneficial at tribological interfaces. The tribological surfaces of a magnetic recording head and disk are coated with very thin, diamond-like-carbon (DLC) films and the disk has an uppermost coating of a near monolayer of a fluorocarbon polymer. The substitution of one or both of these surfaces with a fluorinated DLC may improve the tribological and surface properties of the head-disk interface. In the present work we have carried out deposition of fluorocarbon films using plasma deposition technique and characterize the surface properties. The chemical composition of the deposited fluorocarbon films has been investigated using Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy and Raman spectroscopy. The film structure was studied using atomic force microscopy and cross-sectional transmission electron microscopy. The chemical and structural properties of the fluorocarbon films as well as their wear, coefficient of friction, wettability with water, hardness, fracture toughness, index of refraction, dielectric constant, and resistivity were compared to DLC films. The relationship between deposition conditions, structure, and properties is discussed.

3:10 PM
Role of Precursors on the Formation of CN_x Deposited by PE-HF-CVD: *Daniela Dumitriu*[1]; Rosendo Sanjines[1]; *Ayatolah Karimi*[1]; [1]Swiss Federal Institute of Technology Lausanne, Dept. of Phys., Lausanne CH-1015 Switzerland

We prepared CN_x films with N content up to 30 at% using various precursors including ammonia, dimethylamine, 3-amino-1,2,4-triazole, methane, ethene and their halogenated derivatives. Using precursors in which N is directly bonded to C, was found to improve the film properties in terms of N incorporation and hardness. Halogenated precursors increase deposition rate and decrease substrate temperature at which deposition occurs, from 700°C down to 200°C. The level of doping with halogen is below 2 at%. The H content of the film drastically decreases when unsaturated compounds are used. This paper deals with the relationship between the growth mechanisms and films properties.

3:25 PM Invited
Tailored Nanomaterial-Based Hard Coatings on Metal and Polymer Substrates: *Ganesh Skandan*[1]; Amit Singhal[1]; [1]Nanopowder Enterprises, Inc., 120 Centennial Ave., Piscataway, NJ 08854 USA

A great deal of emphasis has been placed recently on developing nanostructured films and coatings on metallic and polymeric substrates to impart improved wear properties. The interest stems from the fact that the mechanical behavior of nanostructured materials appears to be significantly different from its coarse grained counterpart. Our recent work shows that the maximum benefit is achieved when the structure of the coating is a composite of micron and nano scale features, and is suitably tailored for any given application. We have successfully applied this concept to two classes of protective coatings: (1) tungsten carbide-cobalt coatings on metal substrates, wherein the agglomerated powder, used as feedstock material in a high velocity oxy-fuel thermal spray gun, is composed of a mixture of micron and nanoscale WC grains, and (2) optically transparent hard coatings on polycarbonate substrates, wherein the coating is composed of relatively large ceramic particles dispersed in a polymer nanocomposite matrix. The structure and properties of these ìmultimodalî coatings, as they are now called, will be discussed.

3:50 PM Break

4:05 PM Invited
Ultrananocrystalline Diamond: Synthesis, Properties and Applications: *A. V. Sumant*[1]; O. Auciello[1]; D. M. Gruen[1]; J. A. Carlisle[1]; J. Birrell[1]; N. Moldovan[2]; D. Mancini[2]; D. Ersoy[3]; E. Stach[4]; A. Erdemir[5]; [1]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL USA; [2]Argonne National Laboratory, Experimental Facility Div., Argonne, IL USA; [3]University of Illinois, Dept. of Mechl. Eng., Chicago, IL USA; [4]Lawrence Berkeley Laboratory, Natl. Ctr. for Electron Micros-

copy, Berkeley, CA USA; [5]Argonne National Laboratory, Energy Tech. Div., Argonne, IL USA

Diamond is an ideal material for various applications because of its outstanding physical, chemical, mechanical, and electronic properties. Conventional CVD diamond deposition methods have been explored to produce diamond-based microelectromechanical system (MEMS) devices. However, these methods, based on hydrogen-rich plasma chemistries, result in either coarse-grained pure diamond layers with extremely rough surfaces (≥1 µm rms roughness) or in fine-grained diamond films with a significant amount of intergranular nondiamond carbon (~ 50%), which limits its use to specific applications. At Argonne National Laboratory, we are able to produce phase-pure ultrananocrystalline diamond (UNCD) films grown by microwave plasma CVD using C60-Ar or CH4-Ar plasmas. This technique yields films that have 3-5 nm grain sizes and atomically abrupt grain boundaries. In addition, recent studies on N2 doping have shown that this material can have tunable electrical conductivity from insulating to semimetallic. The work at ANL has demonstrated that UNCD films have unique combined mechanical, tribological electron emission, electronic, electrochemical, and biological compatible properties that make this material applicable to a broad range of multifunctional devices from field emitter cold cathodes to MEMS and biocompatible devices. The synthesis and properties of UNCD films will be reviewed in view of these multiple applications.

4:30 PM

Characteristics of Boron Carbide Films Prepared by Cathodic Arc Deposition: *Othon R. Monteiro*[1]; Marie-Paule Delplancke-Ogletree[2]; C. Christopher Klepper[3]; [1]Lawrence Berkeley National Laboratory, One Cyclotron Rd., MS 53-004, Berkeley, CA 94720 USA; [2]UniversitÈ Libre de Bruxelles, Indl. Chem., 50 ave. F. D. Roosevelt, CP 165/63, Bruxelles 1050 Belgium; [3]HY-Tech Research Corporation, 104 Centre Ct., Radford, VA 24141 USA

Boron carbide films are attractive materials for use as tribologcal coatings for low and high operating temperatures, especially for applications that require high hardness. We have investigated properties of boron carbide films prepared by cathodic arc using a heated B_4C cathode. The film composition is very close to that of the cathode. The effect of substrate bias on the film characteristics was studied. Hardness, elastic modulus, scratch resistance and coefficient of friction were determined for films prepared at bias voltages of: 0V, -100V and -500V. Surface morphology was also imaged with scanning electron microscopy and atomic force microscopy. It was observed that hardness and modulus increased by over 30% when a -100V bias was applied to the substrate.

4:45 PM

Mechanical Properties and Microstructural Evaluation of Chromium Containing Hard Coating on Fe-Mn-Al Alloy: *Jyh-Wei Lee*[1]; Jenq-Gong Duh[2]; [1]Tung Nan Institute of Technology, Dept. of Mechl. Eng., Taipei 222 Taiwan; [2]National Tsing Hua University, Dept. of Matls. Sci. & Eng., R405, 4th Eng. Bldg., Hsinchu 300 Taiwan

Engineering components with hard coatings usually exhibit improved service behavior and increased lifetime after appropriate surface modification. Pack chromization and r.f. magnetron sputtering techniques were employed to deposit chromium carbides and chromium nitride, respectively, on the surface of austenitic Fe-30.6Mn-6.8Al-0.9C alloy. The $(Cr,Fe)_2N_{1-x}$ and $Cr_{23}C_7$, phases were found on the chromized surface, while the internal part of chromized layer was Cr_7C_3 phase. The CrN phase was observed in the sputtered film. The phase transformation and related microstructure of chromium containing hard coatings and matrix were studied with X-ray diffractometer (XRD) and electron probe microanalyzer (EPMA). The hardness and elastic modulus of deposited coatings were evaluated by an indentation and cracking tester (ICT). Scratch tests were further conducted to investigate the adhesion of hard coatings. The conformal cracking and chipping failures were found in scratch track of chromized layer, whereas the buckling and chipping failures were observed in CrN coating. It is concluded that the chromized layer exhibits better microhardness and adhesion properties than that of sputtered film on Fe-Mn-Al alloy in the present study.

5:00 PM

Mechanical Characterization of Polymers and Glass using Instrumented Indentation: *Ethel PoirÈ*[1]; [1]Micro Photonics, Inc., 21 Morgan Ste. 100, Irvine, CA 92618 USA

Making small imprints and measuring optically the diagonals of indents is a well-established technique used to determine the hardness of materials and coatings. This technique can unfortunately not be used for every application. In some cases, measurement can be complicated by the tiny size of the indent or by the fact that the indent cannot be seen under the microscope because of the surface texture/appearance. This paper will present specific cases for which instrumented indentation is more advantageous as a mean to measure hardness. Instrumented indentation is based on the analysis of the load-penetration curve measured while doing an imprint. It is computer-controlled and it is not influenced by the operatorís judgment of the imprintís shape. Youngís modulus and fracture toughness ranking will also be presented

5:15 PM

Comparison in Characteristics of Electroless Deposited and Magnetron Sputtered Ni-P-W Coatings: *Fan-Bean Wu*[1]; Yi-Ying Tsai[2]; *Jenq-Gong Duh*[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 429R, 4th Eng. Bldg., Hsinchu 300 Taiwan; [2]Industrial Technology Research Institute, Opto-Electr. & Sys. Labs., 908R, 51st Bldg., Chutung 310 Taiwan

Two kinds of different coating techniques, the electroless deposition and the magnetron sputtering, were employed to deposit the Ni-P-W alloy onto the stainless steel substrate. It was found that the multi-target sputtering including binary Ni-P and single W target provided a better control of the tungsten concentration in the Ni-P-W coating. Through X-ray phase identification, the sputtered Ni-P-W exhibited amorphous phase due to the co-sputtering of the tungsten, whereas metastable Ni_xP_y phases were observed in the binary Ni-P coating. The precipitation of the Ni_3P phases was retarded by the tungsten element in the coating, especially in the sputtered Ni-P-W thin film after heat treatment up to 450°C, which was higher than that for the ternary electroless Ni-P-W coating. In addition, adhesion strength and scratch failure modes for both the electroless and magnetron sputtered Ni-P-W coating were also evaluated.

5:30 PM

Cathodic Process of Electrodeposited RE-Ni-W-P-SiC Composite Coating: *Zhongcheng Guo*[1]; Xiaoyun Zhu[1]; Dacheng Zhai[1]; Keyi Ma[1]; [1]Kunming University of Science and Technology, Fac. of Matl. & Metal. Eng., Kunming 650093 China

Cathodic deposition current density of the composite coatings increases when SiC particles and rare earth (RE) were added in the bath,and it is profitable for Ni-W-P alloy to deposit in the cathod, forming Ni-W-P-SiC and RE-Ni-W-P-SiC composite coatings. On the contrary, the addition of PTFE in the bath decrease cathodic depositing current density of the coatings. The current density increases a little when the amount of RE is 7~9 g/l; however, the density increases greatly with increasing amount of RE, and it reaches peak value when the amount of RE is 11~13 g/l. But if the amount of RE is raised further, the current density decreases.

Teaching and Learning Hydrometallurgical Science and Engineering: Dissolution & Aqueous Solutions

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

Wednesday PM	Room: 601
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Thomas W. Chapman, National Science Foundation, Div. of Cheml. & Transport Sys., 4201 Wilson Blvd., Ste. 525, Arlington, VA 22230 USA; Kenneth N. Han, SD School of Mines and Technology, Matls. & Metall. Eng., 501 E. St. Joseph St., Rapid City, SD 57701-3995 USA

2:00 PM Keynote

Dissolution of Metals in HydrometallurgyñAn Interdisciplinary Subject: *Kenneth N. Han*[1]; [1]SD School of Mines and Technology, Matls. & Metall. Eng, 501 E. St. Joseph St., Rapid City, SD 57701-3995 USA

Hydrometallurgy covers a wide spectrum of areas and its knowledge base is enlarged and enhanced by contributions from many other fields. Dissolution of metals and alloys in aqueous media will be used to illustrate how rich the field of metal extraction can be. Comprehensive understanding of the fundamentals of metal and alloy dissolution in aqueous systems requires creative application of thermodynamic prinicples, solution chemistry, ligand complexation theory, electrochemistry, transport phenomena, and heterogeneous kinetics. In addition, recent developments in instrumentation in the fields of interfacial phenomena and corrosion have tremendously advanced the field of metal extraction in aqueous systems. The close relationship between metal extraction in aqueous reactions and various physicochemical phenomena occurring in corrosion and interfacial systems will be discussed and illustrated with examples.

2:35 PM Invited

An Electrochemical Model for the Oxidation of Chalcocite by Ferric Sulfate: *David G. Dixon*[1]; *Samuel A. Bolorunduro*[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Previous kinetic studies of chalcocite oxidation have been carried out without controlling the redox potential, which is expected to fall drastically as oxidant is consumed. Considerable uncertainties arise in the interpretation and modeling of such data. In this work, oxidation of high grade chalcocite minerals in acidic ferric/ferrous sulfate solutions was investigated under controlled redox potentials. Two stages of chalcocite oxidation were observed and the rate of reaction is described by an electrochemical model in which the mixed potential is determined principally by the reversible potentials of the mineral and that of the oxidant. The first stage reaction was rapid at all temperatures and the rate is controlled by mass transfer of the oxidant to the mineral surface. The second stage reaction was characterized by the conversion of the secondary covellite to cupric ions and elemental sulfur, and the mixed potential corresponds to the reversible potential of the ferric/ferrous couple, leading to a fractional order dependence of rate on the ferric/ferrous ratio.

3:00 PM Invited

Potentiometric Measurement of pH in High Temperature Aqueous Processes: *Vladimiros G. Papangelakis*[1]; Dhehinie S. Seneviratne[1]; Xiangyang Zhou[2]; Serguei N. Lvov[2]; [1]University of Toronto, Dept. of Cheml. Eng. & Appl. Chem., 200 College St., Toronto, Ontario M5S 3E5 Canada; [2]The Pennsylvania State University, The Energy Inst. & Dept. of Energy & Geo-Environmental Eng., University Park, PA 16802 USA

The measurement of pH in pressure hydrometallurgy is gradually becoming a reality. In spite of significant advancements in the chemical modelling of high temperature aqueous systems, actual measurement of pH constitutes an important and necessary verification tool of aqueous thermodynamics. Our recent developments in this area are summarised in this communication. A novel experimental technique for measuring pH in high-temperature and concentrated process sulphate solutions is presented. An Yttria-Stabilized Zirconia sensor (YSZ) is tested on synthetic solutions simulating the hydrometallurgical processing of nickeliferous laterites by pressure acid leaching. A flow-through Ag/AgCl electrode (FTERE) is also used as an external reference electrode. Experiments were conducted using a custom-made flow-through titanium electrochemical cell specially designed for high temperature potentiometric pH measurements. Measurements were performed on simple and on progressively more complex multicomponent electrolyte solut ions containing sulphuric acid and sulphates of aluminum and magnesium. An acceptable agreement was found between the measured pH values and those predicted by theoretical calculations.

3:25 PM Invited

Systematic Modelling of Heap Leach Processes for Optimisation and Design: *Jochen Petersen*[1]; David Grant Dixon[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Design and operation of heap leach processes are still largely based on operator experience and rule of thumb. Although considered a low-cost, low-tech route for mineral extraction, the underlying physical, chemical and, in some cases, biological processes are actually rather complex and elude simple mathematical description. Numerous attempts have been made to model heap leaching, but the majority of models grossly simplify the true process and have remained useful only for idealised scenarios in the laboratory. There is, however, considerable interest in the minerals industry to approach design and operation of heap leach processes in a much more rigorous fashion to achieve efficient and rapid mineral extraction. To this end, a systematic process model of the heap is needed. The modelling approach presented here combines the more conventional reaction-diffusion model with models accounting for heat conservation, chemical speciation in solution, bacterial growth and propagation, 3D axis-symmetrical bulk adve ction-diffusion flow, gas flow, etc. The model versatility and applicability are demonstrated with a number of scenarios investigated in recent laboratory and field studies. It becomes clear that both, a flexible modelling tool and a systematic laboratory/pilot investigation, are necessary to fully appreciate the complexity of any particular heap leach scenario. Once such a comprehensive model is in place, however, optimisation of existing operations or optimal design of new heaps are relatively straightforward.

3:50 PM Break

4:00 PM Invited

Magnetic Activated Carbon: An Old Product with a New Future: *Saskia Duyvesteyn*[1]; Gustavo A. Munoz[1]; Jan D. Miller[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

While the concept of activated carbon that is magnetic is not new, a new magnetic activated carbon (MAC) product has been developed at the University of Utah. The MAC is produced by mixing a fine particulate carbon source with a magnetic precursor and subjecting the mixture to a series of chemical and heat treatments. The fine particle size results in rapid adsorption kinetics, and the magnetic properties allow for recovery from solutions or slurries by means of magnetic separation. The characteristics of MAC for gold adsorption from alkaline cyanide solutions, such as capacity, selectivity, and elution will be discussed in terms of particle size, surface area, and, of particular importance, in terms of surface functional groups. The influence of surface modification on uptake of heavy metal ions, such as Cu, Cd and Zn, will be discussed as well.

4:20 PM Invited

A Tutorial on Synthesis and Use of Adsorbents for Hydrometallurgical Separations: *Lawrence L. Tavlarides*[1]; [1]Syracuse University, Cheml. Eng., 334 Hinds Hall, Syracuse, NY 13244 USA

The presentation will describe methods to synthesize adsorbents for targeted metal ion separations for the hydrometallurgical industry. Functional group selection and various methods of sorbent synthesis such as covalent attachment and sol-gel techniques will be described. Characterization of these materials will be illustrated with specific examples. The utility of several sorbents will be shown for examples such as noble metal separation and acid mine drainage cleanup.

4:45 PM Invited

NSF Programs Related to Hydrometallurgy: *Thomas W. Chapman*[1]; [1]National Science Foundation, Div. of Cheml. & Transport Sys., 4201 Wilson Blvd., Ste. 525, Arlington, VA 22230 USA

The mission of the National Science Foundation (NSF) is to advance the nationís welfare by promoting scientific progress. Specific goals involve the development of people, ideas, and tools for science and engineering. This presentation will provide an overview of NSF programs that are relevant to the advancement of hydrometallurgy. These programs include support of not only academic research but also educational development and collaboration between universities and industry. Examples will be given of specific research projects funded by NSF and related to hydrometallurgical processes.

5:10 PM Invited

A Short History of Hydrometallurgy: *Fathi Habashi*[1]; [1]Laval University, Dept. of Mining, Metlgcl., & Matls. Eng., Quebec City, Quebec G1K 7P4 Canada

Thousands of years ago people had learned how to build furnaces and use fire to melt rocks and produce metals but the use of water and aqueous solutions for ore processing came much later, mainly at the time of the alchemists when acids and alkalies became known and used. Modern hydrometallurgy, however, can be traced back to the end of the nineteenth century when two major operations were discovered: the cyanidation process for gold and silver extraction and the Bayer Process for bauxite treatment. Later, in the 1940ís, a breakthrough came during the Manhattan Project in USA in connection with uranium extraction. Since then, it has been advancing progressively and even replacing some pyrometallurgical processes. Canadian contribution is significant particularly in the recovery of uranium, nickel, cobalt, and zinc.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Zinc, Lead and PGM's

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead, Zinc, and Tin 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

Wednesday PM	Room: 607
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Andreas H. Siegmund, RSR Technologies Incorporated, 2777 Stemmons Hwy., Ste. 1800, Dallas, TX 75207 USA; Markus A. Reuter, Delft University of Technology, Appl. Earth Scis., 2628 Rx Delft, Mijnbouwstraat 120, Delft 2628 RX The Netherlands

2:00 PM

The Behaviour of Iron as Impurity in Zinc Roasting: *Satu Jyrkˆnen*[1]; Pekka A. Taskinen[1]; Maija-Leena Mets‰rinta[1]; Aija Rytioja[2]; Jens Nyberg[2]; [1]Outokumpu, Outokumpu Rsrch. Oy, PO Box 60, Pori FIN-28101 Finland; [2]Outokumpu Zinc Oy, PO Box 26, Kokkola FIN-67101 Finland

In Kokkola Zinc smelter as well as in most other roasting plants it is a trend towards using concentrates with increasing impurity content nowadays. This also often means that the size distribution of the concentrates gets finer. Iron is present with highest percentage as a single impurity. The behavior of iron in zinc roasting was studied in laboratory experiments. Commercial zinc concentrates with low and high (1.5-16 wt-%) iron content as pyrite or in the lattice of sphalerite were partially roasted to establish the reaction mechanisms of iron in roasting. Microscopical studies were carried out on the calcine, and the findings were presented in numerous photographs of the polished cross sections. SEM/EDS was used for analyzing the phases present in the samples. A calcination mechanism of zinc concentrates with high iron content was suggested.

2:25 PM

Agglomeration in Zinc Fluidized Bed Roasters: *J. Pierre Constantineau*[1]; John R. Grace[1]; Greg G. Richards[2]; C. J. Lim[1]; [1]University of British Columbia, Chem. & Bio. Eng., 2216 Main Mall, Vancouver, BC V6T 1Z4 Canada; [2]Cominco Research, Trail, BC V1R 4S4 Canada

The use of fluidized beds for the roasting of zinc sulphide concentrates has been of major benefit in the electrolytic production of zinc. The operation consists of reacting zinc sulphide with air or oxygen-enriched air to produce zinc oxide (calcine), subsequently acid-leached, and sulphur dioxide, directed to a sulphuric acid plant. The fluidized bed roasting process aims at maximizing the sulphide conversion, minimizing sulphate and zinc ferrite production, while maintaining excellent productivity. Stable operation of fluidized bed zinc roasters requires an adequate particle size distribution to minimize the risk of defluidization and to help maintain carryover within manageable levels. However, the understanding of the roasting process is limited and changes in operating conditions mainly rely on intuition, experience, and trial and error. A research program has been initiated at UBC to study the phenomena that influence the particle size distribution of the fluidized bed. This paper will present the approach, describe the apparatus that has been constructed and provide some preliminary results on the agglomeration phenomena occurring during fluidized bed roasting of industrial zinc concentrates.

2:50 PM

Improvements of Zinc Roaster Waste Heat Boiler: *Juha J‰rvi*[1]; Jussi Vaarno[1]; Jens Nyberg[2]; Heikki Siiril‰[2]; [1]Outokumpu Research Oy, PO Box 60, Pori FIN-28101 Finland; [2]Outokumpu Zinc Oy, PO Box 26, Kokkola FIN-67101 Finland

Outokumpu Zinc Oy has two separate roasting lines with fluidized bed furnaces and waste heat boilers for calcine collection and heat recovery. During the last 30 years the zinc production in Kokkola plant has continuously increased and at the same time raw-materials have changed. Therefore the waste heat boiler has been under continuous research and development. The waste heat boiler has a significant role in the process and therefore the reliability and the efficiency of the device is important for the effectiveness of the entire process. Many changes have been made to the boiler during the last decades. The new boiler design and optimization were carried out by using physical water and air-dust modeling as well as computational fluid dynamics. The modeling work agree well with experiences in practice. The latest developments have increased the operating hours per year and the effectiveness of the waste heat boiler.

3:15 PM

Technology for Processing of Lead-Bearing Materials from Copper Metallurgy at KGHM Polska Miedz S.A.: *Ryszard Prajsnar*[1]; Zbigniew Smieszek[1]; Jozef Czernecki[1]; Seweryn Plucilski[2]; Marian Warmuz[2]; Leszek Garycki[2]; [1]Institute of Non-Ferrous Metals, ul. Sowinskiego 5, 44-100 Gliwice Poland; [2]KGHM Polska Miedz S.A., HM Glogow, ul. Zukowicka 1, 67-231 Zukowice Poland

During copper producting in shaft and flash smelting processes at KGHM Polska Miedz S.A., lead-bearing material of sulfide, sulfate, and oxide character is generated such as slime from shaft furnace gas handling systems, converter dusts, and dusts from an electric furnace for copper removal from flash smelting slag. These materials are polymetallic concentrates of Pb, Zn, and Cu, with As, Cl, and Cd as impurities. Shaft furnace slime, containing about 50% PbS and 15% bituminous substances, is the main lead-bearing material. At the Glogow copper mill, a new plant was commisioned in June, 2001 to process 80,000 tonnes per year of lead bearing materials to produce 26,000 tonnes per year of crude lead. The plant consists of a feed conditioning system, three rotary rocking furnaces with a capacity of 16 tonnes of lead feed fired with natural gas and oxygen-enriched air containing up to 45 vol% O_2, and a gas handling system. The gas handling system includes a post-combustion chamber, a gas cooling chamber with Cl neutralization, gas mixing chamber, bag filter, and dry limestone scrubbing system for SO_2 removal. The lead-bearing feed is smelted with 5wt% sodium carbonate and 13wt% iron scrap at a temperature of 950-1050°C for 3 hours. The smelting process consists of 4 stages; feed drying, firing of gaseous hydrocarbons and solid coal from the feed, lead reduction from slag, and separation of molten products. The flow rates of natural gas and oxygen to the burners are controlled to ensure combustion of the bituminous material in the furnace. The process produces crude lead bullion containing (wt%) 0.1 to 0.5% Cu, 0.02 to 0.5% As, 0.05 to 0.07% Bi, 0.1 to 0.5% Sb, 0.1 to 0.6%Sn. The process also produces a micture of sodium silicate slag, Fe-Zn-Pb-Cu-S matte, and Fe-As speiss containing (wt%) 5 to 10% Pb, 2 to 6% Cu, 10 to 15% Zn, 1 to 3% As, 10 to 15% S, 0.5 to 3.0% Cl, 2 to 4% K, 4 to 7% Na, 20 to 30% Fe, and 5 to 10% SiO_2. The dust is made up of PbO, ZnO, $PbSO_4$ and $PbCl_2$, KCl, and NaCl with a chemical composition (wt%) of 35 to 50% Pb, 5 to 15% Zn, 3 to 6% As, 4 to 8% Cl, 2 to 5% K, and 2 to 5% Na. The construction of the new lead production plant at the Glogow copper mill will process both current lead-bearing materials as well as lead-bearing material that have been stockpiled over the last 15 years to give a significant improvement in protection of the natural environment.

3:40 PM Break

3:55 PM

ConRoast: DC Arc Smelting of Dead-Roasted Sulphide Concentrates: *Rodney T. Jones*[1]; [1]Mintek, Pyrometall. Div., PB X3015, Randburg 2125 S. Africa

Mintek has developed and piloted a novel process for the treatment of nickel-copper and PGM (platinum group metal) sulphide concentrates. The ConRoast process is based on the removal of sulphur by roasting, followed by smelting of the dead-roasted concentrate in a DC arc furnace, using an iron-based alloy as a collector for nickel, copper, cobalt, and PGMs. The environmental benefits with respect to sulphur emissions are considerable, in that essentially all of the sulphur is removed from the enclosed roasting equipment in a continuous stream of SO2 of an appropriate strength for feeding to a sulphuric acid plant. This process allows great flexibility with respect to the selection of ore types, and does not impose limits on the minimum quantities of contained base metals or sulphur, and can tolerate very high contents of chromite in the concentrate. The furnace alloy is water-atomized prior to leaching. Iron may be rejected from the alloy hydrometallurgically, by precipitation as hematite for example. The ConRoast process achieves very high metal recoveries, and produces high-purity metals, and a clean high-grade PGM concentrate.

4:20 PM

Online Prediction of Actual Melt Chemistry in an Ausmelt Converter using a Thermodynamic-System Identification Hybrid Modeling Technique: *Jacques J. Eksteen*[1]; G. A. Georgalli[1]; Markus A. Reuter[2]; [1]University of Stellenbosch, Dept. of Cheml. Eng., PB X1, Matieland, Western Cape 7602 S. Africa; [2]Delft University of

Technology, Dept. of Raw Matls. Tech., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

Lance based copper-nickel matte converters have become an established technology in the field of base metal sulphide smelting and converting. An Ausmelt“ converter is currently being constructed at Anlgo Platinum in South Africa to convert a nickel-copper rich matte containing platinum group metals (PGMís). The accurate prediction of the matte chemistry is required to control the composition at the endpoint which influences segregation of PGMís during the subsequent slow cooling and magnetic separation processes. A hybrid modelling approach is introduced in this paper that incorporates both the effect of thermochemistry and system dynamics. It will be shown how the model relates to fundamental conservation laws, while still providing enough flexibility to adapt to changing operating conditions. It will also show off-line modelling can be incorporated into a robust on-line model. Based on the data from many pilot plant trials, the model prediction performance is shown to be within the accuracy requirement set by industry and therefore constitutes a reliable basis for model based control.

4:45 PM

Platinum from a Chrome FurnaceñItís a Matter of Design: *Bruce Nourse*[1]; J. Malan[1]; R. Moolman[1]; H. Joubert[1]; [1]Pyromet Technologies (Pty.), Ltd., PO Box 61582, Marshalltown 2107 S. Africa

The depressed market for Ferrochrome worldwide has forced most producers in this industry to scale down production. Inevitably, many furnaces have been mothballed for prolonged periods, awaiting an upturn in the Ferrochrome market. In contrast, the Platinum industry is experiencing relative stability combined with good prices and they have been looking for cash cows to take advantage of these market conditions. It is, therefore, rather convenient that Platinum and Ferrochrome producers are virtually neighbours in the Bushveld complex of South Africa, since there is an abundance of spare furnace capacity around (albeit for Ferrochrome). Against this backdrop, Pyromet Technologies (Pty) Ltd, who are currently designing a slag cleaning furnace for Anglo Platinum, was requested to perform pilot scale testwork on a 200kW furnace, in order to establish the suitability to process platinum concentrate and converter slag in a furnace designed for Ferrochrome. This paper provides an overview of the configuration, test conditions, operation and results achieved during the testwork. It also discusses the critical issues, such as refractory consumption, copper cooling and electrical parameters that need to be addressed when converting a Ferrochrome Smelter into a Sulphide Smelter.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Sulfide Smelting Fundamentals II

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead, Zinc, and Tin 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

Wednesday PM	Room: 609
February 20, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Fathi Habashi, Laval University, Dept. of Mining, Metlgcl. & Matls. Eng., Quebec City, Quebec G1K 7P4 Canada; Adrian C. Deneys, Praxair Inc., 777 Old Saw Mill River Rd., Bldg. 765, Tarrytown, NY 10591-6714 USA

2:00 PM

Thermophysicochemical Database for Batch or Continuous Processes in Nickel Smelting and Converting: *F. Kongoli*[1]; I. McBow[1]; S. Llubani[1]; [1]Flogen Technologies, Inc., 5757 Decelles Ave., Bureau 511, Montreal, QC H3S 2C3 Canada

A computerized thermophysicochemical database is being developed for various processes involved in nickel smelting and converting. This database includes optimized models for slag, matte, liquid and solid alloys in the multicomponent system(s) as applied to nickel smelting and converting processes, batch or continuing ones. This database can be used to quantify the multicomponent and multiphase slag/matte/alloy/gas phase equilibria, liquidus and solidus surface, tie-lines, solubility and/or losses of nickel and other components in the slag, distribution coefficient, components activities, heat and mass balance, etc. The database also includes optimized models for the viscosity of the slags associated with any of the above processes. Both databases are coupled and this constitute a very effective method to quantify the effect of each component on several parameters of the smelting and converting processes and to diffuse the considerable confusion that exist in the literature. Several application examples have been given to demonstrate the usefulness of this database for quantitative prediction of the thermophysicochemical properties of the phases involved in actual or developing processes, even in the areas where experiments are difficult or impossible. The considerable cost reduction that results from the use of this database as compared to other methods in the improvement of existing technologies or in the development of new ones is discussed along with continuing new developments of this database.

2:25 PM

Quantification of the Liquidus Surface of Some Iron Oxide Slags of Industrial Interest at Constant CO2/CO Ratio: *Florian Kongoli*[1]; Akira Yazawa[2]; [1]Flogen Technologies, Inc., Metall., 5757 Decelles Ave., Ste. 511, Montreal, QC H3S 2C3 Canada; [2]Tohoku University, 16-32, Niizaka, Aoba-ku, Sendai 981-0934 Japan

Liquidus surface of multicomponent slags is an important parameter in various smelting and converting processes. It helps not only to optimize the slag chemistry of current processes and their fluxing strategies, but also to determine the availability of new slags for more advanced technologies. In a series of our previous publications, the liquidus surface of some multicomponent iron oxide slags has been quantified at several constant oxygen potentials and the effect of the latter, ignored until that moment, was quantified along with the effect of some minor components. In this work, the liquidus surface of some iron oxide slags is quantified at constant CO2/CO ratios. This is a new convenient way for the quantitative description of the slag liquidus surface as well as of the effect of several fluxes, especially in those processes, such as slag solidification, where the oxygen potential changes continuously. This type of diagram also describes more dynamically the effect of oxygen potential, clarifies the relation between CO2/CO ratio and oxygen potential in terms of the liquidus surface (not widely understood by metallurgist today) and reduces the gap between laboratory work and industrial experience.

2:50 PM Invited

Phase Relations and Activities in the Cu-Fe-S-As and Cu-Fe-S-Sb Systems at 1473K: Dexter G. Mendoza[1]; Hector M. Henao[1]; Mitsuhisa Hino[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Knowledge of thermodynamic properties of minor elements in the copper matte is of practical importance for understanding their behaviors in the copper smelting processes. In the present study, phase relations and activities of arsenic and antimony as minor elements in each miscibility gap in the Cu-Fe-S-As and Cu-Fe-S-Sb systems were determined at 1473K by using a quenching method and a double Knudsen-cell mass spectrometer method, respectively. The distribution ratios of arsenic and antimony between the matte and metal phases were around 0.1, that means both elements distribute preferentially into the metal phase. The molar ratio of NFe/NCu in the miscibility gap had no serious effect on the distribution ratio. The activity coefficients of arsenic and antimony in the matte phase were found to be about one order of magnitude larger than those in the metal phase. By using the obtained data, elimination of arsenic and antimony in the copper phase by means of slagging was discussed and the fractional distribution of these elements among the gas, slag, matte and metal phases was thermodynamically evaluated.

3:15 PM Invited

Phase Equilibrium between Ni-S Melt and Slags under Controlled Partial Pressures: *Hector M. Henao*[1]; Mitsuhisa Hino[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. for Multidisciplinary Rsrch. for Adv. Matls., Katahira 2-1-1, Aoba-Ku, Sendai, Miyagi 980-8577 Japan

To provide thermodynamic data for converting nickel matte to liquid nickel at high temperature, an experimental study was conducted on the phase equilibrium between the Ni-S alloy and the FeO_X-SiO_2, FeO_X-CaO or CaO-Al_2O_3 base slag in a MgO crucible at 1773 K under controlled PSO_2 of 0.1 atm and PO_2 in a range between $5x10^{-7}$ and 10^{-5} atm by using CO-CO_2-SO_2 gas mixtures. The distribution ratio of nickel between the slag and alloy phases at a given PO_2 was found to be minimum for the CaO-Al_2O_3-MgO slag with NCaO/NAl_2O_3 molar ratio of 1.7. The solubility of nickel in this slag at PO_2 of $6x10^{-6}$ atm (just before the precipitation of solid NiO) was 5 mass %. These results

WEDNESDAY PM

suggest the availability of $CaO-Al_2O_3$ base slag in converting the Ni-S melt to nickel.

3:40 PM Break

3:55 PM Moved to Monday AM
Kinetics of Sulfide Smelting in Mitsubishi Process: *Zenjiro Asaki*

4:20 PM
Predicting Phase Equilibria in Oxide and Sulphide Systems: *John A. Gisby*[1]; Alan Dinsdale[1]; Ian Barton-Jones[2]; Alan Gibbon[3]; Pekka A. Taskinen[4]; Jeff R. Taylor[5]; [1]National Physical Laboratory, NPL Matls. Ctr., Queens Rd., Teddington, Middlesex TW11 0LW UK; [2]Rio Tinto, PO Box 50, Castlemead, Lower Castle St., Bristol BS99 7YR UK; [3]Mineral Industry Research Organisation, 75 Peppard Rd., Sonning Common, Berkshire RG4 9RN UK; [4]Outokumpu Research Oy, PO Box 60, Pori FIN-28101 Finland; [5]Johnson Matthey Technology Centre, Sonning Common, Reading, Berkshire RG4 9NH UK

Phase equilibria for multi-component systems can be calculated reliably from critically assessed thermodynamic data for smaller sub-systems using software packages like MTDATA, written at the National Physical Laboratory (NPL). Models and data developed in an ongoing Mineral Industry Research Organisation (MIRO) project make such calculations possible for the complex systems encountered during sulphide smelting, involving interactions between matte, slag, metal and gas phases. Predictive calculations can be made which are directly relevant to industrial processes, covering composition and temperature ranges beyond the scope of hard-copy phase diagram compilations, allowing the feasibility of more economic and environmentally sound routes to be explored, better choices of materials to be made and pilot plant studies to be directed more efficiently. Current applications include the selection of suitable fluxing practices in copper and nickel smelting, analysing the operation of the Kennecott copper smelter, optimising the recovery of precious metals and investigating refractory wear.

4:45 PM
Minor Element Distribution between Copper Matte and Reverb Furnace Slag: *Natasa Mitevska*[1]; Zivan D. Zivkovic[2]; [1]Copper Institute, Zeleni Bulevar 35, Bor 19120 Yugoslavia; [2]Belgrade University, Tech. Fac., Bor 19210 Yugoslavia

The control of minor element behaviour during pyrometallurgical processing of copper concentrate is very important for anode copper production as well as influencing the production economics and minimizing environmental impact. The transfer of minor elements from the matte to the slag can be considered as either impurity elimination or loss of valuable components into the slag. On the basis of chemical analyses of samples taken during stable operation of the No.2 reverb furnace in the Copper Smelter and Refinery, RTB BOR (Yugoslavia), the distribution coefficients for Ag, As, Au, Bi, Co, Cu, Ni, Pb, Sb, Se, Te, and Zn between copper matte and slag are determined. The influence of the matte grade on the minor element distribution coefficients between copper matte and slag is also analyzed.

5:10 PM Cancelled
Simulation of Combustion in Copper Flash Smelter: *Xin-feng Li*

Notes

Aluminum Reduction Technology: Magnetics and Modelling

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

Thursday AM — Room: 6B
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Marc Dupuis, GeniSim, Jonquiere, Quebec G7S2M9 Canada

8:30 AM
Magnetohydrodynamic (MHD) Analysis of Anode Change and Lowering of Anode: A Useful Tool for Analyzing the State of an Operating Pot: *J. Antille*[1]; [1]Alcan Primary Metals Group Europe, CH 3965, Chippis Switzerland

A numerical simulation model solves the magnetohydrodynamic equations for the real geometries and bus bar arrangements of operating cells. This model predicts the natural oscillation frequencies of the cell observable in cell voltage and anode currents. The model calculates how an anode change and a lowered anode affect the natural frequencies. Measurements were made for a simulated anode change (simply isolating the anode), and for a real change (where material was deliberately allowed to sink and freeze to the cathode). The fluctuations of cell voltage and anode currents predicted were in good agreement with those measured in both cases, the frequency spectrum of the anode current indicating the presence or absence of bottom crust. Thus, analyzing the variations of anode current and/or cell voltage during normal operation can help in improving pot control.

8:55 AM
Metal Pad Roll Instabilities: *N. Ligonesche*[1]; T. Lelievre[2]; J. F. Gerbeau[3]; C. Le Bris[2]; C. Vanvoren[1]; [1]Aluminium Pechiney Research Center LRF, BP 114, 73300 Saint Jean de Maurienne France; [2]CERMICS, Ecole Nationale des Ponts et Chaussees, 6 & 8 ave. Blaise Pascal Cite Descartes, Champs-sur-Marne, 77455, Marne La Vallee, Cedex France; [3]INRIA, Projet M3N-Rocquencourt, BP 105, F-78153 Le Chesnay, Cedex France

The magnetohydrodynamic (MHD) phenomena which govern the movements of fluids (cryolite and aluminium) in Hall Heroult cells are extremely complicated. Indeed, the hydrodynamic and magnetic equations are closely coupled, through the Lorentz force (F = J ^ B) and the induced currents [J = (E + u ^ B)]. One of the phenomena which is observed in industrial cells and which has been much investigated over the past few years is metal pad rolling. The aim of most of the theoretical and applied MHD studies of cell has been to understand, forecast and avoid this phenomenon. The originality of our approach consists in the choice of a variational formulation which is only based on the basic physical equations (Navier Stokes, Maxwell and Ohmís law) without any simplification or linearisation and in the way the coupling between the velocity and the magnetic field is naturally taken into account. Through our simulations, we have observed the phenomenon of metal pad rolling in a circular cell. Moreover, we have noticed that sufficiently small vertical field does not lead to instability, which shows that the nonlinear approach may usefully complement the linear one and slightly correct its conclusion in some situations.

9:20 AM
Modeling Power Modulation: *Marc Dupuis*[1]; [1]GeniSim, Inc., 3111 Alger St., Jonquiere QC G7S 2M9 Canada

With, the recent power shortage in the USA, aluminum smelters are getting strong incentive to reduce their power consumption during peak demand. This power modulation can be quite harmful to the cells if not done properly. Yet, some smelters in Brazil are now successfully managing power modulation on a routinely basis following a long and expensive learning curve. Nowadays however, efficient dynamic cell simulator can be used in order to accelerate this learning curve and reduce the risk involved in performing power modulation without enough background experience. In this paper, two dynamic models are applied to study power modulation: an ANSYS based 2D+ full cell slice thermo-electric model and a much faster ìlump parameter+î model.

9:45 AM
The Complex Mechanisms Inducing Anode Effects in Aluminium Electrolysis: *Helmut Vogt*[1]; J. Thonstad[2]; [1] TFH Berlin-University of Applied Sciences, Berlin D-13353 Germany;† [2]Norwegian University of Science and Technology, Dept. of Matls. Techn. and Electro.,7491Trondheim, Norway

The very fast increase in cell voltage manifesting the incipience of anode effects in industrial alumina reduction technology is the result of interactions of various processes. As evidenced by mathematical models, the decrese in wettability with decreasing alumina content, although shifting the current distribution to the side walls, raises the actual current density on the active area and affects mass transfer of reactant and product. The resulting voltage is accelerated with decreasing wettability. The anode effect occurs as the limiting current density on the wetted area is approached. The interpretation incorporates most, but not all, of the interpretations proposed during the past century.

10:10 AM Break

10:20 AM
Methodology of the Technico-Economical Analysis of the Potroom Activity: *O. O. Rodnov*[1]; P. V. Poliakov[1]; A. I. Berezin[1]; P. D. Stont[1]; [1]Scientific Technological Center, Light Metals, Post Box 14144, 95 Krasnoyarsky Rabochy St., Krasnoyarsk, 660025 Russia

It is necessary for smelters to have expert systems of analysis, which will permit to conduct deep evaluation of the technology of aluminum electrolysis with the purpose to determine the reasons, which provoke technology disturbances, to investigate and optimize operations. We offer methodology to analyze parameters of the potroom activity. This methodology establishes dependence between changes of the control actions and electrolysis parameters on one hand and amount of the produced aluminum, energy and raw materials consumption and economical figures on the other. Methodology includes 3 consecutive stages: 1. Preliminary preparation of the initial information (data base of the automatic system of management of the technological processes ASM TP), using classical statistical analysis as well as neural network technologies. This stage permit to evaluate the main dependences and interactions of the process. 2. Optimization of the process according to the technological criteria. We use the new method to built special simplex phase diagrams with criteria, which limits the area of optimization. Multi parametrical optimization with the purpose to investigate different regimes and to find out optimal control actions and parameters has been used. 3. Optimization according to the economical criteria including standard economical analysis where the results of the previous stage are used as input data for stage 3. The parameters of a real potroom have been analyzed. It was shown that decreasing or increasing target voltage resulted in aluminum production increase. Acceptable variant can be determined by economical analysis taking into account prices for energy, fluorides and other materials.

10:45 AM
Effect of Current Distribution on Current Efficiency in 160KA Prebake Cells: *Zeng Shuiping*[1]; Zhang Qiuping[1]; Ding Weian[2]; [1]North China University of Technology, Automation Inst., Beijing 100041 China; [2]Beijing General Institute of Nonferrous Metal, Beijing 100041 China

This paper investigates the relations between the CE and the current distribution in 160KA prebake cells. On the bases of the viewpoint of zone current efficiency, a mathematic model to describe their relationship was established. And by use of cross design method, we simplified the relation and got a algebraic equation, which is the foundation to analyze the effect of current distribution on CE. Because both the current distribution and the current efficiency are uneven, the change of current distribution causes the change of CE, but not the strict even distribution gives the best CE, which is perhaps caused by uneven magnetic distribution. The paper also gives some suggestions to improve the cell operation and raise the CE.

11:10 AM
Study on Temperature Field and Incrustation of 160 KA Prebake Cells in the Guizhou Aluminum Smelter: Feng Naixiang[1]; Sun Yang[1]; *Li Hongpeng*[2]; Leng Zhengxu[2]; Feng Shaozhong[2]; [1]Academy of Materials and Metallurgy, Northeast University, Shenyang 11006 China; [2]Guizhou Aluminum Smelter, Guiyang 550014 China

According to the measured results of the cell side temperature, the temperature field in the 160kA prebake anode reduction cell and the coefficient of heat transfer between the incrustation and electrolyte melt, liquid aluminum were calculated by using finite element method and thermal flow pipe. From the calculated results, it can be seen that the coefficient of heat transfer between the incrustation and electrolyte melt, liquid aluminum is major. It was perhaps caused by unreasonable bus design, which can make the melt flow fast. In the present paper, the effects of using different inner lining materials and different cell voltage on the thickness of the incrustation, molecular proportion of electrolyte melt and current efficiency were calculated.

11:35 AM
Finite Element Analysis of Magnetohydrodynamics Stability of an Aluminium Reduction Cell: *Wu Jiankang*[1]; Huang Ming[1]; Huang Jun[2]; Yao Shihuang[2]; [1]Huazhong University of Science & Technology, Mech. Dept., Wuhan China 430; [2]Guiyang Aluminum-Research Institute, Guiyang 550004 China

This paper employed finite element method to solve eigenvalues of 2D shallow-water perturbed magneto-hydrodynamic (MHD) system for stability analysis of an aluminum reduction cell. Stability analysis of 230 KA reduction cell was carried out in this paper. A series of frequency response figures of a reduction cell, which give disturbance growth rate and corresponding frequencies, was presented. The numerical results indicate that the presence of magnetic field always induces instability of a few long-wave modes of interface oscillations of electrolyte bath and aluminum liquid. meanwhile, the short-wave modes are stable. It is found that the growth rates of unstable modes strongly depend on current density, vertical magnetic field, mass density differences of electrolyte and molten aluminum, and liquid thickness of bath and molten metal. The numerical calculations confirm that the stability of reduction cell can be improved by increasing anode-cathode distance (ACD) and thickness of aluminum liquid.

Aluminum Sheet and Plate Rolling & Finishing Technology and Applications: Aluminum Sheet and Plate Applications

Sponsored by: Light Metals Division, Aluminum Association, *Program Organizers:* Michael H. Skillingberg, The Aluminum Association, Inc., Washington, DC 20006 USA; John P. Brandimarte, Precision Coil, Inc., Clarksburg, WV 26302-2650 USA; Karl Eminger, Alcoa, Inc., Alcoa Mill Products, Lancaster, PA 17604 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Jeff J. Kadilak, Alcan, Inc., Oswego, NY 13126 USA; Leland R. Lorentzen, Nichols Aluminum, Davenport, IA 52801 USA; Douglas N. McLeod, ARCO Aluminum, Inc., Louisville, KY 40242 USA; Ken Schreckengast, Coastal Aluminum Rolling Mills, Inc., Williamsport, PA 17701 USA

Thursday AM — Room: 608
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Douglas N. McLeod, ARCO Aluminum, Inc., 9960 Corporate Campus Dr., Ste. 3000, Louisville, KY 40242 USA; Michael H. Skillingberg, The Aluminum Association, Inc., 900 19th St. N.W., Washington, DC 20006 USA

8:30 AM Invited
Aluminum Sheet for Automotive Body Applications-Challenges and Opportunities: *Andrew M. Sherman*[1]; [1]Ford Motor Company, 20000 Rotunda Dr., MD3182, Rm. 2160, SRL, Dearborn, MI 48121 USA

Over the past 25 years, motivated by the need to reduce vehicle weight in order to increase fuel economy, the use of aluminum in production automobiles has increased by more than 150%. Current average usage is about 250 pounds per vehicle; most of this, ca. 200 pounds, is in the form of castings. Applications of sheet include heat exchangers, heat shields, trim and a limited, but growing number of body panels (hoods, deck lids, fenders). While casting applications will continue to increase, by far the largest potential for growth in the use of aluminum is for body structures and closure panels. It has been shown that both closure panels and body structures can be designed to meet all vehicle requirements and that aluminum components can be manufactured in high-volume at rates the same as steel. However, a number of limitations remain: both material cost and manufacturing cost are higher, as are lead-time and investment. In addition, the infrastructure to support widespread application of aluminum bodies needs to be developed. This talk will outline some of the technical developments necessary to address these issues and discuss the elements of a long-range strategy for the cost-effective application of sheet aluminum in vehicles.

9:00 AM
The Global Market for Foil: *Kelly J. Driscoll*[1]; [1]CRU International, Aluminium Grp., 31 Mount Pleasant, London WC1X 0AD UK

Foil is one of the principal end-uses for aluminium sheet. It has shown impressive growth rates in most developed world economies in recent yearsñbut can that continue? In this paper we investigate historical trends and future growth prospects for foil production and consumption, analyse the threats to the foil market from substitution and the impact of recycling and legislation, and analyse the threats and opportunities posed by product innovation and down-gauging. We identify the major opportunities for growth, innovation, and new technologies.

9:30 AM
Opportunities for the Use of Aluminum in the Residential and Commercial Construction Markets: *Richard Bus*[1]; [1]ATAS International, Inc., 6612 Snowdrift Rd., Allentown, PA 18106 USA

Abstract Unavailable

10:00 AM Break

10:15 AM
Aluminum Applications in the Automotive Industry: *Richard L. Klimisch*[1]; Joseph C. Benedyk[3]; Michael H. Skillingberg[2]; [1]The Aluminum Association, Inc., Detroit Office, One Towne Sq., Ste. 230, Southfield, MI 48076 USA; [2]The Aluminum Association, Inc., 900 19th St. N.W., Ste. 300, Washington, DC 20006 USA; [3]98 Schiller St., Lake Zurich, IL 60047 USA

An overview of recent developments and trends in the use of aluminum in the worldwide automotive industry is presented. Applications for various vehicle systems are discussed including those for the powertrain, structure, body and chassis. Recent developments in the production and assemby of aluminum automotive components, including the areas of forming and joining, are reviewed. The implications of the rapid growth of aluminum in automobiles is discussed as related to environmental sustainability, safety and vehicle performance. Aluminumís role in improving automotive fuel efficiency is addressed in light of the current heightened awareness of energy consumption.

10:45 AM
Enabling Communications about Aluminum Mill Products via Industry Standards: *Peter Pollak*[1]; *Parvaneh Shafiee*[1]; [1]The Aluminum Association, Inc., Techl., 900 19th St., N.W., Ste. 300, Washington, DC 20006 USA

Aluminum industry standards cover quantifiable attributes of aluminum mill products. They provide the basis of a common language for commerce and are an indispensable tool for communicating about aluminum alloys, tempers and mill products. The Aluminum Association promulgates the basic alloy and temper designation systems, which go through the American National Standards Instituteís procedures for recognition as American National Standards. Even though there are several designation systems for different aluminum products (wrought, cast, primary, hardeners, etc), this paper focuses on standards for wrought mill products, and describes how the basic information is registered and is subsequently used in other codes and standards. Finally it covers the worldwide harmonization of aluminum industry standards, and how this is facilitated through international accords between the Aluminum Association, and its foreign counterparts.

11:15 AM
Studies of Chemical and Physical Emissions from Welding Aluminum Alloys: *Seymour G. Epstein*[1]; [1]The Aluminum Association, Inc., 900 19th St. N.W., Washington, DC 20006 USA

Fabrication and repair of aluminum components and structures commonly involves the use of electric arc welding. In order to ensure the safety of the welders and others in the local environment, it is important to understand the nature of the chemical and physical emissions that are inherent to this joining process. Since aluminum is seldom used as the pure metal but is far more often alloyed with other metals to improve strength and various physical properties, the interaction of the arc and the metal being welded generates ultraviolet radiation, various metallica oxides and fumes, and gases. The exact composition of these emissions depends on the aluminum alloy(s) being welded. In order to provide needed data on welding emissions, The Aluminum Association sponsored several studies to characterize the emissions from arc welding by the metal inert gas (MIG) and tungsten inert gas (TIG) processes for various combinations of base and filler alloys. In some of the studies emissions from other processes, such as grinding and plasma arc cutting, were also investigated. In all cases the tests were conducted to provide a means of estimating the ìworst caseî exposures.

Cast Shop Technology: Casting

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

Thursday AM — Room: 6A
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Christian Pluchon, Pechiney Group, Center Alp, BP 27, Voreppe 38341 France; Gary B. Parker, Wise Alloys LLC, Alabama Reclamation Ops., 1009 Ford Rd., Muscle Shoals, AL 35661-1119 USA

8:30 AM Keynote
D.C. Casting of Aluminium Alloys-Past, Presence, Future: *Wolfgang A. Schneider*[1]; [1]VAW aluminium AG, R&D, Georg v. Boeselager Str. 25, Bonn 53117 Germany

The principles of D.C. casting of aluminium alloys were invented in 1936. Due to the importance of this process to the industry for the fabrication of semi-finished products intensive development work has taken place over the last 30 years. Increasing demands on ingot quality necessitated the development of innovative casting technologies. This has led, for example, to the introduction of air-assisted hot top moulds for the casting of extrusion ingots and electro-magnetic moulds for the casting of rolling ingots. Further techniques and tools were also developed to optimize the use of modern mould techniques and to increase the safety and reliability of the D.C. casting process. A review will be given of the history of D.C. casting and the state of the art of the process concerning casting technologies and ingot quality. Finally, future development trends and challenges will be discussed.

9:00 AM
The Manufacturing, Design and Use of a New Reusable Molten Metal Distributor for Sheet Ingot Casting: *Sylvain P. Tremblay*[1]; *Martin Lapointe*[2]; [1]Pyrotek High Temperature Industrial Products, Inc., 1623 Manic St., Chicoutimi, Quebec G7K 1G8 Canada; [2]Aluminerie de Becancour, Inc., 5555, Pierre-Thibault St., Ville de Becancour, Quebec G9H 2T7 Canada

In the last decade, the molten aluminum distribution in the ingot head of DC sheet casting ingots has been achieved using mostly a combo bag made of fiberglass fabrics. Some of these fabrics are open-weave materials while others are solid fiberglass fabrics sewn together. This bag can deform and this can affect not only the distribution but also the molten metal temperature profile around the mold and at the end, the final ingot quality. This paper will review the use of a new distributor for DC sheet ingot casting. The paper is divided in two parts. The first part will deal with the principle and design of the new resusable molten aluminum distributor (ReMAD) to replace the standard combo bag. Description of the ReMAD materials used, presentation of water modeling and mathematical modeling experiments will complete the first part. Part two will present the test results of the ReMAD used at Aluminerie de Becancour Inc. (ABI) to cast AA-1045, AA-3003 and AA-5052. The following variables and measures will be discussed: Cast start-up, actual metal flow observations, ingot surface finish, temperature profile around the mold, ingot cut slice analyses, Podfa analyses and finally, results from the rolling plant. Comments from an operation point of view on the use of the reusable molten aluminum distributor will complete this presentation.

9:25 AM
On the Mechanism of Surface Cracking in DC Cast 7XXX and 6XXX Extrusion Ingot Alloys: *Steinar Benum*[1]; Hallvard Fjaer[2]; Oddvin Reiso[1]; Dag Mortensen[2]; Hilde Gunn Overlie[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

When applying the Hydro variant of the Show Denko gas slip technology for casting of 7xxx alloys surface cracks occurred and gave a significant scrap rate in the cast house. Especially caused one alloy with 0.3 wt.% Cu problems. In order to identify the problem the casting process for this alloys were simulated by a coupled stress, thermal and fluid flow model (ALSIM/ALSPEN). The simulations were designed as a factorial trial where casting speed, ramping of the speed, casting temperature, cone height of the starting block, cooling water efficiency and primary cooling were varied systematically. The hoop stress in the surface at the temperature when 95% of the material was solidified was used as a crack sensitivity indicator. Three stages were found: (I) At the start a maximum hoop stress evolved, (II) then a minimum stress occurred (III) before the stress reached a stable level. For an AA6xxx alloy the stress was found to be zero in the stable stage while the 7xxx alloy had tension stresses during the stable stage. Based on the factorial analysis it was found that the stable stress increased with increasing casting speed and decreased with an increased primary cooling. Furthermore, the initiation and propagation of cracks are discussed based on investigations on real cracks. Here it was found that cracks tended to initiate when oxide lumps were released from the hot top.

9:50 AM
Nature and Formation of Surface Cracks in DC Cast Ingots: *Qingyou Han*[1]; Srinath Viswanathan[1]; Douglas Spainhower[2]; Subodh K. Das[3]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [2]Logan Aluminum, Inc., PO Box 3000, Russelville, KY 42276 USA; [3]Secat, Inc., 1505 Bull Lea Blvd., Lexington, KY 40511 USA

Surface cracks are critical defects that increase the cost and reduce the yield of the DC casting process. However, there is some debate about the nature of surface cracks in the literature. In this study, surface cracks on a 3004 alloy ingot were examined in detail. Optical microscopy showed that the crack occurred along grain boundaries. Dendrites were clearly seen on the fracture surface using Scanning Electron Microscopy. Auger analysis indicated that silicon and magnesium were highly segregated on the fracture surface. When coupled with a simulation of the solidification of 3004 alloy, the results suggest that the surface cracks that occur during DC casting of aluminum alloys are hot tears that form above the solidus temperature, rather than cold cracks that form below the solidus temperature.

10:15 AM Break

10:30 AM
Experimental Study of the Heat Transfer along the Surface of a Water-Film Cooled Ingot: *Laszlo Istvan Kiss*[1]; Thomas Meenken[1]; Andre Charette[1]; Yves Lefebvre[2]; Robert Levesque[3]; [1]Universite du Quebec a Chicoutimi, Dept. des Sciences Appliquees, 555 blvd. de líUniversite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Betz-Dearborn Canada, Inc., 3451 Erindale Station Rd., Mississauga, Ontario L5A 3T5 Canada; [3]Alcoa Baie-Comeau, 1001 Rt. Maritime, Baie-Comeau, Quebec G4Z 2L6 Canada

In the recent years many research efforts have been directed toward the determination of the heat transfer coefficients between the solidifying ingot and cooling water during the semi-continuous casting of aluminum ingots. The present study aims to the analysis of the influence of water quality and ingot properties on the cooling capacity. An experimental technique was developed to follow closely the variations of the surface heat flux without modifying the surface properties of the solid. The results are presented in the form of surface heat flux vs. surface temperature and heat transfer coefficient vs. temperature diagrams. The correlation between the different definitions of local and average heat flux coefficients is discussed.

10:55 AM Cancelled
The Formation of Surface Segregates during Twin Roll Casting of Aluminium Alloys: *B□rge Forbord*

11:20 AM
Thixoforming Raw Material Development by Means of Optimized Design of Experiments (DoE): *T. Noll*[1]; B. Friedrich[1]; [1]IME-Metallurgische Prozesstechnik und Metallrecycling, RWTH Aachen, Intzestrafle 3, 52056 Aachen Germany

Thixocasting and Forging belongs to modern forming processes. The process is a soft forming process in which semi solid slurries with a stiffness of ìbutterî can be formed under low pressure and temperature substantially lower than those used for casting. Thixoforming gains an increasing interest. The automobile industryñfor exampleñ looks for process technology which connect complex parts with improved mechanical properties. Potential of energy- and costs-saving is not to be neglected. Although this process was already developed in 1972 by MIT (Massachusetts Institute of Technology, Cambridge) the required improvement of process stability and the introduction of suitable quality management systems must be improved. Progress in process stability should start with raw material development. The first part of this paper is a stock taking and gives an introduction into the Thixoforming process and its typical process steps. The most important technologies of raw material preparation will be presented, too.

In the second part of this paper raw material development by chemical grain refinement and determination of suitable DC-casting process parameters by means of optimized design of experiments will be presented.

11:45 AM
Automatic Bleedout Detection System Leading Safety Development: *Todd Snyder*[1]; Andrew J. Widrig[1]; *Helmut Suppan*[2]; [1]Wagstaff, Inc., 3910 N. Flora Rd., Spokane, WA 99216 USA; [2]Aluminium Ranshofen Huttengiesserei Ges.m.b.H., Postfach 35, Lambrechtshausner Strasse, Braunau, Ranshofen A-5282 Austria

As total production dynamics in the casthouse changes, principally to higher capacity equipment with reduced exposed man hours per ton produced, Wagstaff, Inc., has successfully introduced the StopCast(TM) Automatic Bleedout Detection System and plug off mechanism to the industry. This paper will outline the operational details of the StopCast system and share experiences from our first installation at Aluminium Ranshofen H¸ttengiesserei Ges.m.b.H.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: Design of New Materials

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

Thursday AM Room: 307-308
February 21, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: John Knott, University of Sheffield England; David Embury, McMaster University, Matls. Sci. & Eng., 1280 Main St., W., Hamilton, Ontario L8S 4L7 Canada

8:30 AM Invited
The Electrochemical Fatigue Sensor-Development Update: Y. F. Li[1]; J. Wang[1]; M. Wang[1]; A. Witney[1]; J. DeLuccia[1]; *Campbell Laird*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

The Electrochemical Fatigue Sensor (EFS) has undergone considerable development for commercial purposes in the last three years, and advances have been made in both understanding its mechanism and in applying it. The present understanding by which the sensor current responds to mechanical stress and fatigue damage is described and an up-to-date description of the developed device is presented. Since the EFS gives real time information about the state of damage in a specimen undergoing fatigue, it offers considerable advantage as a laboratory tool for understanding fatigue mechanisms. Examples of its use in studying small crack growth behavior and in cumulative damage under variable loading are provided.

9:05 AM Invited
Advanced Metal Science Based on Nano-Metallurgy: *Kenji Abiko*[1]; [1]Tohoku University, Inst. for Matls. Rsrch., 2-1-1 Katahira, Aoba, Sendai, Miyagi 980-8577 Japan

We have carried out the fundamental research of ultra-high purity metals which were melted by a newly designed induction-melting furnace with a cold copper crucible using ultra-high vacuum technology. The purity of Iron melted is analyzed to be higher than 99.9989 mass%. The ultra-purified iron possesses the incredible properties such as mechanical properties, deformation behavior, corrosion resistance and so on. These experimental results can not be predicted from the conventional metallurgy. It is concluded that the ìNano-Metallurgyî which is the metallurgy based on the ultra-purification and the microstructure control of metals in nano-scale is effective to find the inherent properties of metals.

9:40 AM Invited
Grain Boundary Engineering for Alleviating Weld Sensitization and Stress Corrosion Cracking in Nickel-Based Alloys: *Gino Palumbo*[1]; [1]Integran Technologies, Inc., 1 Meridian Rd., Toronto, Ontario M9W 4Z6 Canada

Advances in the modeling of microstructural evolution and in material characterization techniques (e.g., automated electron diffraction) have led to the emergence of innovative and cost-effective thermomechanical processing methods for the control and optimization of grain boundary structures in conventional polycrystalline materials; these processing methods being designed to yield increased populations of structurally-ordered high angle grain boundaries in the microstructure. In this presentation, an overview of the theoretical basis and the practical application of grain boundary engineering concepts to the design and development of high performance Nickel-based alloys (e.g., 600, 625, 690, 738, 800, C-22 etc.) will be presented and discussed. The grain boundary engineering approach is shown to enhance the resistance of these alloys to weld sensitization, intergranular corrosion and stress corrosion cracking, and can be used to improve component reliability and extend service life in numerous applications (e.g., nuclear plant components, nuclear waste storage, recovery boilers, combustion turbines etc.)

10:15 AM Invited
Materials by Design: Quantum Steel: *Gregory B. Olson*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

A systems approach integrates process/structure/property/performance relations in the computational design of materials as multilevel dynamic structures. For the case of ultrahigh-strength martensitic steels, quantum mechanical total energy calculations using the FLAPW method address the thermodynamic and electronic basis of segregation effects on grain boundary cohesion identified by McMahon and coworkers. Model predictions of cohesion enhancing components are integrated with a set of microstructural design models to design a first generation of Quantum Steels exploiting quantum mechanical predictions to enhance resistance to intergranular hydrogen stress corrosion cracking. Research performed in collaboration with W-T. Geng, A.J. Freeman and C. Kantner under ONR sponsorship.

10:50 AM
Grain-Boundary Segregation of Trace Elements in Iridium Alloys and Effects on Mechanical Properties: *E. P. George*[1]; L. Heatherly[1]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6093 USA

Iridium is attractive for high-temperature structural applications because of its high melting point and good oxidation/corrosion resistance. A major drawback is its low tensile ductility when tested at conventional strain rates at low temperature, and at high strain rates at elevated temperature. Microalloying has been used to strengthen the grain boundaries and suppress brittle intergranular fracture in Ir alloys. Auger studies indicate that Th and Ce have a strong tendency to segregate to Ir grain boundaries and suppress intergranular fracture at elevated temperatures. We review here the properties of the Th-doped alloys that have been successfully developed for space power applications at temperatures to 1400∞C as well as the Ce-doped alloys that are currently under development for future applications. We will also discuss the grain-boundary segregation of Si and its role in the severe embrittlement of iridium alloys. Research sponsored by the Office of Space and Defense Power Systems and the Division of Materials Science and Engineering, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:10 AM
Failure Mechanisms of Thermal Barrier Coatings-Effect of Alloying Elements and Impurities: *Jeff Pfaendtner*[1]; Irene Spitsberg[1]; [1]GE Aircraft Engines, Matls. & Proc. Eng. Dept., One Neumann Way, MD M89, Cincinnati, OH 45215 USA

The chemical and thermo-mechanical factors leading to spallation of EB-PVD Thermal Barrier Coatings (TBC) from platinum-aluminide-coated nickel-superalloy substrates are discussed. Reactive element and refractory element diffusion into the bond coat from the substrate during cyclic high-temperature exposure are shown to have a strong effect on TBC spallation life. This effect can result in a significant increase in TBC life depending on the superalloy used as substrate. The presence of interstitial impurities in the bond coat has been shown to counteract the effectiveness of these substrate elements. The effect of these diffused elements on the major components of the mechanism of TBC failure, e.g. growth rate and adhesion of the thermally growth oxide (TGO), and ratcheting of the TGO/bond coat interface, will be discussed.

11:30 AM
Evolution of Grain Boundary Planes in Grain Boundary Engineering: *Christopher Schuh*[1]; Mukul Kumar[1]; Wayne E. King[1]; Lan Nguyen[1]; [1]Lawrence Livermore National Laboratory, Matls. Sci. & Tech., 7000 East Ave., L-350, Livermore, CA 94550 USA

The process of grain boundary engineering, whereby the fraction of ìspecialî grain boundaries is increased through sequential thermomechanical processing, gives rise to considerable improvements in, e.g., intergranular cracking resistance, intergranular corrosion resistance, creep strength, and ductility of low to medium stacking fault energy FCC metals. The special properties of such grain boundaries are usually correlated only with the relative misorientation across the boundary, and neglect the plane of the boundary with respect to the crystallographic axes of the two grains. In the present work, we describe experiments on a nominal Ni-Cr-Fe solid solution alloy in which both of these geometrical parameters are measured using electron backscatter diffraction orientation mapping on serial sections. Specifically, we explore the effect of thermomechanical processing on the evolution of both the misorientation and the plane of grain boundaries. The results are discussed in the context of the crystallographic constraint imposed by the triple junctions and their connectivity.

Computational Phase Transformations: Computational Materials Design

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

Thursday AM — Room: 201
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Zikui K. Liu, Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM
Transformation Modeling in Computational Materials Design: *Greg B. Olson*[1]; H.-J. Jou[2]; [1]Northwestern University, Dept. Matl. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]QuesTek Innovations, LLC, 1820 Ridge Ave., Evanston, IL 60201 USA

A range of dynamic models based on the Thermocalc/DICTRA system is applied in the computational design of materials as dynamic multilevel structures. For the case of complex multicomponent martensitic steels, a kinetic model predicting Ms temperatures has been extended by G. Ghosh to predict the full evolution of lath martensite microstructures controlling retained austenite content. A model of bainitic transformation as a coupled diffusional/displacive mechanism predicts bainite-start C-curves for control of hardenability. The new PrecipiCalc software for simulation of precipitation reactions based on the Langer-Schwartz framework is being applied to the simulation of precipitation hardening of Ni-base aeroturbine disc alloys under an initiative aimed at acceleration of the full materials development and qualification cycle.

9:15 AM
Calphad and the Phase-Field Method: *John ≈gren*[1]; [1]KTH, Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

The phase-field method has been tremendously successful in generating realistic micro structures, e.g. dendrites during solidification and different variants of martensite. It is possible to take various factors like stresses and crystallographic anisotropy into account. However, so far mostly very simple and not very realistic systems have been treated, e.g. heat flow in pure substances, isothermal diffusion in binary systems etc. Unfortunately, the materials properties have been rather far from those of real multi-component alloys. The phase-field community has thus not made much use of the recent advances in thermodynamic modelling and high-quality databases presented within the Calphad community. Nevertheless, the governing equations of the phase field-method may be derived from the Gibbs energy of entropy functions given by the Calphad method. The most severe difficulty when coupling thermodynamic softwares to phase-field computations is the computational speed. In this presentation the basic theory will be reviewed and some recent applications will be discussed. Different ways of increasing the computational speed will be discussed.

9:45 AM
Linking Phase-Field Model with CALPHAD: Application to Morphological Evolution of Gamma-Prime Precipitates in Ni-Base Alloys: *Jingzhi Zhu*[1]; V. Vaithyanathan[1]; Z. K. Liu[1]; L. Q. Chen[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

This presentation reports our preliminary results of a multi-year effort in developing a set of computational tools for modeling the thermodynamics, microstructure evolution, and mechanical properties of Ni-base superalloys. In particular, we describe a phase-field model which has the following unique features: (1) it uses a local free energy as a function of composition and order parameters directly constructed using the semi-empirical CALPHAD method; (2) it employs an efficient iterative method for solving the elastic equations for elastic inhomogeneous systems; (3) it takes into account the composition and order parameter dependence of atom mobilities; and (4) the kinetic equations are solved using an efficient semi-implicit Fourier-spectral method. With all the important thermodynamic and kinetic parameters either from a database or from independent experimental measurements, the three-dimensional morphology of gamma-prime (Ni_3Al) precipitates in a disordered fcc matrix is studied as a function of precipitate size. Results from the simulation are compared with existing experimental observations in Ni-base alloys. The work is supported by NASA.

10:05 AM Break

10:15 AM
Development of Multicomponent Diffusion Mobility Databases: *C. E. Campbell*[1]; [1]NIST, Metall., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

As the demand for improved materials performance and processing efficiency increases, a better understanding multicomponent multiphase diffusion has become essential. One of the first steps in this process is the assembling of diffusion mobility databases, which can be used with thermodynamic databases to calculate composition-dependent diffusivities. The mobility databases are constructed using a CALPHAD approach that assumes that quaternary and higher order interactions can be neglected allowing binary and ternary assessments to be combined to extrapolate to higher order systems. Construction of a Ni-based diffusion mobility database is used to demonstrate the database development process, which includes the analysis of available diffusion data, the assessment of diffusion activation energies in metastable states, and the validation of the extrapolation to higher order systems. Finally, comparison of measured and simulated composition profiles from a diffusion couple of two commercial superalloys allows the non-monotonic diffusion behavior predicted by the composition-dependent diffusivities to be evaluated.

10:45 AM
Some Attempts of Hybridized Calculation of Phase-Field Method and Cluster Variation Method: M. Ohno[1]; *T. Mohri*[1]; [1]Hokkaido University, Div. of Matls. Sci. & Eng., Grad. Sch. of Eng., Sappro 060-8628 Japan

In order to gain both atomistic and microstructural evolution processes, a hybridized calculation of Cluster Variation Method and Phase-Field Method is attempted. Particular focus is placed on disorder-B2 and disorder-L10 transitions. In addition to microstructural evolution process, atomistic evolution process is visualized based on the time evolutions of cluster correlation functions obtained by CVM. The distinctive feature of existence(L10) and non-existence(B2) of triple point at Anti Phase Boundary is confirmed. Furthermore, the relaxation behavior of Long Range Order parameter is critically examined and is compared with the one obtained by Path Probability Method.

11:05 AM
Microstructure Simulations of a 5-Component Ni-Base Model Alloy: *Nils Warnken*[1]; Bernd Boettger[1]; Dexin Ma[1]; Victor Vitusevych[1]; Nathalie Dupin[2]; Suzana Gomes Fries[1]; [1]ACCESS e.V., RWTH-Aachen, Intzestr. 5, Aachen D-52072 Germany; [2]Calcul Thermodynamique, 3, rue de líavenir, Orcet F-63670 France

In order to investigate solidification microstructure and microsegregation in Ni base superalloys, a model alloy containing the five elements Ni, Cr, Al, Ta and W was designed and produced. Directional solidified samples were prepared in a Bridgman type furnace to provide the benchmark data for the simulations. The simulations are done using the ACCESS Multiphase, Multicomponent Phase Field Model, coupled to a thermodynamic database. The same model is also applied to simulate the homogenization of the cast structure during heat treatment. The simulation results show reasonable agreement with the solidification experimental data for the formation of the primary (A1) and secondary phases (L12). Equilibrium experiments are being performed in order to fine tune the thermodynamic database. This work has been done within the Collaborative Research Center 370 ìIntegrated Modeling of Materialsî of the Deutsch Forschungsgemeinschaft (DFG).

11:25 AM
A Pseudo-Front Tracking Technique for the Modelling of Solidification Microstructures in Multi-Component Alloys: *Alain Jacot*[1]; Michel Rappaz[1]; [1]Ecole Polytechnique FÈdÈrale de Lausanne, LMPH, EPFL, MX-G, Lausanne 1015 Switzerland

A two-dimensional model for the simulation of microstructure formation during solidification in multi-component systems has been developed. The model is based on an new pseudo-front tracking technique to describe the evolution of interfaces that are governed by solute diffusion and Gibbs-Thomson effect. The diffusion equations are solved in the primary solid phase and in the liquid using an explicit finite volume method formulated for a regular hexagonal grid. The interface curvature is obtained from the field of the signed distance to the interface and a PLIC (Piecewise Linear Interface Calculation) technique, which allows to reconstruct the position of the interface in these mushy volume elements. The concentrations at the solid-liquid interface are calculated using thermodynamic data provided by the phase diagram software Thermo-Calc. Different coupling strategies between the microstructure model and Thermo-Calc have been developed, in particular a computationally-efficient direct coupling using the TQ-interface of Thermo-Calc. After testing the accuracy of the model with respect to solute and curvature calculation, comparisons are made with predictions obtained with the marginal stability theory, a 1D front-tracking method and 2D phase-field simulations of dendritic growth in binary alloys. The model is then used to describe the formation of several grains in an Al-1%Mg-1%Si alloy, as a function of the heat extraction rate and inoculation conditions. It is shown that the model is capable of reproducing the transition between globular and dendritic morphologies.

11:45 AM
High-Speed Thermodynamic Calculations for Kinetic Simulations: *Henrik Strandlund*[1]; [1]Royal Institute of Technolgy, Div. of Physl. Metall./Dept. Matl. Sci. & Eng., Brinellvagen 23, Stockholm 100 44 Sweden

Simulation of phase transformations may be divided into two parts; the simulation of kinetics and the calculation of thermodynamic quantities. A number of softwares for thermodynamic calculations are available and it is thus convenient to use such software to obtain an accurate description of thermodynamic properties and couple them to a software simulating the kinetics. A major problem then is that the computational work to evaluate the thermodynamic quantities is too heavy unless very simplified thermodynamic models are used. That problem may be solved by calculating the thermodynamic quantities in selected points and time steps only and apply an artificial neural network to obtain the values in all other points. This approach has successfully been applied to diffusion in alloys and examples will be given showing how this approach enables us to incorporate thermodynamic data from Thermo-Calc into simulations of diffusion in a very efficient way.

Deformation and Stresses in Small Volumes: Fatigue and Fracture of Small Volumes

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

Thursday AM — Room: 303
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Gene A. Lucadamo, Sandia National Laboratories, PO Box 969, MS 9161, Livermore, CA 94551-0969 USA

8:30 AM Invited
On the Mechanism of High-Cycle Fatigue in Si Thin Films for MEMS Devices: C. L. Muhlstein[1]; E. A. Stach[2]; *R. O. Ritchie*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA 94720 USA

Fracture and fatigue resistance of brittle, silicon-based, structural films are important properties for the design and long-term durability of micromechanical components, especially MEMS devices. Recent work has established that such thin, micron-scale, films of single- and ploy-crystalline silicon are susceptible to premature failure under cyclic loading conditions, with failure occurring at long lives at approximately one half of the single-cycle fracture strength. However, the mechanism(s) of such failures have been a mystery. In this presentation, we propose a mechanism for the fatigue of thin-film LPCVD polysilicon based on extensive stress-life fatigue testing and high voltage transmission electron microscopy. It is proposed that the fatigue process is ìsurface dominatedî and involves the mechanically-induced thickening of the oxide film, followed by environmentally-assisted subcritical cracking of the thickened film. Indeed, procedures, e.g., monolayer coatings, to exclude moisture and oxygen from the silicon surface are an effective means to suppress the susceptibility to fatigue failure in these materials.

9:00 AM
The Effects of Cyclic Loading on the Deformation and Fracture of Metal-Oxide Multilayers using Nanoindentation: *Christy L. Woodcock*[1]; David F. Bahr[1]; Neville R. Moody[2]; [1]Washington State University, Mechl. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA; [2]Sandia National Laboratories, Livermore, CA 94551-0969 USA

Thin metal and oxide films play an important part in the rapidly expanding microelectronics industry. In particular, the system of films with a ìhard-soft-hardî gradient is vital to new microelectromechanical systems (MEMS). In this study, systems of TiO2/Ti/Si, and Al2O3/Al/SiO2 were chosen as models for two film/substrate systems. Properties of these films are measured using nanoindentation methods with both an unloading analysis and the continuous stiffness technique. The continuous stiffness technique generates a ìpop inî event during loading which is not present in monotonically loaded samples. The constant displacement oscillation value is shown to have a large impact on the frequency and peak load of these oxide excursions. Controlling the amplitude of cyclic loading from between 3 nm to 9 nm generates pop in events at decreasing loads as the amplitude is increased. Atomic force microscopy is used to show this does not appear to cause through thickness film fracture events. The possibility of interfacial failure and substrate cracking are discussed in light of the lack of exposed cracks on the surface. In addition, a finite element analysis (FEA) is used in conjunction with nanoindentation to model the depth dependent properties in materials that exhibit this same hardness gradient.

9:20 AM
Fracture of Electrodeposited Multilayered Ni/Cu Composites: *Fereshteh Ebrahimi*[1]; Alirio J. Liscano[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

Thick (20 to 40 micrometer) films of nanolayered Ni/Cu composites were produced using the electrodeposition technique. The strength and fracture behavior of free-standing films were evaluated by testing dog-bone shaped samples in tension. X-ray diffraction, transmission electron microscopy and scanning electron microscopy were employed to characterize the growth mechanism and microstructure of the multilayers. The results of this study suggest that the fracture behavior, and hence the strength, of nanolayered Ni/Cu composites is influenced significantly by the microstructure. Compatible deformation of the nickel and copper layers in samples with flat layers parallel to {100} planes resulted in knife-edge type fracture behavior. Incompatible deformation in samples with incoherent Ni/Cu interfaces parallel to {100} planes led to delamination and brittle fracture. Twining caused zig-zag growth of non-coherent layers which also resulted in constrained plastic deformation and brittle-like fracture. Development of pores during the deposition process significantly reduced the strength of the deposits.

9:40 AM
AE Monitoring of Contact Induced Plasticity and Fracture in Ultra-Small Volumes: *Natalia Tymiak*[1]; Antanas Daugela[1]; Thomas Wyrobek[1]; William W. Gerberich[2]; [1]Hysitron, Inc., 5251 W. 73rd St., Minneapolis, MN 55439 USA; [2]University of Minnesota, CEMS, 421 Washington Ave. S.E., Minneapolis, MN USA

Present study evaluates plasticity and fracture in ultra-small volumes utilizing Acoustic Emission (AE) monitored nanoindentation. Recently developed AE sensor integrated into an indenter tip provided a greatly enhanced sensitivity to contact loading induced transient processes and eliminated sample size effects. This enabled detection of AE events for the ultra-light contacts below 1 mN and provided an adequate basis for the AE signal analysis. Evaluations involved thin films ranging from several nanometers thick native oxides on metals to 100 nm thick SiC films on Si. Evaluated phenomena included cohesive film fracture, film/substrate delamination and yield initiation in oxidized metal surfaces. Indentation curves and in-situ images of the indented areas were correlated with the AE waveforms. Advanced procedures of AE signal decomposition provided additional information on separation of plasticity and fracture induced contributions of AE signals.

10:00 AM Break

10:20 AM
Stick-Slip Behavior of Polymer/Oxide Interfaces under Four-Point Bending: *Yvete Toivola*[1]; Mat Ivill[2]; Brian Somerday[2]; Neville R. Moody[2]; [1]University of Minnesota, Chem. Eng. & Matls. Sci., 151 Amundson Hall, Box 128, 421 Washington Ave. S.E., Minneapolis, MN 55455 USA; [2]Sandia National Laboratories, PO Box 969, Livermore, CA 94550-0969 USA

This presentation focuses on the adhesion of interfaces analogous to those encountered during MEMS component fabrication using LIGA. These metal/metal-oxide/polymer interfaces are studied using four-point bend sandwich test specimens. After interfacial crack initiation, the observed load-displacement (P-d) response, under displacement control, is dominated by stick-slip behavior arising from cycles of crack initiation, propagation and arrest. The origin of this stick-slip behavior was investigated through a study in which, prior to testing, sandwich specimens were exposed for seven days to a controlled temperature (either 23 or 65°C) and humidity (either dry or immersed in water) environment. Results indicated that moisture played a role in the P-d response while elevated temperatures only served to aid water diffusion through the interface. The stick-slip P-d response was repeatable over three-orders of magnitude variation in the imposed displacement rate from 2.5*10-5 to 2.5*10-3 mm/s.

10:40 AM
Macro Stress Mapping on Thin Film Buckling: *Philippe Goudeau*[1]; Pascale Villain[1]; Nobumichi Tamura[2]; [1]University of Poitiers-CNRS, LMP, UMR 6630, SP2MI, Blvd. Marie et Pierre Curie, BP 30179, Futuroscope, Chasseneuil, Vienne 86962 France; [2]LBNL, ALS, 1 Cyclotron Rd., MS 2-400, Berkeley, CA 94270 USA

Thin films elaborated by sputtering techniques generally exhibit large residual stresses which may be responsible of spontaneous detachment of the film from the substrate and in the case of compressive stresses, thin film buckling. Although these effects are undesirable for future applications, one may take benefit of it for thin film mechanical properties investigation. Since 1980, a lot of theoretical works have been done to develop mechanical models with the aim to get a better understanding of driven mechanisms giving rise to this phenomenon and thus to propose solutions to avoid such problems. Nevertheless, only a few experimental works have been done on this subject to support these theoretical results and nothing concerning local stress measurement mainly because of the small dimension of the buckling. In this communication, we present micro beam x-ray diffraction measurements which have been done on 3rd generation synchrotron radiation sources for stress/strain mapping analysis of thin film buckling.

11:00 AM
In-Situ Acoustic Emission Monitoring of Nanoindentation: *Antanas Daugela*[1]; Natalia I. Tymiak[1]; Thomas J. Wyrobek[1]; [1]Hysitron, Inc., R&D, 5251 W. 73rd St., Minneapolis, MN 55439 USA

A newly developed in-situ Acoustic Emission (AE) monitoring technique is a synergy of high bandwidth nanoscale contact characterization and advanced digital signal processing algorithms. The technique targets brittle films and substrates. High speed elastic and Surface Acoustic Wave (SAW) propagation phenomena are monitored simultaneously with quasi-static loading, by means of an AE sensor integrated into the nanoindentation test instrument. Nanometer scale contact wave propagation phenomena is observed starting from a few hundred kHz to several MHz. Advanced signal processing is an essential feature of the newly developed technique. Digital wavelet transforms and joint time-frequency analyses enhanced by neural network optimization are used for AE signal decomposition. Experimental results of AE based characterization of W(100), MgO and thin SiC films are presented, where interface cracking, delamination and fracture modes are differentiated by means of the statistical and digital signal processing analyses.

11:20 AM
Alloy Strengthening Effects on Adhesion of Gold Films: *Neville R. Moody*[1]; David F. Bahr[3]; David P. Adams[2]; Megan J. Cordill[3]; [1]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA; [3]Washington State University, Mechl. & Matls. Eng., Pullman, WA 99164-2920 USA

Adhesion is a critical factor in the performance and reliability of gold-on-chromium hybrid microcircuits where diffusion of copper from attached leads during processing and service can alter the adhesive strength of gold films. As a result, we have studied the interfacial fracture of gold, gold-on-copper, and gold-copper alloy films on sapphire substrates to assess the effects of copper on hybrid microcircuit reliability. Nanoindentation showed that the hardness values and strengths of the gold and gold-on-copper films were similar but significantly less than corresponding values for the gold-copper alloy film due to solid solution strengthening. Tungsten overlayers and nanoindentation were then used to trigger delamination and blister formation in these three film systems from which fracture energies and interfacial bond strengths were obtained using mechanics-based models. The results revealed that the interfacial fracture energies for the gold-copper alloy film were four times higher than gold on copper and eight times higher than pure gold films. In this presentation, we will describe the techniques used to determine interfacial fracture properties, and use the results to show how alloy strengthening affects interfacial fracture. This work supported by USDOE Contract DE-AC04-94AL85000.

11:40 AM
Micro-Cracking in Contact Mechanics: *Ismail Demir*[1]; [1]King Saud University, Dept. of Mechl. Eng., PO Box 800, Riyadh 11421 S. Arabia

Contact mechanics has been developed to investigate the behavior of materials to improve their wear and fatigue resistance. Moreover nano-indentation has been widely used to obtain some material properties and measure residual stresses. During contact loading and indentation process microcracking is observed as one of the failure modes. This can be seen in different forms as cone cracks, subsurface cracks or radial cracks on the surface. A fully coupled exact formulation of microcrack generation and interaction model in a half-plane is presented in the current study. Different forms of pressure distributions and friction conditions are assumed to investigate crack generation history during the indentation process. Two-dimensional and axisymmetric three-dimensional contacts are analyzed first. Stress intensity factors are monitored for each microcrack to control the failure and crack closure conditions are also taken into considerations. The modifications for a coating material on a substrate are outlined. The current analysis is kept in the frame of linear elastic fracture mechanics and further additions for nonlinear response are discussed for future studies.

Fatigue and Creep of Metal Matrix Composites: Fatigue of Metal Matrix Composites - II

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

Thursday AM	Room: 206
February 21, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Russ Chernenkoff, Ford Motor Company, Mfg. Sys. Dept., MD3135, Rm. 2022B, 2101 Village Rd., Dearborn, MI 48124 USA; Yu-Lin Shen, University of New Mexico, Dept. of Mechl. Eng., Albuquerque, NM 87131 USA

8:30 AM Keynote

Thermal Cycling and Thermal Fatigue Behavior of Metal Matrix Composites: *Krishan K. Chawla*[1]; [1]University of Alabama at Birmingham, Dept. of Matls. Eng., BEC 254, 1530 3rd Ave. S., Birmingham, AL 35294-4461 USA

Thermal expansion mismatch between a ceramic reinforcement and a metallic matrix in a metal matrix composite can lead to thermal stresses under varying temperature conditions. Over the years we have studied the behavior of a variety of metal matrix composites under the thermal cycling conditions. In order of increasing complexity these are: composites with a single crystal matrix, polycrystalline matrix, precipitation hardenable matrix, and composites with metallic matrix that reacts with the ceramic reinforcement at high temperatures. Various modes of observed microstructural damage caused by thermal stresses include plastic deformation of the ductile matrix, void formation at the interface, and possible fracture of the ceramic reinforcement. Under certain conditions, quantitative damage parameters involving loss in stiffness and density can be used to study damage evolution as a function of thermal cycles. These parameters can also provide useful design information.

9:10 AM

The Cyclic Plastic Strain Response, Deformation and Fracture Behavior of 2009 Aluminum Alloy Metal Matrix Composite: *T. S. Srivatsan*[1]; Meslet Al Hajri[1]; [1]The University of Akron, Div. of Matls. Sci. & Eng., Akron, OH 44325-3903 USA

Development and emergence of discontinuously reinforced metal-matrix composites has created an inescapable need for a thorough and systematic understanding of microstructural effects on: (a) deformation (quasi-static and cyclic) and fracture characteristics, and (b) cyclic strain resistance and concomitant stress response characteristics and fatigue life. This technical presentation will focus on rationalizing, using principles of materials science and mechanics of solids, the underlying mechanisms governing cyclic strain-amplitude controlled fatigue response and fracture behavior of a silicon carbide particulate (SiCp)-reinforced 2009 aluminum alloy metal-matrix composite. The emphasis will be on understanding the synergistic influence of composite microstructure and test temperature on fatigue performance and associated failure modes. The key damage mechanisms will be elucidated and rationalized in light of the concurrent and mutually interactive influences of nature of loading (i.e. cyclic strain amplitude), response stress, and intrinsic composite microstructural effects. Material provided by: DWA Aluminum Composites (Chatsworth, CA, USA); (Program Manager: Mr. C. Smith).

9:30 AM

Fatigue Life Prediction of Titanium Matrix Composites by Substructuring Method: *Zhenhai Xia*[1]; W. A. Curtin[1]; [1]Brown University, Div. of Eng., 184 Hope St., Providence, RI 02912 USA

A three dimensional finite element model for predicting both crack growth and fiber breakage is developed and applied to predict fatigue crack growth and fatigue lives in a SiC-fiber reinforced Ti matrix composite. A substructuring technique is used in the finite element calculation, enabling to simulate fatigue crack growth in large-size composites without losing the details of the composite structure. Furthermore, a special elimination method is used to make the simulation process very efficient. The matrix fatigue crack is assumed nucleated on the first loading cycle by the formation of a crack in the reaction layer around a fiber and on the surface of the specimen. We first calculate both the matrix crack tip stress intensity factor and the local fiber stress concentrations due to the matrix crack, and then allow the crack to grow according to Paris-law model, and fibers to break due to statistics of the fibers. This process is repeated until composite failure occurs. Fiber failure preferentially occurs within the matrix crack region, where the fiber stresses are comparatively high, and composite failure occurs when the damage in this region is sufficient to drive fiber failure throughout the remainder of the composite in a fracture-like mode. At high stresses near the quasistatic tensile strength of the material, the central fiber inside the reaction layer crack fails upon the first loading and permits much faster initial growth of the matrix crack. Predictions for the low-cycle fatigue of Ti-matrix (IMI834) reinforced with SCS-6 SiC fibers compare well with available experimental data at high stresses.

9:50 AM Break

10:10 AM Invited

Thermo-Mechanical Characterization of MMCs by Mechanical Spectroscopy: *EfraÌn CarreÒo-Morelli*[1]; [1]University of Applied Sciences of Western Switzerland, Competence Grp. Matls. & Design, Rte. de Rawyl 47, Sion CH-1950 Switzerland

Damping capacity, stiffness and dimensional stability are important properties in MMC components, especially in applications requiring positioning accuracy in a changing thermal environment. Mechanical spectroscopy (damping, shear modulus and torsional deformation measurements as a function of temperature) allows to characterize the response of MMCs subjected to thermal cycling. A transient damping maximum, which is absent in monolithic materials, is observed during cooling. It is strongly dependent on reinforcement geometry and volumetric fraction, matrix strength, and interfacial strength. The damping level increases with the cooling rate, and decreases with the frequency and oscillation amplitude. This behavior originates in the relaxation of interface thermal stresses by micro-creep of the matrix around the reinforcements. An overview of damping behavior, shear modulus evolution and strain ratcheting of MMCs during thermal cycling will be presented. The results will be discussed in terms of the development of plastic zones in the matrix, near the metal-ceramic interfaces.

10:40 AM

Fracture and Fatigue of Bulk Metallic Glass Composites: *John J. Lewandowski*[1]; Peravudh Lowhaphandu[1]; Sergey Solvíyev[1]; [1]Case Western Reserve University, Dept. Matls. Sci. & Eng., Cleveland, OH 44106 USA

The fracture and fatigue behavior of bulk metallic glass composites are being determined on bend specimens tested at temperatures ranging from -125C to 225C. The presentation will begin with a reveiw of work conducted to determine the static fracture toughness of bulk metallic glass using both notched and fatigue precracked specimens, followed by a review of the fatigue crack growth behavior of the bulk metallic glass tested at R=0.1 Tests conducted on toughened bulk metallic glass containing a dispersion of a crystalline metallic toughening phase will then be presented. The effects of the toughening regions on the fracture and fatigue behaivor under the different conditions will be described. Partial support provided by DARPA-SAM, Reference Metals, and AFOSR-AASERT.

11:00 AM

Fatigue Behavior of Single Ceramic Fibers: *Matthew Kerr*[1]; Jason Williams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

Characterizing the behavior of fibers is of great importance, regardless of whether the fiber is used in woven form or as a continuous fibrous reinforcement in a composite. In a composite, it is the fiber, of course, that typically imparts high strength and stiffness to the composite. Testing of single fibers, however, is quite problematic, particularly when the fibers are brittle and exhibit poor handling characteristics. A sophisticated microforce testing system was used to conduct single fiber testing. Reproducible and stable cyclic loads were applied to study the cyclic fatigue behavior of single fibers. The elastic modulus, fiber strength (characterized by a Weibull distribution), and stress versus cycles behavior of several high performance ceramic fibers were determined. The effect of processing-induced flaws and the effect of fiber microstructure on mechanical behavior will be discussed.

11:20 AM

Fatigue Property Evaluation of Thixoformed SiCp/AZ91D Magnesium Composites: *Shae K. Kim*[1]; Young-Jig Kim[1]; [1]Sungkyunkwan University, Sch. of Metall. & Matl. Eng., 300 Chunchun-dong, Jangan-gu, Suwon, Gyeonggi-do 440-746 Korea

Magnesium metal matrix composites are gaining increased importance for transport and electronics applications where low inertia is required, with their improved stiffness, wear resistance, and elevated-temperature properties. Although the use of ceramic particulate instead of fibrous reinforcements has help to reduce the over-all material cost, however, the processing-related cost should also be considered. A novel low cost Rotation-Cylinder method has been developed for preparation of particulate reinforced MMC materials, with the aim of rapid incorporation and homogeneous distribution of reinforcement particulates in an ambient atmosphere. RCM claims to significantly reduce the time required for incorporation and particulate agglomerations by the U-shaped melt surface with the Rankine vortex. Sound magnesium composites can be produced in conjunction with subsequent investment casting or thixoforming. This paper presents the results of the fatigue and wear properties of these cost effective SiCp/AZ91D magnesium composites, which were prepared by RCM and then subsequently processed by thixoforming.

Fundamentals of Advanced Materials for Energy Conversion: Complex Hydrides II

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

Thursday AM — Room: 613
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: George Louis Powell, Y-12 National Security Complex, BWXT-Y12, LLC, PO Box 2009, Bear Creek Rd., Oak Ridge, TN 37831-8096 USA; James (Jim) E. Klein, Westinghouse Savannah River Company, Savannah River Tech. Ctr., Bldg. 773-A, Aiken, SC 29808 USA

8:30 AM Invited

The Hydriding Kinetics of Organic Hydrogen Getters: *George Louis Powell*[1]; [1]BWXT-Y12, LLC, Y-12 Natl. Security Complex, PO Box 2009, Bear Creek Rd., Oak Ridge, TN 37831-8096 USA

The aging of hermetically sealed systems is often accompanied by the gradual production of hydrogen gas that is a result of the decay of environmental gases and the degradation of organic materials. In particular, the oxygen, water, hydrogen ìequilibriumî is affected by the removal of oxygen due the oxidation of metals and organic materials. This shift of the above ìequilibriumî towards the formation of hydrogen gas, particularly in crevices, may eventually reach an explosive level of hydrogen gas or degrade metals by hydriding them. The latter process is generally delayed until the oxidizing species are significantly reduced. Organic hydrogen getters introduced by Allied Signal Aerospace Company, Kansas City Division have proven to be a very effective means of preventing hydrogen gas accumulation in sealed containers. These getters are relatively unaffected by air and environmental gases. They can be packaged in a variety of ways to fit particular needs such as porous pellets, fine or coarse [gravel] powder, or loaded into silicone rubber. The hydrogen gettering reactions are extremely irreversible since the hydrogen gas is converted into an organic hydrocarbon. These getters are based on the palladium-catalyzed hydrogenation of triple bonds to double and then single bonds in aromatic aryl compounds. DEB (1,4 bis (phenyl ethynyl) benzene) typically mixed with 25% by weight carbon with palladium (1% by weight of carbon) is one of the newest and best of these organic hydrogen getters. The reaction mechanisms are complex involving solid state reaction with a herteogeneous catalyst leading to the many intermediates, including mixed alkyl and aryl hydrocarbons with the possibilities of many isomers. The kinetics of the reaction as a function of hydrogen pressure, stoichiometry, and temperature for hydrogen and deuterium near ambient temperature for pressures near or below 100 Pa over a wide range (in some cases, the complete) hydrogenation range. *Managed by BWXT Y-12, LLC for the US Department of Energy under contract DE-AC05-00OR22800.

8:55 AM Plenary

Hydrogen-Solid Interaction to Store Energy for Mobility: *Louis Schlapbach*[1]; Andreas Z¸ttel[2]; Christophe Emmenegger[2]; Philipp Mauron[2]; Patrick Sudan[2]; [1]EMPA-Swiss Federal Lab for Materials, Research and Testing, D¸bendorf 8600 Switzerland; [2]University of Fribourg, Phys. Dept., CH-1700 Friburg Switzerland

Mobility is a socioeconomic reality for which we spend about one third of our energy consumption producing significant amounts of CO2. Hydrogen, when produced from water and a clean primary energy is an attractive synthetic fuel with a high ratio of chemical energy to mass. As hydrogen is a molecular gas at room temperature compacting is an issue. High strength composite containers mechanically allow pressures up to 500bar, however, the gas-surface interaction has to be taken into account. Liquifaction goes across ortho-para conversion at a solid surface. The interaction of gaseous hydrogen with nanostructured graphitic carbon leeds to physisorption (too weak for room temperature storage) or to the formation of stable hydrocarbons. There is no clear evidence for an intermediate state. It turns out that hydrogen sorption into crystalline and amorphous metals with the formation of hydrides is a practicable and safe solution, so far limited to below 5 mass % when operated near room temperature. Several light weight metal systems, well known to form to stable hydrides of high capacity, look now more attractive due to improved catalyzed kinetics.

9:20 AM Invited

Modern Neutron Methods for the Study: *John J. Rush*[1]; T. J. Udovic[1]; [1]National Institute Standards & Technology, Ctr. for Neutron Rsrch., 100 Bureau Dr., Stop 8562, Gaithersburg, MD 20899-8562 USA

Neutron scattering and analysis techniques are extremely powerful, often unique, probes for the study of the location, bonding states and diffusion of hydrogen in all classes of materials. Developments over the past decade at NIST and elsewhere have greatly increased the sensitivity and dynamic range of neutron methods. We will review these developments and the latest state-of-the art capabilities, and provide examples of recent applications in the study of hydrogen in a number of technologically interesting systems, including metal hydrides, fuel cell materials, fullerness and carbon nanotubes, and zeolites. Examples will include measurements of dynamic behavior and vibrational spectra over a time regime from 10-8 to 10-14s.

9:45 AM

Thermal Cycling of Cold-Worked V-0.5 at.%C Alloy for Hydrogen Storage: *Dhanesh Chandra*[1]; Archana Sharma[1]; William N. Cathey[1]; Robert C. Bowman[2]; Franklin Lynch[3]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., Mackay Sch. of Mines, MS 388, Reno, NV 89557 USA; [2]NASA Jet Propulsion Laboratory, MS 79-24, Pasadena, CA 91109 USA; [3]Hydrogen Consultants, Inc., 12400 Dumont Way, Denver, CO 80125 USA

The effects of thermal cycling and cold-work of V-0.5 at.%C alloy on the thermodynamic and structural parameters were investigated. Thermal cycling between β and γ phase hydrides increased the hysteresis but the desorption pressure did not significantly change. Prestraining this alloy also increased the hysteresis but the desorption pressure decreased slightly as compared to that of the unstrained alloy. Microstrains, <ε^2>^1/2, in the β phase of the thermally cycled hydrides decreased after 778 cycles and the domain sizes increased. However in the γ phase, both the microstrains and the domain sizes decreased after thermal cycling. The dehydrogenated a phase after 778 thermal cycles, obtained by heating the hydrides in a differential thermal analyzer, showed residual microstrain in the lattice, similar those observed for the intermetallic hydrides. The effects of thermal cycling and cold-work on absorption and desorption pressures, H/M ratio, microstrains, long range strains and domai n sizes in the β and γ phase hydrides of V-0.5 at.%C alloy are presented.

10:10 AM Invited

Engineering Properties of Complex Hydrides: *George J. Thomas*[1]; Gary C. Sandrock[2]; Karl J. Gross[2]; [1]University of Nevada-Reno, 537 Spirit Ridge Ct., Reno, NV 89511 USA; [2]Sandia National Laboratories, Livermore, CA 94550 USA

The complex hydrides, NaAlH4 and Na3AlH6, have demonstrated rapid and reversible hydrogen storage when doped with Ti and other catalysts. These materials exhibit much greater intrinsic storage capacities, by weight, than intermetallic hydrides and have better thermodynamic properties (e.g, higher equilibrium pressures at lower temperatures) than light weight covalent hydrides. However, little is known concerning the engineering of hydrogen storage beds using these new materials. We have investigated the properties of these, as well as other related materials, which are important for their use in engineering applications. Specifically, determinations of packed bed properties, including hydrogen absorption and desorption kinetics, thermal conductivity, packing density and expansion/contraction behavior have been made. These results will be presented and discussed in terms of hydrogen storage applications.

10:35 AM Break

10:45 AM
Synthesis of Nanocrystalline Metal Hydrides by High Temperature Milling: *Jacques Huot*[1]; Robert Schulz[1]; [1]Hydro-Quebec, 1790 Boul. Lionel-Boulet, Varennes, Quebec J3X 1S1 Canada

It has recently been shown that magnesium-based metal hydrides such as Mg2Ni could be synthesized by high energy mechanical alloying. This process creates a nanocrystalline alloy that have enhanced hydrogen sorption properties due to the high density of defects and grain boundaries. Unfortunately, this process also requires a long synthesis time (up to 100 hours in some cases). In order to reduce processing time and cost of synthesis, we have investigated the effect of milling at elevated temperature under an inert atmosphere. Milling at high temperature improves interdiffusion of the elements and reduces the number of structural defects such as holes, dislocations and internal strain. It was found that by milling elemental powders at 200∞C, a complete synthesis of Mg2Ni could be achieved in 8 hours. The hydrogen sorption kinetics and capacity of this material are comparable to Mg2Ni synthesized at room temperature.

11:05 AM
Is Carbon Material Promising for Hydrogen Storage?: *Tetsu Kiyobayashi*[1]; Susumu Kajita[2]; Atsushi Ueda[1]; Hiroshi Shioyama[1]; Nobuhiro Kuriyama[1]; [1]National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan; [2]Matsushita Electric Works, Ltd., Elect. & Plastic Matls. Co., R&D Div., 1048, Kadoma, Osaka 571-9696 Japan

Since the intriguing claims that certain carbon materials can store a huge amount of hydrogen, we have been examining the gas phase hydrogen adsorption and desorption experiments on various materials. So far we have not been able to confirm those claims. In the course of the measurement we encountered several problems which we had to circumvent in order not to obtain erroneous result. In the present paper we will explain how we can make the gas phase adsorption and desorption measurements reliable as well as our result with using the carbon materials we produced.

11:25 AM
SAES ST909 Material Phases after Methane Cracking: *James E. Klein*[1]; Arthur R. Jurgensen[1]; [1]Westinghouse Savannah River Company, Savannah River Tech. Ctr., Bldg. 773-A, Aiken, SC 29808 USA

SAES St909 is a ZrMnFe alloy with 10 wt% aluminum as a pellet binder and is used to ìcrackî or getter impurities from various gas streams. Bench scale methane cracking tests were performed using a stack of ten whole pellets with feed compositions ranging from 2.5 to 100 vol% methane with helium and hydrogen carrier gases, from 600 to 800∞C at 760 torr and 10 sccm. XRD analyses performed on the St909 after methane cracking tests were complicated by the number of alloys that can be formed by the Zr, Mn, Fe, Al, and carbon. XRD results showed the expected ZrC alloy, but also the formation of aluminum transition metal carbides as the carbon content and the temperature of the cracking test increased. Some Al4C3 was also found in some of the pellets.

11:45 AM
Research on New Ca-Ni Based Alloys for Hydrogen Storage: *Hiroyuki T. Takeshita*[1]; Yoshiyasu Sakamoto[1]; Toshio Oishi[2]; Nobuhiko Takeichi[1]; Tetsu Kiyobayashi[1]; Hideaki Tanaka[1]; Nobuhiro Kuriyama[1]; Hiroshi Senoh[1]; [1]National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan; [2]Kansai University, Dept. of Matls. Sci. & Eng., Yamada, Suita, Osaka 564-8680 Japan

Ca-Ni based alloys were examined on structures, hydrogenation properties and the effect of additional elements on hydrogenation properties. Some of the alloys were decomposed to form a new hydride which coexisted with Ni and had a CsCl-type structure on the constituent metal atoms at high temperatures such as 673K under a hydrogen atmosphere. In order to improve the stability of the original compound, the candidates of elements which were substituted for Ca or Ni in the alloys were investigated based on Miedemaís semi-empirical model and Pd was chosen as a result. The effect of Pd addition on the hydrogenation properties was examined and discussed.

Fundamentals of Advanced Materials For Energy Conversion: Gas Clathrate Hydrates

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

Thursday AM	Room: 614
February 21, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Claudia Rawn, Oak Ridge National Laboratory, 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA; E. Dendy Sloan, Center for Hydrate Research, Cheml. Eng. Dept., Colorado School of Mines, Golden, CO 80401 USA

8:30 AM Plenary
Clathrate Hydrates as Materials: *E. Dendy Sloan*[1]; [1]Center for Hydrate Research, Cheml. Eng. Dept., Colorado Sch. of Mines, Golden, CO 80401 USA

This address is an overview of clathrate hydrates and their properties. If we wish to quantify hydrate properties, it is vital to measure the hydrate phase, rather than the associated phases. Acceptable samples are important for three reasons: (1) sample integrity and replication provides a basis for all experimental work, (2) laboratory and pipeline hydrates are significantly different from in situ hydrates, and (3) there are at least four hydrate structures from which to discriminate. Once the sample integrity is assured, three ways of measuring hydrates crystals are: (1) microscopic means e.g. X-ray/neutron diffraction, NMR, or Raman spectroscopy, (2) mesoscopic means such as SEM, single crystal formation, and growth, (3) macroscopic means such as crystal size distribution measurements. This address concerns mostly time-independent hydrate properties, and should provide a background for the other addresses in the hydrates session.

9:10 AM Invited
Physical and Thermodynamic Properties of Gas Clathrate Hydrates Determined by In Situ Neutron Scattering: *Claudia J. Rawn*[1]; *Bryan C. Chakoumakos*[1]; Camille J. Jones[2]; Simon L. Marshall[1]; Laura A. Stern[3]; Susan Circone[3]; Stephen H. Kirby[3]; Brian H. Toby[2]; Daniel C. Dender[2]; Yoshinobu Ishii[4]; [1]Oak Ridge National Laboratory, Metal & Cer. Div., PO 2008, Bldg. 4515, Oak Ridge, TN 37831-6064 USA; [2]National Institute of Standards & Technology, 100 Bureau Dr., MS 8562, Gaithersburg, MD 20899 USA; [3]US Geological Survey; [4]Japan Atomic Energy Research Insitute, Tokai Japan

Neutron scattering methods are being applied to determine the physical behavior of technologically and environmentally important gas clathrate hydrates as a function of temperature, pressure, and composition. To date, we have studied methane deuterohydrate (Type I), CO2 deuterate (Type I), THF deuterohydrate (Type II), and a mixed methane-ethane deuterohydrate (Type II) in this way. In our Rietveld refinements, the rotationally disordered guest molecules are modeled with appropriately disordered rigid bodies, which allows the temperature dependence of the mean-square-displacement of each guest molecule to be determined. This provides a means of evaluating the positional disorder of the guest molecules within the oversized cages. These results will be applied to the assessment of the stability of clathrate hydrates in geological environments. Long-term, this work will impact the development of technology for methane recovery from natural gas hydrate deposits as well as deep-ocean sequestration of industrial waste carbon dioxide. Research sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory (ORNL), managed by UT-Battelle, LLC for the US Department of Energy under Contract No. DE-AC05-00OR22725.

9:40 AM Invited
Initial Experiments on Ocean CO2 Sequestration and the Effects of CO2 Clathrate-Hydrate Formation: *Edward T. Peltzer*[1]; Peter G. Brewer[1]; [1]Monterey Bay Aquarium Research Institute, R&D, 7700 Sandholdt Rd., Moss Landing, CA 95039-9644 USA

Sequesteration of CO2 in the deep ocean as a means of reducing the atmospheric impact of fossil fuel combustion and possibly ameliorating greenhouse gas induced warming was first proposed by Marchetti (1977). A variety of ocean sequestration options have been proposed but few have been investigated beyond preliminary modeling studies or simple laboratory experiments. Through the use of MBARIís advanced

ROV technology, we have begun to explore some of the ocean sequestration options. During the past five years, we have conducted a series of 7 experiments addressing some of the possible modes of ocean sequestration including mid-water releases of droplet plumes and benthic releases in deep (3000-3600 m) environments. We have observed the physical behavior of liquid CO2 in a cold, high pressure environment where clathrate hydrate formation is thermodynamically favored. Properties and processes under investigation included liquid CO2 density and buoyancy relative to the surrounding seawater, rates of dissolution of both liquid CO2 droplets and CO2 hydrate, rates of hydrate formation, effects of dissolved CO2 on pH, and the impact of CO2 sequestration on marine organisms.

10:10 AM Break

10:25 AM Invited
Dynamics of Gas Hydrates: *Julian Baumert*[1]; *Christian Gutt*[2]; *Werner Press*[3]; *John S. Tse*[4]; Stefan Janssen[5]; Mark Johnson[6]; [1]Universitaet Kiel/ILL, IEAP/Compg. for Sci., 6, rue Jules Horowitz, BP 156, 38042 Grenoble France; [2]Universitaet Dortmund, Experimentelle Physik I, Dortmund Germany; [3]Universitaet Kiel, IEAP, Kiel Germany; [4]NRC, Ottawa Canada; [5]PSI, Villigen Switzerland; [6]ILL, Grenoble France

Despite the similarities between ice and hydrates, the hydrates display some unique properties. One of the outstanding anomalies is the glass-like temperature behavior of the thermal conductivity. A main contribution to this phenomenon is thought to arise from a strong coupling between low-frequency guest and host lattice vibrations which promotes an effective scattering mechanism for the heat carrying acoustic phonons. We were able to observe the low frequency rattling modes in a high-resolution inelastic neutron experiment on Xenon hydrate [1,2]. Measured intensities point to a strong coupling which lattice dynamical calculations describe as a symmetry avoided crossing between the flat Einstein-like guest modes and the acoustic host modes. When we extended our study to methane hydrate the observed frequencies rose and the coupling mechanism changed drastically. Apparently the size and mass of the guest molecule have a strong influence on the coupling mechanism. [1] J. S. Tse, V. P. Shpakov, V. R. Belosludov, F. Trouw, Y. P. Handa, W. Press, Europhys. Lett., 54 354 (2001). [2] C. Gutt, J. Baumert, W. Press, J. S. Tse, S. Janssen, to be published.

10:55 AM Invited
Structure Characterization and Sono-Stimulated Kinetic Study with Tracers in Pressure Vessels: An Integrated Approach to the Study of Gas Hydrates: *Devinder Mahajan*[1]; T. F. Koetzle[1]; L. Brammer[2]; W. T. Klooster[3]; R. L. McGraw[1]; R. K. McMullan[1]; G. Senum[1]; [1]Brookhaven National Laboratory, Tech. Dept., Chem. Dept. & Energy Scis., Upton, NY 11973 USA; [2]University of Missouri-St. Louis, Dept. of Chem., St. Louis, MO 63121 USA; [3]ANSTO, Lucas Heights, NSW Australia

We are using an integrated approach to study the structure and decomposition kinetics of gas hydrates. First, neutron and X-ray diffraction studies are being carried out on laboratory prepared single crystals. Building upon our earlier work including neutron and X-ray studies of the cubic II systems, 3.5Xe.8CCl4.136D2O and xH2S.8CS2.136H2O, and the unusual tetragonal bromine hydrate 20Br2.172D2O, we initially plan to study deuterated methane hydrate. This study will provide the ability to accurately characterize the hydrogen-bonded clathrate water-cage structures, including the hydrogen (deuterium) atoms and their attendant disorder. Second, two powerful techniques, sonolysis and perfluorocarbon tracers (PFTs) will be combined to conduct a kinetic study that is realistic for direct subsurface application. This study will involve methane hydrate formation in the presence of a tracer of appropriate size, followed by sono-stimulation of localized hydrate crystals in which PFT will be tracked without thermal perturbation of the surroundings. The proposed studies will be conducted in an integrated Bench-Scale Gas-Hydrate Kinetic Unit that we have specially modified for this purpose.

11:25 AM Invited
Methane and Carbon Dioxide Hydrate Investigations Utilizing a 70 Liter Pressure Vessel: *Tommy J. Phelps*[1]; Olivia R. West[1]; [1]Oak Ridge National Laboratory, Environmental Scis. Div., POB 2008-6036, Oak Ridge, TN 37831-6036 USA

The Seafloor Process Simulator (SPS) is a 72 liter pressure vessel developed to investigate physical and biogeochemical processes affecting the formation, stability and dissociation of methane and carbon dioxide hydrates. The vessel is 31 cm in diameter and 91 cm long containing 41 ports. The size of the SPS enables upscaling and visualizations of processes under controlled experiments. Nucleation and dissociation conditions for methane hydrates revealed that 200 mg/liter suspensions of bentonite clays in water lowered observed nucleation overpressures ~50%, corresponding to 6° of subcooling. Suspensions of silica powder lowered temperatures observed for the dissociation equilibrium ~1°. Experiments with carbon dioxide revealed dense hydrate-containing rods that sank within the SPS. Accordingly, the SPS has demonstrated usefulness for examining kinetics, formation, stability, and dissociation characteristics under controlled conditions. Importantly, upscaling of processes such as the sequestration of carbon dioxide has been demonstrated facilitating their examination under in situ conditions.

Fundamentals of Structural Intermetallics: High Temperature Strength of TiAl

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

Thursday AM	Room: 615-616
February 21, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA; Pelagia I. Gouma, SUNY Stony Brook, Matls. Sci. & Eng., 314 Old Engineering, Stony Brook, NY 11794-2275 USA

8:30 AM Invited
Creep Behaviour of TiAl Alloys with Enhanced High-Temperature Capability: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strasse, Geb 45, Geesthacht D-21502 D-21502 Germany

The paper describes an experimental study of creep processes in two-phase titanium aluminide alloys involving mechanical testing and electron microscope observations. Conventional lamellar alloys suffer from insufficient creep resistance due to diffusion assisted dislocation climb processes. There are also significant microstructural changes which give rise to a complete spheriodisation of the lamellar morphology and degrade the creep strength. In an attempt to overcome these problems a novel class of alloys with the base line composition (in at.%) Ti-45Al-(5-10)Nb was developed and subjected to precipitation hardening by carbon additions. Optimized alloys exhibit significantly improved creep resistance and structural stability at the intended service conditions. The creep behaviour of these alloys was characterized in terms of the Dorn equation by thermodynamic glide parameters and detailed electron microscope analysis of the relevant micromechanisms. The results are related to processing and technical application of the alloys.

9:00 AM
Theory of Interface Properties for Carbide Precipitates in TiAl: *Roy Benedek*[1]; [1]Argonne National Laboratory, Bldg. 223, Argonne, IL 60439 USA

Various additions to gamma-TiAl have been investigated in recent years with the objective of improving host mechanical properties, particularly high-temperature creep. Of particular interest have been the carbide precipitates Ti3AlC (cubic perovskite), and Ti2AlC (hexagonal). Properties of the interfaces of such precipitates with the matrix influence their interaction with dislocations, and thereby affect mechanical properties. First principles calculations will be presented of properties of interfaces of the above-mentioned carbides with TiAl. Interface energies and atomic structure for the different possible interface terminations will be compared. An approximate correction to the coherent interface calculations for the effect of lattice constant mismatch will be described.

9:20 AM
Effect of Aging on Creep in K5 Gamma Alloys with/without Carbon Content: *Pelagia I. Gouma*[1]; Young-Won Kim[2]; [1]SUNY Stony Brook, Dept. MSE, 314 Old Engineering, Stony Brook, NY 11794-2275 USA; [2]UES Materials & Processes Division, Dayton, OH 45432 USA

Multicomponent gamma alloys containing increased amounts of refractory elements and/or decreased aluminum contents have shown considerably improved creep resistance over the first generation alloys such as Ti-48Al-2Nb-2Cr and XD alloys. These improvements are considered due to solid solution hardening as well as increased volume fraction of hard alpha-2 phase especially in fully-lamellar material. K5 (Ti-46Al-2Cr-3Nb-0.2W) is one example. By adding small amounts of carbon (C) and/or carbon + silicon (C+Si) to K5 alloy fully-lamellar materials were produced with to improved the creep resistance. The resulting microstructure consists of alternating layers of gamma laths and particle arrays. This paper discusses the role of isolated particle arrays during creep deformation. Fully lamellar materials were produced from three K5 alloys (K5, K5-0.2Si, and K5-0.2Si-0.1C). The results show that the aging treatment reduced creep deformation for all alloys, however, in the increasing order, indicating that alpha-2 particles interact with dislocations.

9:40 AM
Microstructure Stability during Creep Deformation of Hard Oriented PST Crystal of TiAl: *Hee Y. Kim*[1]; Kouichi Maruyama[1]; [1]Tohoku University, Dept. of Matl. Sci., Grad. Sch. of Eng., Aobayama02, Sendai 980-8579 Japan

The hard orientation with the lamellar plates oriented parallel to compression axis were deformed at 1150K under the applied stress of 158-316MPa. Microstructural changes were examined quantitatively for PST crystal during creep deformation. The six orientation variants are present with the similar proportion, but the most frequently observed γ interface is true twin type. After the creep deformation significant microstructure changes have been recognized. These are the dissolution of α_2 lamellae, the formation of ledges at lamellar interfaces, and the globularization of lamellae. Lamellar coarsening by dissolution of γ interface was observed. The fraction of 120° rotational interface and the pseudo twin interface decreased, while the faction of true twin interface increased. The twin related γ interfaces migrated by ledge formation and growth, and the interfaces disappeared by edge migration. The 120° rotational interface migrated substantially and formed equiaxed grains. The stability of α_2/γ and γ/γ interfaces during creep deformation and its effect on creep resistance were discussed. The effect of stress and strain on microstructural evolution was also investigated associated with the creep deformation behavior.

10:00 AM Invited
Effect of Twinning and Stress-Induced Phase Transformation on the Primary Creep of TiAl Intermetallic Alloys: *Wei-Jun Zhang*[1]; Seetharama C. Deevi[1]; [1]Chrysalis Technologies, Inc., Rsrch. Ctr., 7901 Whitepine Rd., Richmond, VA 23237 USA

TiAl-base alloys generally exhibit a large primary strain during creep at relatively high stress levels. The mechanisms responsible for this large primary strain are not well understood yet. In this paper, the stress dependence of primary creep strain in various TiAl alloys is analyzed through modeling of the creep strain curves using theta-concept formula. It is found that the primary strain increases significantly with increasing stress beyond a threshold stress, sth. Analysis of the experimental data suggests that the sharp increase is due to the activation of twinning and stress-induced phase transformation (SIPT) at higher stresses. Twinning and SIPT result in a large instantaneous strain e0. The influence of alloying additions and heat treatment on the sth and e0 are discussed. Appropriate aging treatment appears to be critical for engineering application of TiAl-base alloys.

10:30 AM
Comparison of Primary Creep Behaviors at Low and High Stress Region as a Function of Fully Lamellar Morphology in P/M Ti-48Al-2W: *Dong Yi Seo*[1]; Jonathan Beddoes[1]; Linruo Zhao[2]; [1]Carleton University, Dept. of Mechl. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]Institute for Aerospace Research, Natl. Rsrch. Council, Struct., Matls. & Propulsion Lab., 1500 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

This paper correlates the primary creep behaviour of a near γ-TiAl+W with different fully lamellar (FL) morphologies in two different stress states. Minimizing the total primary creep and understanding the primary creep behaviour are key for several envisaged applications.Primary creep consists of two major components-the instantaneous strain and a subsequent primary transient. The primary creep behaviour at 760°C and 140 or 276MPa of P/M TiAl+W in FL conditions is evaluated. Different FL structures are produced by two step cooling from the α phase or aging treatments.The aged condition significantly improves primary creep resistance.The contribution to primary creep of the instantaneous strain and subsequent primary transient depends on the microstructural state and can be correlated to tensile yield behaviour at 760°C. At 140MPa, slightly higher instantaneous strains are observed when lamellar interface precipitates are present, but the opposite trend occurs at 276MPa. The deformation response such as dislocation structure, dislocation reaction with third phase precipitates,and twinning at lamellar and grain boundary regions are compared in the samples crept at the different stress conditions. The results are discussed in terms of the microstructural factors controlling primary creep deformation.

10:50 AM
Interface-Controlled Diffusional Creep in Lamellar TiAl with Refined Microstructures: *Luke L. Hsiung*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-352, PO Box 808, Livermore, CA 94551 USA

Creep mechanisms of lamellar TiAl with refined microstructures at an intermediate temperature range (650~810°C) have been investigated. A nearly linear creep behavior (i.e. the steady-sate creep rate is nearly proportional to the applied stress) was observed under stresses below ~300 MPa. Since the operation and multiplication of lattice dislocations within constituent lamellae are very limited at a low stress level, creep mechanisms based upon glide and/or climb of lattice dislocations become insignificant, instead the mobility of interfacial dislocation arrays on lamellar interfaces becomes predominant. The mobility of interfacial dislocation arrays is primarily controlled by the impingement of lattice dislocations, and the diffusion of solute segregation in lamellar interfaces. The nearly linear creep behavior observed at low-stress regime can be rationalized by the viscous glide of pre-existing interfacial dislocation arrays. It is suggested that the segregation of W to lamellar interfaces has a beneficial effect to the creep resistance of refined lamellar TiAl at the low-stress regime by decreasing the mobility of interfacial dislocations.

11:10 AM
Strengthening Mehanisms in the Ti-(40-48)Al-(0-12)Nb System: *Young-Won Kim*[1]; Dennis M. Dimiduk[2]; [1]UES, Inc., Matls. & Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 10th St., Ste. 1, Bldg. 655, Wright-Patterson AFB, OH 45433 USA

The strengthening mechanisms of the Nb-containing two-phase gamma TiAl alloys were studied for the compositions within the representative alloy system Ti-(40-48)Al-(0-12)Nb. Alloy materials having systematically chosen compositions were produced in arc-melted cigar forms, weighing 300 g each. These were hot-die forged into plates. Samples from forged plates were annealed at 1000∞C for 192h, and the resulting microstructures consisted of uniformly distributed fine equiaxed grains of constituent phases (γ, α2, or B2). Through detailed microprobe composition analysis, an isothermal section of the phase boundaries for the alloy system was constructed. Compressive yield strengths were measured at RT, and were found to vary with composition. A fully-lamellar material was also produced for each alloy under fixed annealing and cooling conditions. Each FL material aged at 1000∞C for 24h was compression tested for yield strength measurements. An attempt is underway to explain the strength variation based on the fractions and morphologies of phases present and the solution hardening of each constituent phase as the result of Nb-partitioning. Grain size variation with composition will also be taken into account.

11:40 AM
Beta Phase Decomposition in a Grain-Refined Creep-Resistant Titanium Aluminide Alloy: Hongwei Yang[1]; Tai-Tsui Cheng[1]; *Mark Aindow*[1]; [1]University of Connecticut, Metall. & Matls. Eng., 97 N. Eagleville Rd., Unit 3136, Storrs, CT 06269-3136 USA

In recent work it has been shown that both the properties and the microstructural stability of lamellar TiAl-based alloys can be enhanced by heavy alloying. Alloys with compositions in the range Ti-44Al-8 (Nb,Ta,Zr,Hf)-(0-0.2)Si-(0-l)B (in at. %) were investigated and it was shown that the microstructures of these were significantly different from those of binary or 48-2-2 type alloys, with lamellar plus gamma and transformed beta regions. These differences led to significant increases in the strength, creep resistance and/or ductility of these alloys depending on composition and thermal history [1,2]. In the present study we have considered the microstructural development in one of these alloys, Ti-44Al-4Nb-4Zr-0.2Si-0.3B, in more detail. This alloy was selected as having the most promising properties of those considered in the previous work. In this paper we will present TEM data obtained from this alloy after heat-treating at 1350∞C and cooling to room temperature at various rates. It will be shown that the main effect of cooling rate is upon the morphology and distribution of the decomposition products in the transformed beta regions of these microstructures. In all cases the beta phase firstly orders to B2 and then decomposes to give a mixture of B2, w and g phases depending on the cooling rate. The w phase forms from the B2 both via the usual displacive/re-ordering mechanism, and by direct precipitation giving two distinct orientation relationships. In contrast, the transformation to the g phase occurs by discontinuous coarsening of existing g lamellae. The effect that these microstructural variations might have on the mechanical behavior of the alloys will be discussed. [1] T. T. Cheng and M. H. Loretto, Acta Mater. 46:4801 (1998). [2] T. T. Cheng, M. R. Willis and I. P. Jones, Intermetallics 7:89 (1999).

12:00 PM
Effects of Gd Addition and Lamellar Spacing on Tensile and Compressive Creep Behaviours of a Fully Lamellar Ti-44Al-1Mn-2.5Nb Alloy: *Kenong Xia*[1]; Xiaolin Wu[1]; Dan Song[2]; [1]University of Melbourne, Dept. of Mechl. & Mfg. Eng., Victoria 3010 Australia; [2]Northeastern University, Sch. of Matls. & Metlgcl. Eng., Shenyang, Liaoning 110006 China

A quaternary Ti-44Al-1Mn-2.5Nb (at%) alloy and an equivalent alloy with 0.15 at% rare earth element Gd were prepared by casting. Following heat treatments to obtain a fully lamellar microstructure, both alloys had an average grain size of ~270 µm and lamellar spacing of the order of 0.5 µm. Different heat treatments using faster cooling produced much finer lamellar spacings of the order of 0.05 µm in both alloys. Tensile creep tests showed that with the same microstructure the secondary creep rate of the Gd-containing alloy was lower than that of the quaternary alloy, and for the same alloy the material with finer lamellar spacings exhibited a significantly lower secondary creep rate than that with coarser lamellar spacings although the steady state creep observed in the latter material disappeared in the finer spaced material. However, compressive creep tests showed that a steady state creep existed in all the materials, and although the materials with finer lamellar spacings still displayed lower creep rates the effect of Gd was not observed. The results were discussed based on TEM and SEM observations.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Grain Boundaries

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

Thursday AM	Room: 617
February 21, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Dorte Juul Jensen, Riso National Laboratory, Matls. Rsrch. Dept., Fredriksborgvej, Roskilde DK 4000 Denmark; Tadao Watanabe, Laboratory of Materials Design and Interface Engineering, Dept. of Mach. Intell. & Sys. Eng., Grad. Sch. of Eng., Tohoku University, Sendai, Japan

8:30 AM Invited
In-Situ Determination of Grain Boundary Migration during Recrystallization: *Dorte Juul Jensen*[1]; [1]Riso National Laboratory, Matls. Rsrch. Dept., Fredriksborgvej, Roskilde DK 4000 Denmark

By a novel high energy synchrotron technique it is possible to determine the crystallographic orientation and the elastic strain of micron-sized local volumes in the bulk of metal samples. This technique is referred to as 3D x-ray diffraction (3DXRD) microscopy, and it has been used to follow the growth of individual grains in-situ during re-crystallization. It has been found that the grains could be detected already when they were about 1µm in size, i.e. the early growth of the nuclei is also observed. In total the growth of several hundred nuclei/grains were characterized in 2 aluminium alloys. It was found that each nuclei/grain has its own growth characteristics and that the growth rates vary significantly in time. Based on the data growth rate distributions are deduced. In the presentation, the 3DXRD microscope and the experimental procedure is shortly described. The results are summerized and discussed with reference to earlier growth rate determinations using more traditional techniques. Finally the implications of a growth rate distribution on recrystallization modelling is discussed.

8:55 AM Invited
Grain Boundary Engineering under External Fields: *Tadao Watanabe*[1]; [1]Laboratory of Materials Design and Interface Engineering, Dept. of Mach. Intell. & Sys. Eng., Grad. Sch. of Eng., Tohoku University, Sendai Japan

Grain boundaries are important microstructural elements which can produce a wide variety of functions and also bring about a drastic improvement of performance of structural and functional polycrystalline materials. Since the concept of grain boundary design and control for high performance polycrystalline materials was proposed by the author, the concept has been widely tested and proved to be powerful for designing and developing high performance polycrystalline engineering materials, not only metallic, also ceramic and semiconductor materials, through the control of new microstructural parameters associated with grain boundaries; ìGrain Boundary Character Distribution GBCDî, ìGrain Boundary Connectivityî and grain Boundary geometrical configurations. The present paper will discuss the ways in which grain boundaries can be effectively manipulated during processings under external fields, e.g. magnetic field, stress field, and temperature gradient, on the basis of recent investigations made by the authorís group and others. High performance magnetostrictive ferromagnetic material for micromachines such as actuator and high performance photovoltaic material like polysilicon for solar cell, have been developed. I believe that it is the right time when materials scientists and engineers should recognize and utilize the importance, variety and potential of grain (including interphase) boundaries in development of high performance advanced materials even nanocrystalline materials with a huge density of boundaries, in the 21st century.

9:20 AM Invited
Atomistic Aspects of Grain Boundary Fracture: *Diana Farkas*[1]; [1]Virginia Tech, Dept. of Matls. Sci., Blacksburg, VA 24060 USA

These studies use interatomic potentials and molecular level simulations to investigate the detailed atomistic nature of the fracture mechanisms along interfaces in metals and alloys. The many body interatomic potentials (of the embedded atom type) used for the calculations are obtained from the perfect lattice experimental properties and from first principle quantum mechanical calculations. Mixed potentials are created to describe the experimental thermodynamics of the system. Large-scale simulations of cracked bi-crystals allow the study of the fracture processes in these alloys. Dislocation emission from the crack tip and/or crack propagation are observed, and correlated with the ductile/brittle response. The role of the detailed atomistic structure of the grain boundary was found to be essential in brittle crack propagation along the interface.

9:45 AM Invited
Characterization of Grain Boundaries in the Five Dimensional Space of Mesoscopically Observable Parameters: *Gregory S. Rohrer*[1]; David M. Saylor[1]; Adam Morawiec[2]; Kevin W. Cherry[1]; F. H. Rogan[1]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, MSE, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]Instytut Metalurgii i Inzynierii, Materialowej PAN, Reymonta 25, 30-059, Krakow Poland

The character of a grain boundary can be determined by five mesoscopically observable parameters: three that describe the lattice misorientation and two that describe the boundary inclination. While the distribution of lattice misorientations is frequently derived from the analysis of planar sections, grain boundary inclinations are rarely reported for more than a handful of boundaries. We have used a combination of SEM, OIM, and serial sectioning to characterize the grain boundary network in polycrystalline MgO. All five degrees of freedom were defined for 7x106 square microns of grain boundary surface area in 1 cubic millimeter of the sample. We have found that there is a significant texture in the space of grain boundary planes (for boundaries of fixed misorientations). Peaks in the distribution suggest that ìspecialî boundaries occur with high probability. The relative energies of the grain boundaries were reconstructed under the assumption that the grain boundary triple junctions were in local equilibrium. The relatively low energy boundaries correspond to peaks in the grain boundary distribution. However, simple geometric models involving dislocation arrays or coincident lattices do not predict that all of these boundaries should be special. In this paper, we will describe details of the distribution and energy anisotropy and advance plausible explanations for the observations.

10:10 AM
Coincidence Site Lattice Theory of Triple Junctions and Quadruple Points: *Val Y. Gertsman*[1]; [1]Pacific Northwest National Laboratory, PO Box 999, P8-16, Richland, WA 99352 USA

The coincidence site lattice theory of grain boundaries is extended to multi-crystallite ensembles, in particular to triple junctions and quadruple points. It provides a basis for analyzing the junction structures as well as polycrystalline microstructure. Examples of application of the theory to some model microstructures are presented. Implications of the theory for the grain boundary engineering approach are discussed.

10:30 AM
Stochastic Method for the Evolution of Microstructures with Particular Reference to Grain Boundary Networks: *Mukul Kumar*[1]; Roger W. Minich[1]; James S. Stˆlken[1]; Christopher Schuh[1]; [1]Lawrence Livermore National Laboratory, 7000 E. Ave., L-356, Livermore, CA 94550 USA

Grain boundary engineering has been successfully applied to improve materials properties such as intergranular corrosion and cracking, creep, and weldability. This has been attributed to an increase in the fraction of special grain boundaries from thermomechanical processing. Our investigations, however, have led to the conclusion that the basis for these improvements is most likely to lie in the break-up of the connectivity of random boundary networks. The experimental determination of the network topology has been accompanied by a microstructure modeling effort that is derived from the percolative nature of the network. The problem of grain boundary networks, under crystallographic constraint, has been simulated using a stochastic approach that considers the probability distribution function for each boundary based on the fluctuations in strain energy and intrinsic characteristics such as interfacial energy. The influence of both multiplicative and additive fluctuations toward microstructural evolution during recrystallization and grain growth will be elaborated. Experimentally quantified microstructures using grain boundary character and triple junction distributions will be compared to those obtained from simulations.

10:50 AM
Clustering, Agglomeration, Pushing and Engulfment of Inclusions at High Temperature Metallurgical Melt Interfaces: *Sridhar Seetharaman*[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Pittsburgh, PA 15213 USA

Recent results obtained with a High Temperature Confocal Scanning Laser Microscope to visualize interfaces between molten metals and slags and advancing solidification fronts are presented. Especially, the behavior of inclusions is elucidated with respect to (i) clustering, agglomeration and separation at slag/metal interfaces and (ii) particle pushing/engulfment at solidification fronts.

Magnesium Technology 2002: Magnesium Corrosion and General Sessions

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

Thursday AM	Room: 606
February 21, 2002	Location: Washington State Conv. & Trade Center

Session Chairs: Alan Luo, General Motors, Staff Rsrch. Eng., Matls. & Proc. Lab, General Motors R&D Ctr., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA; Howard I. Kaplan, Magnesium Corporation of America, 238 N. 2200 W., Salt Lake City, UT 84116 USA

8:30 AM
Emerging Trends in Corrosion Protection of Magnesium Die-Castings: *Jan Ivar Skar*[1]; Darryl Albright[2]; [1]Norsk Hydro Research Center, Sect. for Matls. Tech., PO Box 2560, Porsgrunn N-3907 Norway; [2]Hydro Magnesium, Magnesium Mkt. Dvlp., 39209 Six Mile Rd., Ste. 200, Livonia, MI 48152 USA

Due to the improved corrosion resistance of magnesium alloys brought forward by the development of the high purity grades 20 years ago, the use of die cast magnesium components has moved towards more and more demanding applications. The corrosion resistance is sufficient so that magnesium is used in the under-body environment of vehicles without additional general corrosion protection, although special attention must be given to galvanic corrosion protection. There has recently been an increasing interest in both semi-exterior and exterior automotive parts. Since magnesium alloys are not compatible with standard automotive phosphate treatments, the parts need to be pre-coated prior to assembly. For such components, the challenges are to find efficient, environmentally friendly pre-treatments and robust coating systems that can maintain their integrity through the assembly process. The present paper reviews some of the established and newly developed methods for corrosion protection and finishing of die cast magnesium automotive parts.

9:00 AM
Corrosion and Wear Resistance of Electroless Nickel on Magnesium Alloys: *George E. Shahin*[1]; [1]Atotech USA, 1750 Overview Dr., Rock Hill, SC 29731-2000 USA

Magnesium is the worldís lightest metal with a specific gravity of 1.74. Aluminum weighs 150% more, iron and steel 400% more, copper and nickel alloys 500% more and some glass filled plastic weigh 25% more. This lightness combined with a good strength-to-weight ratio of magnesium has found increasing utilization in the transportation and electronic fields. Magnesium is also the most electrochemically active metal. This high reactivity of magnesium limits its application to certain environments. This paper will discuss the pretreatment of magnesium alloys in addition to electroless nickel plating magnesium alloys. Also discussed will be the corrosion resistance of magnesium alloys with various electroless nickel coatings and the wear resistance on electroless nickel on magnesium alloys. The corrosion resistance will be evaluated by neutral salt spray and various electrochemical techniques.

9:20 AM
A Novel Technique to Evaluate the Corrosion Behavior of Magnesium Alloys: *Basant L. Tiwari*[1]; John J. Bommarito[1]; [1]General

Motors, R&D Ctr., Matls. & Proc. Lab., MC 480-106-212, Warren, MI 48090-9055 USA

A novel technique for measuring the intrinsic corrosion rate of magnesium alloys has been developed. As a magnesium alloy sample dissolves in 5% NaCl solution, the dissolution rate is determined by measuring the amount of HCl added to the NaCl solution to control the pH between 5 and 7. The corrosion rate is determined from the slope of the curve where the dissolution rate reaches a steady state. The technique was used to compare the corrosion behavior of newly developed creep-resistant magnesium alloys with several known magnesium alloys. It was shown that the corrosion rate of AC52 (Mg-5%Al-2%Ca) is comparable to AZ91D. This technique provides a fast way to screen the effect of small composition changes on corrosion behavior of Mg alloys. Because of the short test time, this technique provides a powerful tool to accelerate alloy and product development with Mg alloys.

9:40 AM

Aqueous Corrosion Characteristics of Mg-4Ni-xAl Alloys in Acid-Chloride Solution: *Yoon-Seok Choi*[1]; Jung-Gu Kim[1]; Shae K. Kim[1]; Young-Jig Kim[1]; [1]School of Metallurgy & Material Engineering, 300 Chunchun-dong, Jangan-gu, Suwon, Gyeonggi-do 440-746 Korea

Aqueous corrosion properties of Mg-4Ni-xAl alloys in acid-chloride solution were studied. Three NA40, NA41 and NA42 alloys were prepared by Rotation-Cylinder Method. The corrosion behavior of them was investigated at room temperature by polarization tests, pH measurement, hydrogen evolution test, galvanic corrosion tests and surface analyses. The results of polarization test indicated that with increasing Al content, (a) the corrosion potential had more active potential while corrosion rate decreased and (b) the segregation of the ·-Mg/Mg2Ni eutectic phase at grain boundary was reduced. This is because micro-galvanic reactions (a) between the Mg matrix and the ·-Mg/Mg2Ni eutectic phases and (b) between the ·-Mg and Mg2Ni in the eutectic phase itself were reduced, which decreased the corrosion rate of Mg alloys. Galvanic corrosion tests for consideration of Mg-4Ni-xAl alloys as structural materials were also done compared to AZ91D Mg alloy coupled with low carbon steel, Al alloy and Cu alloy.

10:00 AM

Improved Corrosion Performance of AZ91D Magnesium Alloy by the Keronite‘ Coating Process: *S. Shrestha*[1]; A. Sturgeon[1]; P. Shashkov[2]; A. Shatrov[2]; [1]TWI, Ltd., Granta Park, Great Abington, Cambridge CB1 6AL UK; [2]Keronite, Ltd., PO Box 700, Granta Park, Great Abington, Cambridge CB1 6ZY UK

This paper describes the results of a study on the corrosion behaviour of a die cast magnesium alloy type AZ91D coated with the Keronite‘ process. This process is a new commercially available and environmentally friendly electrolytic coating method applicable to all types of magnesium alloys. The process involves creation of a hard ceramic oxide layer onto a light substrate alloy by plasma electrolytic oxidation (PEO) in a low concentrated alkaline solution. Corrosion performances were assessed in a 3.5% NaCl solution using an accelerated electrochemical potentiodynamic polarisation method (ASTM G61) and a 1000-hour of salt spray exposure (ASTM B117). Galvanic corrosion tests were performed according to ASTM G71. For comparison purposes in terms of performance, parallel tests were carried out on an uncoated AZ91D alloy. The study demonstrated the improved corrosion resistance of Keronite coated AZ91D magnesium alloy and its potential for use in severe corrosive environments.

10:20 AM Break

10:35 AM

Sealing Methods for Enhanced Corrosion Protection of Anodized Magnesium Alloy WE43A-T6: *Richard George Rateick*[1]; Shen-Jiang Xia[2]; Viola I. Birss[2]; [1]Honeywell International, Engines & Sys., 717 N. Bendix Dr., S. Bend, IN 46637 USA; [2]University of Calgary, Chem. Dept., 2500 University Dr. N.W., Calgary, Alberta T2N 1N4 Canada

High voltage anodizing processes continue to be developed for providing corrosion protection to magnesium alloys. However, the large pore structure in these coatings represents a barrier to further increases in corrosion resistance. The objectives for the work reported here were to develop methods of sealing the pores in complex parts where reentrant surfaces preclude the use of spray type top coating systems. A second objective was that the coating not flake off of internal surfaces, thus eliminating a source of foreign object damage for aerospace applications. Salt fog, electrochemical impedance, throwing power and adhesion data will be presented for anodized magnesium alloy WE43A-T6. The anodization was performed using both a commercial and a developmental process. The sealing operations were performed using Parylene grades C and HT. The Parylenes are vapor deposited polymers.

10:55 AM

Joining of Light Hybrid Constructions Made of Magnesium and Aluminum Alloys: *Adi Ben-Artzy*[1]; Avraham Munitz[2]; Gavri Cohn[1]; Avigdor Shtechman[1]; [1]Rotem Industries, Ltd., Metal Forming Grp., POB 9046, Beer-Sheva 84190 Isreal; [2]N.R.C.N, POB 9001, Beer-Sheva 84190 Isreal

Magnesium alloys are widely used in automotive applications due to reduction in fuel consumption and to their good energy absorption. The growing demand for improved mechanical properties and longer fatigue life has increased the implementation of wrought magnesium alloys, especially for moving and safety parts. For some applications, joining of different magnesium alloys produced by extrusion, forging, or high-pressure die-casting, is essential to enable the manufacturing of complex parts and hybrid structures. Since magnesium alloys occupy only a small fraction of materials used for cars or aircraft construction, it is essential to join (weld) the alloys to other frames and body parts. Therefore, joining (welding) of dissimilar materials i.e. aluminum or steel alloys to magnesium alloys is essential. When high-pressure die-cast alloys such as AM50 was welded to other alloys, the welding heat popped up the close porosity of the cast parts, and weakened the joint area by creating foam-like zones. It was found that use of high energy focused welding system like an electron Beam (EB) or a laser, the effect of heat on the die cast alloy is reduced, and a strong joint is formed. In the present study, Al-6063 and different magnesium alloys were EB and GTAW welded. It was found that Beta-Mg17Al12 phase is formed in the fused zone. The presence of this inter-metallic phase is the main reason causing brittleness and low strength of the joints. There are indications that use of proper welding electrode composition may reduce this problem, and eventually enable the welding of dissimilar joints of aluminum and magnesium alloys.

11:15 AM

Resistance Spot Welding of Mg AM50 and AZ91D Alloys: *Abraham Munitz*[1]; C. Cotler[1]; Gabrial Kohn[2]; [1]Nuclear Research Center-Negev, PO Box 9001, Beer Sheva 84190 Israel; [2]Rotem Industries, Rotem Industrial Park, PO Box 9046, Beer Sheva 84190 Israel

Resistance spot welding of Mg AM50 and AZ91 2-mm thick cast-plates were successfully performed. The welds were examined by X-ray radiography, conventional optical microscopy and scanning electron microscopy. It was found that the weld nugget consisted of two regions; I) Central molten zone, II) Partially melted Heat Affected Zone (HAZ). The size and morphology of the nugget depend on 3 main factors: i) The alloy composition, i.e. circular morphology for Mg-AM50, and rectangular for Mg-AZ91D. ii) Current. i.e. the higher the current, the larger was the central melted zone. iii) Pulse duration: A minimum pulse duration of 2 s was needed for creating a bond. Above 2 s, longer current duration resulted in larger central molten zones. Under our experimental conditions, the pressure of the electrodes during welding had only a minor effect on the weld morphology. Grain boundary melting was observed in the majority of the HAZ. These created short-cut channels for melt transfer to the outer surface even far away from the central molten zone, which in turn caused depression of the surface of the welded plates, and shrinkage porosity in the nugget. The latter depended also on the porosity in the cast plates near the nugget. The impact of molten grain boundary and thermo-mechanical processing on the nugget morphology and its mechanical properties will be discussed by current solidification theories.

11:35 AM

The Study on Friction Stir Welding of AZ31 Mg Alloy: *Won Bae Lee*[1]; Seung Boo Jung[1]; Yun Mo Yeon[2]; [1]Sung Kyun Kwan University, Dept. of Adv. Matl. Eng., 300, Chun Chun Dong, Jang An Gu, Suwon, Kyung Gi Do 440-746 Korea; [2]Suwon-Science College, Dept. of Automatic-Welding Eng., Whasung, Kyung Gi Do 445-742 Korea

Friction stir welding is a new, solid-state welding technique which was invented by The Welding Institute(TWI) in 1991. The recent studies have restricted to aluminum alloy so few data was published about welding of Mg alloy and other materials. Mg alloy are potential candidate to replace aluminum alloys in many structural application, due to some of their unique properties. Mg alloy have a low density, high strength-to-weight ratio and good castability. They are considered advanced materials for energy conservation and environmental pollution regulation. Joining of Mg parts, which may be crucial for these application, is still limited. Mg alloy are easily oxidized in welding zone because of high chemical reactivity at high temperature. So Mg alloy have a many weld defect by conventional fusion welding method. The purpose of this research is to evaluate the applicable possibility of FSW in AZ31 joining.

11:55 AM
Thermophysical Property Measurement of Magnesium Alloy: AE42: *Deming Wang*[1]; R. A. Overfelt[1]; Yemi Fasoyinu[2]; Mahi Sahoo[2]; [1]Auburn University, Mechl. Eng., 201 Ross Hall, Auburn, AL 36849 USA; [2]CANMET/MTL, 568 Booth St., Ottawa K1A 0G1 Canada

This paper will present some methods to measure thermophysical properties of magnesium alloys in Auburn University. Such as measuring special heat, latent heat, solidus and liqudus temperatures, solid fraction in mushy zone by using the DSC technique; measuring thermal expansion and density by using the dilatometer and laser distance detection technique; measuring viscosities of these molten alloys by using the oscillating cup viscometer technique. Accurately to measure these data is very important for the computer modeling programs. The paper will discuss how to control measurement errors, reduce oxidization and some details about above employed techniques in these measurements of magnesium alloys. In addition, applications and experimental measuring data are presented in the paper for a magnesium alloy, AE42.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - IV

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

Thursday AM — Room: 211
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chung-li Taiwan; S. W. Chen, National Tsing-Hua University, Dept. of Chem. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu 30043 Taiwan

8:30 AM Invited
Phase Equilibria and Solidification Properties of Sn-Cu-Ni Alloys: *Sinn-Wen Chen*[1]; Chih-Hao Lin[1]; [1]National Tsing-Hua University, Cheml. Eng., 101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

Ternary Sn-Cu-Ni alloys were prepared and annealed at 240°C. The annealed alloys were metallographically examined and the equilibrium phases formed were identified based on the compositional determinations and x-ray diffraction analysis. The isothermal section of the ternary Sn-Cu-Ni system at 240°C was proposed based on the experimental results of this study and related phase equilibrium knowledge in the literatures. The binary compounds, Cu6Sn5, Ni3Sn2, and Ni3Sn4 have very extensive ternary solubility. Continuous solid solutions are formed between Cu and Ni as well as between Cu3Sn and Ni3Sn. Besides the isothermal section, the liquidus projection of Sn-Cu-Ni system was determined based on previous literature results and the information of primary solidification phases of various Sn-Cu-Ni alloys examined in this study.

8:55 AM Invited
Copper Migration Phenomena in IC Package under High Temperature Aging: Hen-So Chang[1]; *Ker-Chang K. Hsieh*[1]; Theo Martens[2]; C. C. Chen[2]; [1]National Sun Yat-Sen University, Inst. of Matls. Sci. & Eng., Kaohsiung Taiwan; [2]Philips Electronic Building Elements Industries (Taiwan), Ltd., Tech. Dev. Div., 10, Chin 5th Rd., N.E.P.Z., PO Box 35-48, Kaohsiung Taiwan

Copper migration phenomena found in several IC samples during the new materials evaluation for the IC plastic package. The copper atom can migrate from the copper leadframe through the gold wire surface and reach the ball bond. The Cu-Au alloy phase formed on the gold wire surface as well as the Cu-Au-Al alloy phase formed on the ball bond. Another phenomenon is the copper atom may react with the phosphorus particles, which used as flame retardant in green compound. Cu3P and CuP2 phases formed inside the plastic compound and cause the short circuit failure. The atom migration phenomena are due to three driving forces: diffusion, alloy formation and Galvanic effect.

9:20 AM Invited
Diffusion Soldering for High Temperature Stable Thin Film Bonds: *Rainer Schmid-Fetzer*[1]; Thomas Studnitzky[1]; [1]Technical University of Clausthal, Inst. of Metall., Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

Diffusion soldering (DS), a special form of transient liquid phase bonding (TLP) or isothermal solidification (IS) can be used to form strong bonds at low temperature which remain solid up to a much higher remelting temperature. Thin film processing makes DS especially attractive for micro/optoelectronics or microsystems if the joined parts are exposed to higher temperature in later steps of device fabrication. In conventional DS systems, composed of (near) noble metals with Sn or In, the remelting temperatures are typically limited to around 400 to 500°C. In this work new DS systems involving also (early) transition metals with Sn are investigated, which may show much higher remelting temperatures. Phase formation and reaction kinetics of these systems were studied, they are essential for controlling the DS process.

9:45 AM Invited
Morphological Studies on IMC Formed between Nickel Particles and Sn-Ag Solder: *K. N. Subramanian*[1]; J. Lee[1]; F. Guo[1]; J. P. Lucas[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., E. Lansing, MI 48824-1226 USA

Morphology of the intermetallic compound (IMC) layer formed around mechanically-incorporated Ni particle in eutectic Sn-Ag solder under various reflow conditions was studied. Two distinct morphologies, sun-flower and faceted single crystal, were observed. Effects of heating and cooling rates on the morphological changes were investigated to identify the processing parameters that contribute to these morphologies. Results of IMC formation noted with Ni particles will be compared with those obtained with Ag and Cu particle reinforced composite solders. Acknowledgement: Project funded by Composite Materials and Structures Center at Michigan State University.

10:10 AM Break

10:30 AM
Phase Transformations and Microstructure Evolution in Cu-Ge Alloys: Sumanth Jagga[2]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Convergys, Inc., Cincinnati, OH 45201 USA

Alloys based on the Cu-Ge system, especially those on the ε_1-Cu_3Ge compound, have considerable potential for application as interconnects in microelectronic devices because of their attractive electrical and barrier properties. Though the thermodynamics of this system has been assessed based on available data, knowledge of phase equilibria, structures of phases and phase transformations is far from complete. The present study was undertaken to address these aspects. Arc-melted cigars of five Cu-Ge alloys ranging in composition from 10 to 30 at.%Ge were prepared, homogenized and then subjected to a series of high temperature treatments followed by quenching in various media. Transformations and reaction temperatures were studied by DTA and the microstructures of both as-cast and heat treated samples were characterized by XRD, OM, SEM, EPMA and TEM. The kinetics and temperature dependence of transformations during continuous cooling were studied using a novel, computer-controlled in-situ temperature and electrical resistivity measurement system, coupled with post-mortem analysis of the microstructures. Reaction start and finish temperatures, continuous cooling diagrams, enthalpies and driving forces associated with the ε-ε_2 to ε_1 phase transformation were determined for alloys containing 20 to 30 at.%Ge. New results related to the structure of the high-temperature phases and the nature of the transformation to ε_1 were obtained. These results will be presented and discussed.

10:50 AM
Interfacial Reactions between Ni/Cu UBM and Eutectic Sn-Pb Solder in Flip Chip Technology: *Chien Sheng Huang*[1]; Yen Ming Chen[2]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101, Sec. 2 Kuang Fu Rd., Hsinchu 300 Taiwan; [2]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 300 Taiwan

Interfacial reactions between Ni/Cu under bump metallurgy (UBM) and eutectic Sn-Pb solder in the 63Sn-37Pb/Ni/Cu/Ti/Si3N4/Si multiplayer structure for the flip chip technology were investigated. The types of interfacial reaction products varied with different reflowed times. After the first reflow, the (Cu, Ni)6Sn5 IMC was found between solder and Ni. It was argued that Cu diffused through Ni during the reflow process due to the columnar structure of Ni. There were two interfacial reaction products formed between solder and UBM after three times of reflow. The layered (Ni, Cu)3Sn4 was next to the Ni/Cu UBM. The island-like (Cu, Ni)6Sn5 was formed between (Ni, Cu)3Sn4 and solder. It was also observed that part of the island-like IMC stripped into the solder. The diffusion path for solid-liquid reaction during periods of reflow could be determined with the aid of ternary Sn-Cu-Ni phase diagram.

11:10 AM
Nucleation Mechanisms of Copper during Electrodeposition of Thin Films from EDTA Solutions: *Batric Pesic*[1]; Darko Grujicic[1]; [1]University of Idaho, COMER, Dept. of Metlgcl., Matls. Sci. & Eng., Moscow, ID 83844-3024 USA

The complexing and surface leveling properties of EDTA were studied during the nucleation of copper electrodeposition of thin films. Cyclovoltammetric (CV) and chronoamperometric (CA) electrochemical techniques were utilized in this study. Near atomically smooth glassy carbon was used as the deposition substrate. Morphological characterization was performed by atomic force microcopy. The copper nucleation mechanisms were examined as a function of solution pH, copper concentration, Cu:EDTA ratio. Electrochemical surface pretreatment effect was also examined. It was found that pH, copper concentration, surface pretreatment and the deposition voltage all had profound effects on the morphology of copper nuclei in the thin film. The CV and CA results, as a function of these parameters, will be discussed. The verification of instantaneous nucleation mechanisms will be presented.

Surface Engineering: Science & Technology - II: Characterization/Modeling

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

Thursday AM — Room: 203
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: D. S. Misra, Indian Institute of Technology, Powai, Mumbai 400076 India; Susan Pran Krumdieck, Dept. of Mechl. Eng., Christchurch 8004 New Zealand

8:30 AM Invited
Abrasive Wear Behaviour of Laser-Clad and Post Heat-Treated Tool Steel Coatings: *Sheng-Hui Wang*[1]; Jianyin Chen[1]; Lijue Xue[1]; [1]National Research Council Canada, Integrated Mfg. Tech. Inst., 800 Collip Cir., London, ON N6G 4X8 Canada

A comparative study of abrasive wear behaviour has been conducted on several laser-clad and post heat-treated tool steel coatings. Multilayer cladding with comparable thickness was deposited using a CW CO2 laser in combination with blown powder technique. The abrasive wear resistance of the clad coatings was evaluated using a dry sand/rubber wheel apparatus as per ASTM standard test method, and is compared to that of AISI D2 tool steel (60 HRC) which is often used as a reference material for evaluating abrasive wear resistance. Laser-clad CPM-15V and CPM-10V coatings exhibit superior abrasive wear resistance than D2 baseline. In contrast, the wear resistance of laser-clad CPM-9V and M4 coatings is inferior to that of the D2. The post heat-treatment of laser-clad coatings can either increase or decrease their wear resistance, depending on the material as well as the annealing temperature used. The wear scars of the laser-clad coatings were examined and the related wear mechanism has been elucidated in details.

8:55 AM
In Situ Nanoindentation of Thin Films in a Transmission Electron Microscope: *Andrew M. Minor*[1]; Eric A. Stach[2]; J. W. Morris[1]; [1]University of California, Dept. of Matls. Sci. & Eng., Matls. Scis. Div., Lawrence Berkeley Natl. Lab., One Cyclotron Rd., MS 66-200, Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, One Cyclotron Rd., MS 72, Berkeley, CA 94720 USA

The nanomechanical behavior of small solid volumes exhibits fundamental phenomena that are obscured on coarser scales. We have developed a novel experimental technique of in situ nanoindentation of thin films in a transmission electron microscope. This technique can image in real time the discrete deformation mechanisms of nano-scale volumes, while simultaneously correlating this behavior with quantitative information. Issues to be discussed will be related to the initiation of defects at the surface, the effect of grain size and the role of grain boundaries in deformation of nano-scale volumes.

9:10 AM
Curvature Method as a Tool to Evaluate Shape Memory Effects for TiNiCu Thin Films: *Yongqing Fu*[1]; Hejun Du[1]; Sam Zhang[1]; [1]Nanyang Technological University, Sch. of Mech. & Production Eng., 50 Nanyang Ave. 639798 Singapore

TiNi and TiNiCu films were prepared by co-sputtering of TiNi target with Cu and Ti targets. Curvature method was used to measure residual stress and evaluate shape-memory effects. Deposition at 723 K gave rise to low stress; deposition at room temperature resulted in large tensile stress that could be relaxed through post deposition annealing at 923 K. Upon heating, TiNiCu films generated large tensile stress when transforming from martensite to austenite, whereas during cooling, the stress relaxed significantly when the films transformed back to the ductile martensite phase. Both one-stage and two-stage phase transformations were observed and confirmed by DSC and XRD studies. Residual stresses were measured as a function of temperature that cast light on the SMA filmís crystallization and transformation temperatures, recoverable, thermal, intrinsic and martensitic yield stresses. Effects of film thickness, heating rate, annealing process and cyclic heating/cooling process on martensite phase transformation were discussed.

9:25 AM Cancelled
Characterization of a Platinum Aluminide Coating after Isothermal and Thermocyclic Heating: *Hyungjun Kim*

9:40 AM
Nucleation Mechanisms of Copper during Electrodeposition of Thin Films: *Batric Pesic*[1]; *Darko Grujicic*[1]; [1]University of Idaho, Matls. Sci. & Eng., COMER-McClure Hall, Moscow, ID 83844-3024 USA

The nucleation mechanisms of copper during electrodeposition of thin films from pure copper sulfate solutions were studied by utilizing cyclovoltammetric and chronoamperometric electrochemical techniques. Near atomically smooth glassy carbon was used as the deposition substrate. Morphological characterization was performed by atomic force microscopy. The copper nucleation mechanisms were examined as a function of solution pH and copper concentration. Electrochemical surface pretreatment effect was also examined. It was found that pH, copper concentration, surface pretreatment and the deposition voltage all have important effects on the morphology of copper nuclei in the thin film. Chronoamperometric studies were utilized as a diagnostic tool for mechanisms of copper nucleation. It was found that copper nucleation was happening via instantaneous nucleation mechanisms.

9:55 AM Break

10:10 AM Invited
Synthesis of Functional Materials Utilizing Pulsed Power Technologies: *K. Yatsui*[1]; W. Jiang[1]; H. Suematsu[1]; T. Suzuki[1]; Y. Kinemuchi[1]; S.-C. Yang[1]; [1]Nagaoka University of Technology, Extreme Energy-Density Rsrch. Inst., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan

We have proposed two applications of pulsed power technologies to material synthesis. One is a thin film preparation method by ion-beam evaporation (IBE) and another is a nanosize powder synthesis method by pulsed wire discharge (PWD). Preparation of various functional materials by IBE and PWD has been demonstrated. In IBE, a target is bombarded by an intense, pulsed proton beam. The ablation plasma, which forms from the target, deposits on substrates. Crystallized $SrAl_2O_4$:Eu,Dy, B_4C and TiFe thin films have successfully been prepared by IBE without substrate heating or sample annealing. Typical initial deposition rate is ~5 mm/s, which is almost 10^6 times faster than those by conventional thin film preparation methods. In PWD, thin wires placed in a chamber are discharged and evaporated by charged capacitors. The vapor is cooled by and reacts with gas in the chamber. Various powders, including double oxides, have been synthesized by PWD.

10:35 AM
Surface Phenomena in Diffusion-Limited Capillary Penetration of a Porous Film by a Reactive Fluid: *Rajiv Asthana*[1]; [1]University of Wisconsin-Stout, Mfg. Eng. Prog. Tech. Dept., 326 Fryklund Hall, Menomonee, WI 54751 USA

The capillary-driven penetration of a porous film by a reactive fluid is accompanied by pore shrinkage (or expansion) and contact angle decay, both of which modulate the capillary pressure driving the flow. The kinetics of diffusion-limited capillary flow of a reactive liquid in a porous film is modeled for the case of a shrinking pore and an exponentially decaying contact angle in a manner similar to the case of interface-limited flow presented elsewhere[1]. The computational outcomes for penetration of porous Si3N4 films by AgCuTi brazes, and of porous carbon films by Si confirm that, for an unstable contact angle, greater penetration is achieved at lower values of the diffusion rate constant. A classical capillary flow analysis overestimates the kinetics whereas a shrinking pore model with a constant contact angle underestimates the kinetics. The calculated limiting lengths at pore closure exhibit a better agreement with the recent measurements in the Si/C system as compared to current models of reactive flows. [1] R. Asthana, Surface Engineering in Materials Science ñ I, Seal, Dahotre, Moore and Mishra, eds., TMS, 2000, pp 287-297.

10:50 AM
Influence of Surface Roughness on the Coercivity and Magnetic Interactions in CoCrX (X=Pt,Pd,Ta,B) Thin Film Media: Mehmet Tarakci[1]; *Siva Guruswamy*[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The influences of Al-Mg/NiP alloy disk surface roughness ranging from 2 ≈ to 50 ≈ on the morphology, coercivity and magnetic interactions in Co83Cr17, Co81Cr17B2, Co78.5Cr17Pt4.5, Co78.5Cr17Ta4.5, Co78.5Cr17Pd4.5, Co76.5Cr17Pt4.5B2, Co76.5Cr17Ta4.5B2, Co75.5Cr17Pt4.5Ta3, Co72Cr13Pd12Ta3 and Co84Cr10Ta6 alloy thin films on Al-Mg/NiP/Cr substrates were examined. The grain morphologies were characterized using atomic force microscopy. The samples were prepared under conditions optimized for obtaining in-plane Cr (002) orientation and Co (11-20) orientation. The coercivity and coercivity ratio of the CoCr alloy films increase with increase in the substrate surface roughness, particularly in the range below 10 ≈. The magnetic interactions are strongly influenced by the circumferential substrates surface texture. Exchange-coupling decreases as the surface roughness of the substrates is decreased. Additions of Ta and Cr are effective in reducing the exchange coupling while Pt and Pd additions have an opposite effect at an addition level of 4.5 at.%.

11:05 AM
Effect of Faceting on the Thermal Grain Boundary Grooving of Tungsten: *Joachim H. Schneibel*[1]; Pavlo P. Sachenko[2]; Wen Zhang[3]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Oakland University, Dept. of Mechl. Eng., Rochester, MI 48309 USA; [3]Oakland University, Dept. of Math. & Stats., Rochester, MI 48309 USA

The grain boundary grooving of polycrystalline tungsten annealed at 1350°C was studied by atomic force microscopy. The profiles of the grooves between unfaceted grains were in qualitative agreement with the predictions of Mullins theory of grooving by surface diffusion. In particular, the predicted secondary maxima and minima next to the main groove maxima were often observed. Surface faceting strongly affected the grooving kinetics and groove shapes. In particular, the grooves developed between faceted grains often exhibited smaller ridges than those between unfaceted grains. Grooves forming between faceted and unfaceted grains were often asymmetric. The shapes of the asymmetric grooves could be numerically modeled by assuming that faceting inhibits surface diffusion. This material is based upon work supported by the National Science Foundation under grant DMR-9996087. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:20 AM Cancelled
Molecular Polarizability of Fullerene and Graphite Clusters: *Francisco Torrens*

11:35 AM
Studies on the Structure and Characteristics of Electrodeposited RE-Ni-W-P-B4C-PTFE Composite Coatings: *Zhongcheng Guo*[1]; Xiaoyun Zhu[1]; [1]Kunming University of Science and Technology, Fac. of Matl. & Metall. Eng., Kunming 650093 China

This paper mainly describes friction and wear characteristics of the electrodeposited RE-Ni-W-P-B4C-PTFE composite coating. It also explains the reason for these fine characteristics in view of structure.

Teaching and Learning Hydrometallurgical Science and Engineering: Educational Technology

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

Thursday AM — Room: 601
February 21, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Saskia Duyvesteyn, University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; J. Brent Hiskey, University of Arizona, Col. of Eng. & Mines, PO Box 210072, Tucson, AZ 85721 USA

8:30 AM Keynote
Challenging the Traditional Hydrometallurgy Curriculumñ An Industry Perspective: *Michael B. Mooiman*[1]; David J. Kinneberg[1]; Kathryn C. Sole[1]; [1]Metalor USA Refining Corporation, 255 John Dietsch Blvd., PO Box 255, North Attleborough, MA 02760 USA

The hydrometallurgistís role in industry has changed considerably over the past 20 years. Flowsheet development is now a smaller component of the job and, more often than not, issues of operations management, environmental regulations, worker safety, risk assessment and finances are more important. Moreover, the narrowness of the curriculum defined by the term ìhydrometallurgyî does not encompass the application of hydrometallurgical concepts to related fields such as surface finishing, recycling and the production of advanced materials by aqueous processing techniques. From an industry perspective, the traditional hydrometallurgy curriculum has shortcomings in preparing hydrometallurgists for an industrial career and changes are necessary.

9:05 AM Invited
Bringing Hydrometallurgy into a Global E-Learning Environment: *J. Brent Hiskey*[1]; [1]University of Arizona, Col. of Eng. & Mines, PO Box 210072, Tucson, AZ 85721 USA

Hydrometallurgy, as a method of processing minerals and metals, dates back into antiquity. There are many early references of the application of solutions to ores and the treatment of mine waters. Surprisingly, hydrometallurgy as a branch of the academy is relatively recent. Certainly, elements of hydrometallurgy were presented as university subjects since the mid to late 1900th century. Only after the Manhattan Project (i.e. early 1950s) was curricula formally developed. During this period there is evidence of a strong underpinning in thermodynamics, kinetics, solution chemistry and equilibria, and engineering design. In recent years there has been emphasis on advanced techniques in simulation and modeling, computation fluid dynamics, application of advanced instrumentation, and integration of the biological sciences. Traditionally, hydrometallurgical education/training has been offered at small programs comprising one or two key faculty, and at highly distributed locations (Salt Lake City, Perth, Vancouver, Reno, Johannesburg). Furthermore clients for this discipline are equally small and distributed. With advances in high speed computing, refinements in graphic design tools, and expansion of the internet, distance education has taken on a new meaning in recent years and can hopefully reshape the way we teach hydrometallurgy. As a start two things need to be done in this arena, 1) sources must be linked together to develop meaningful curricula; and 2) clients and uses (both students and practitioners) must be connected with these sources. This paper highlights some of the special characteristics of the field of hydrometallurgy and suggests how the internet can bring about global collaborations. The TriUniversity Masters of Engineering program in Arizona represents one example of this type of collaboration and partnership. This program will be reviewed in the context of developing an e-learning environment for hydrometallurgy.

9:30 AM Invited
Developments in the Teaching of Hydrometallurgy in Australia: *Michael J. Nicol*[1]; [1]Murdoch University, Extractive Metall., South St., Murdoch, WAU 6050 Australia

The teaching of courses in geology, mining and extractive metallurgy in Australian universities is undergoing significant change largely as a result of the initiative taken by the Minerals Council of Australia(MCA). This has resulted in a major program aimed at developing national under- and postgraduate courses in selected areas with the aim of making these available to a consortium of participating universities. Murdoch university, together with the A J Parker CRC for Hydrometallurgy has been commissioned to develop the course in hydrometallurgy. Progress in the development of this course will be discussed in terms of overall philosophy, content and mode of delivery. Aspects of the extension to a Web-based course will also be summarized. Murdoch University has also established a hydrometallurgical pilot plant on the campus and the results of the first continuous operation for several months using under- and postgraduate students as operators will be discussed from the point of view of practical training and providing an interface with the local industry.

9:55 AM Invited
Teaching and Learning Aqueous Processing: A Brazilian Experience: *Virginia S.T. Ciminelli*[1]; [1]Universidade Federal de Minas Gerais, Dept. of Metlgcl. & Matls. Eng., R. Espirito Santo, 35/s206, Belo Horizonte, MG Brazil

The evolution of Hydrometallurgical Education in Brazil is discussed in terms of the context and perspectives of the mining and metallurgical industries, the programs of interaction developed with industry, and research opportunities. Recent reorientation leading to a broader Aqueous Processing approach and a focus on environmental issues is also presented as well as strategies implemented to meet challenges in the competition for research funds and students.

10:20 AM Break

10:35 AM
Teaching Hydrometallurgy to Chemical Engineers and Chemical Engineering to Hydrometallurgists: *Matthew Ian Jeffrey*[1]; [1]Monash University, Chem. Eng. Dept., Clayton, Victoria 3800 Australia

This presentation will discuss the teaching of Hydrometallurgy at Monash University in Victoria, Australia. Being based in a Chemical Engineering Department, the formal teaching of Hydrometallurgy to undergraduates is limited. A reasonable number of our graduates find work in the large Hydrometallurgy based industry in Australia, and hence it is important for them to learn about hydrometallurgical processes; our approach to this problem will be discussed. The presentation will also discuss some of the subjects which are taught to our Chemical Engineers, em phasing the importance of knowledge in subjects such as Process Control, Process Design and Reaction Engineering. It is the presenterís view that Hydrometallurgists should be trained in these areas. The teaching of research in Hydrometallurgy to both the undergraduate and postgraduate students will also be discussed.

11:00 AM Invited
Teaching Corrosion via the Internet using a Variety of Tools to Enhance Lear: *Michael L. Free*[1]; [1]University of Utah, Dept. of Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

An on-line corrosion course was designed and delivered with the goal of providing a high-quality learning environment for students. The course utilizes a variety of electronic and organizational tools such as a virtual laboratory, on-line quizzes, group projects, and multi-media lecture presentations to provide high quality educational opportunities for students. The course organization and development will be discussed and demonstrated.

11:25 AM Invited
The Virtual Laboratory: Reality or a Really Bad Idea: *Saskia Duyvesteyn*[1]; R. Peter King[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

By simulating metallurgical equipment, processes and reactions, the Virtual Laboratory environment makes it possible for students to easily, quickly, conveniently and accurately perform laboratory experiments. Just as in a ìrealî lab, the student must select the necessary materials and appropriate operating parameters, then take readings/samples during the experiment, and finally send the samples for analysis. Throughout these steps, the student must take decisions that impact the outcome of the results. Where the Virtual Lab outshines ìrealityî is that if a wrong decision is made, the experiment can be reset in a matter of seconds. In the last two years that the VLab has been utilized in our courses, we are noticing a substantial improvement in student performance. VLab also makes it possible to add a laboratory component to courses that traditionally have not been able to utilize labs because of time constraints or difficulty in undertaking the necessary experiments.

11:50 AM Closing Remarks

Index

C

D

I

J

K

L

M

N

O

P

Q

R

S

T

U

V

W

X

Y

Z