

2004 USERS MEETING

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FOR THE ADVANCED PHOTON SOURCE

MEETING

PROGRAM & ABSTRACTS

PROGRAM & ABSTRACTS

May 3-6, 2004

The Advanced Photon Source
Argonne National Laboratory
Argonne, Illinois USA



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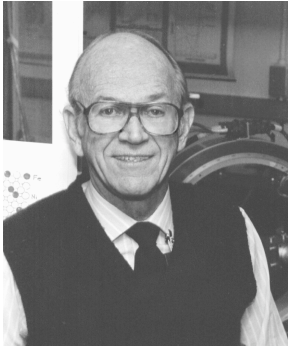
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IN MEMORY

CULLIE J. SPARKS

May 8, 1929 — March 19, 2004

Dr. Cullie J. Sparks, a charter member of the APS user community, a world-renown metallurgist, and a pioneer in x-ray optics, died recently of complications following open-heart surgery.

His seminal x-ray research is legendary in the materials characterization community. In collaboration with Professor Bernard Borie in the 1950s and 1960s, he pioneered the use of diffuse x-ray scattering to study point defects in alloys. Their “Borie-Sparks Method” is still widely used today and forms the basis for modern diffuse x-ray and neutron scattering methods. Also in the 1960s, working with researchers from Union Carbide Corporation, he combined the natural tendency of mosaic graphite crystals to focus in the plane of scatter with out-of-plane focusing based on curved surfaces to create the doubly focusing crystal optics still widely used around the world. In the 1970s, he discovered unexpected “resonant Raman x-ray” or “Sparks” scattering,” which is now routinely used as a method to study the dynamics of x-ray induced atomic transitions. Also in the 1970s, he became an advocate of emerging synchrotron x-ray sources, designing and implementing the first synchrotron-based x-ray fluorescence microprobe. In the 1980s, he proposed a new way to focus x-rays using bent perfect crystals. This method provides an order of magnitude greater intensity than alternative focusing methods and has been adopted at synchrotron facilities around the world.

Dr. Sparks received numerous awards and honors, among them his election as a Fellow of both the American Society of Metals International and the American Physical Society. He was the recipient of the Department of Energy Award for Sustained Outstanding Research and authored or co-authored more than 100 scientific journal articles and book chapters. He served on several national and university science boards, including the USA National Committee on Crystallography, and held memberships in Tau Beta Pi and Sigma Xi honor societies.

Among his APS friends, he will be remembered not just as a scientific pioneer, but also as a genuinely warm, caring individual, who was always willing to stop and help a novice or colleague wrestle with a problem. He will surely be missed.



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PROGRAM & COMPREHENSIVE PROGRAM ABSTRACTS



**2004
Rosalind Franklin
Young Investigator Award**

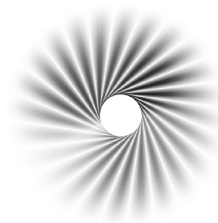
ALEXIS S. TEMPLETON

The Advanced Photon Source (APS) Users Organization is pleased to announce that Dr. Alexis S. Templeton has been chosen to receive the first APS Rosalind Franklin Young Investigator Award. Dr. Templeton's work centers on the influence of microorganisms in the speciation of heavy metals in environmental systems and the role of bacteria in the weathering of basaltic glasses in deep ocean environments.

To simultaneously investigate both the abiotic and biologically mediated reactions at biofilm/mineral interfaces, Alexis has relied heavily on a diverse array of spectroscopic and microscopic techniques. For her graduate work as a graduate student at Stanford University, she used microbeam x-ray fluorescence, conventional and grazing angle x-ray absorption spectroscopy, and long-period x-ray standing wave (XSW) synchrotron-based techniques. A particularly novel development was her combination of the XSW technique with XANES spectroscopy to determine the vertical distribution and speciation of selenium within microbial biofilms formed on oxide surfaces. The combined methods she used provide a fully three-dimensional characterization of trace element distribution and speciation at a complex interface and represent a major advance in the approach to investigating such systems.

Her current work as a Postdoctoral Research Associate at Scripps Institution of Oceanography, University of California, San Diego, involves a multidisciplinary investigation focused on identifying key microorganisms in ocean floor environments that survive through oxidation of Fe(II) and Mn(II) in basaltic glasses. Geochemical measurements are directed towards characterizing the chemical signatures associated with biologically induced weathering and constraining the relative rates of biotic vs. abiotic dissolution and oxidation processes. For this work, she has developed a protocol combining x-ray reflectivity, total reflection x-ray fluorescence, grazing-angle XANES, and x-ray diffraction measurements. These methods can be used to determine the thickness and density of reacted surface layers, identify surface-associated weathering products, and measure the redistribution and redox transformation of metals in the weathered surfaces. This work is an exciting application of synchrotron methods to a complex problem and is likely to have a major impact on our understanding of fundamental biological processes in the deep ocean.

In her brief professional career, Alexis Templeton has authored or co-authored 16 peer-reviewed publications and received several prestigious awards. The APS and the APS Users Organization are happy to add the APS Rosalind Franklin Young Investigator Award to that number.



COMPREHENSIVE PROGRAM

MONDAY, MAY 3, 2004

Registration

Building 402: Lobby
7:30 am – 6:00 pm

Exhibitor Set-up

Building 402: Gallery
8:30 am – 4:00 pm

Wine and Cheese Reception

Joint Event with First Users Meeting for the Argonne Center for Nanoscale Materials
Building 402: Gallery
4:00 pm – 6:00 pm

TUESDAY, MAY 4, 2004

Day-long Workshops

Detailed agendas begin on page 8

- WK 1 **Nanomagnetism**
Building 402: Room E1100/E1200
8:50 am – 4:30 pm
- WK 2 **Future Directions in Synchrotron Environmental Science**
Building 460 (Argonne Guest House): Conference Room A
8:45 am – 4:45 pm
- WK 3 **The Protein Crystallography Technology and Logistics Collaboration**
Building 401: Room A1100
8:30 am – 4:00 pm
- WK 4 **Inelastic X-ray Scattering: Present and Future at the APS**
Building 362: Auditorium
8:50 am – 5:30 pm
- WK 5 **Chemical and Biological Nanoscale Materials**
Building 402: Lecture Hall
9:00 am – 5:00 pm

TUESDAY, MAY 4, 2004 (continued)

Poster Session

Building 400 (Experiment Hall): Sector 32
Reception and refreshments
4:00 – 6:00

Partner User Council Meeting

Building 617, Argonne Exchange Club
6:30 – 8:30

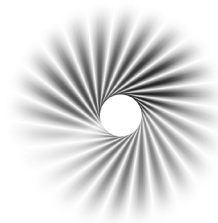
WEDNESDAY, MAY 5, 2004

Opening Session

Building 402: Lecture Hall

Chair: Stephen Durbin, Purdue University

- 9:00 Welcome
Stephen Durbin, Chair, APS Users Organization Steering Committee
- 9:05 Welcome
Herman Grunder, Director, Argonne National Laboratory
- 9:10 View from the Hill
Ellen Burns, Aide to Congressman Vern Ehlers (Michigan)
- 9:45 DOE Perspective
Patricia Dehmer, DOE Office of Basic Energy Sciences
- 10:15 Refreshments
- 10:35 APS Update
J. Murray Gibson, Associate Laboratory Director for the APS
- 11:20 Status of Insertion Device Developments and Operations at the APS
Efim Gluskin, Advanced Photon Source
- 11:45 Facility Beamlines Operations and Research—Expanding Opportunities
Gabrielle Long, Advanced Photon Source
- 12:10 **Lunch**
Building 402: Lower-level patio, under the tent



WEDNESDAY, MAY 5, 2004 (continued)

User Science Session 1

Building 402: Lecture Hall

Chair: Mark Rivers, The University of Chicago

- 1:30 The Scientific Legacy of Cullie Sparks: Contributions to X-ray Optics, Atomic Physics, Materials and Synchrotron Science
Gene Ice, Oak Ridge National Laboratory
- 2:10 Mineral Surfaces: Bridging Absorption and Co-precipitation
Richard Reeder, SUNY Stony Brook
- 2:50 Refreshments
- 3:10 The Compact Light Source: A Miniature Synchrotron Light Source for Your Home Lab
Ronald D. Ruth, Lyncean Technologies, Inc.
- 3:50 Chemical Analysis of Interplanetary Dust Particles: Clues to the First Dust in the Solar System and Preparation for the First Samples Returned from a Comet
George Flynn, SUNY Plattsburgh

Poster Session

Building 400 (Experiment Hall): Sector 32

Reception and refreshments

4:00 – 6:00

Banquet

Argonne Guest House

"A Midwestern Country Evening"

6:45 – ???

THURSDAY, MAY 6, 2004

User Science Session 2

Building 402: Lecture Hall

Chair: Stephan Ginell, Argonne National Laboratory

- 8:30 Molecular Recognition Events in RNA Interference and Glycolipid Transfer Processes
Dinshaw J. Patel, Memorial Sloan-Kettering Cancer Center
- 9:10 Designing Drugs to Fit Targets: Structure-based Drug Design in Drug Discovery
Jonathan Greer, Abbott Laboratories

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THURSDAY, MAY 6, 2004 (continued)

- 9:50 Anthrax Adenylyl Cylcase Toxin
Wei-Jen Tang, The University of Chicago
- 10:30 Refreshments
- 10:50 The Role of Synchrotron Experiments in Understanding Deep Planetary Interiors
Thomas Duffy, Princeton University
- 11:30 APSUO Election Results; Announcement of Student Poster Prize Winners
- 11:45 Rosalind Franklin Award and Acceptance Talk
Alexis Templeton, Scripps Institution of Oceanography
- Lunch**
- 12:15 Building 402: Lower-level patio, under the tent
- APS Users Organization Steering Committee**
- 12:15 Building 401: Room A5000
Joint Luncheon Meeting of the Incoming and Outgoing Members
- Afternoon Workshops**
Detailed agendas begin on page 18
- WK 6 X-ray Detector Technologies: Present and Future Directions
Building 402: Lecture Hall
1:30 – 6:30 pm
- WK 7 Small-Angle X-ray Scattering at the APS
Building 401: E1100/E1200
2:00 – 5:20 pm
- WK 8 Advancing Actinide Science
Building 401: A1100
1:30 – 5:00 pm



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PROGRAM & WORKSHOP AGENDAS & ABSTRACTS

ABSTRACTS

Workshop 1 **NANOMAGNETISM**

Organized by Dongqi Li and Seth Darling
Materials Science Division, Argonne National Laboratory

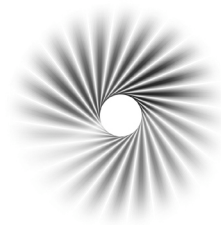
Tuesday, May 4
Building 402: Room E1100/E1200

Morning	8:50–11:50
Afternoon	1:30–4:30

Nanoscale magnetic systems, such as nanoparticles and ultrathin films, display properties distinct from bulk materials. Burgeoning industrial applications such as spintronics and magnetic recording demand a deeper understanding of these systems. Hence, their preparation and characterization are both ongoing challenges as traditional techniques run into intrinsic limitations. In this workshop, fundamental aspects of magnetism in reduced dimensionality, as well as state-of-the-art fabrication techniques, are discussed. This discussion highlights the use of various x-ray based approaches for exploring nanomagnetic systems. *Abstracts begin on page 21.*

Morning Session

- | | |
|-------|---|
| 8:50 | Welcome
<i>Eric Isaacs, Argonne National Laboratory</i> |
| 9:00 | Antiferromagnetic Domains and the Spin Flip Transition in Elemental Chromium
<i>Paul Evans, University of Wisconsin–Madison</i> |
| 9:30 | Temperature-Dependent Magnetic Interface Location in Interdiffused NiMn/Ni Epitaxial Bilayers
<i>Christopher Leighton, University of Minnesota</i> |
| 10:00 | Synchrotron Radiation Studies of Magnetism in Layered Nanostructures
<i>Daniel Haskel, Argonne National Laboratory</i> |
| 10:30 | Break |
| 10:50 | Relaxation Properties of Permalloy Nanoparticles and their Effect on Superconductivity
<i>Myron Salamon, University of Illinois at Urbana-Champaign</i> |
| 11:20 | Controlling Ferromagnetic Properties of Ultra-thin $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ Films by Modulation Doping
<i>Jacek Furdyna, University of Notre Dame</i> |
| 11:50 | Lunch |



Afternoon Session

- 1:30 **Magnetic Nanocrystal Superlattices**
Christopher Murray, IBM
- 2:00 **Micromagnetic Simulation of Absorption Spectra**
John Ketterson, Northwestern University
- 2:30 **Break**
- 2:50 **Towards Magnetic Storage Media Using a Single Nanocrystal per Bit**
Franz Himpel, University of Wisconsin–Madison
- 3:20 **Epitaxial Thin Films, Nanopatterning, and Magnetotransport**
R. Alejandra Lukaszew, University of Toledo
- 3:50 **Are the Half Metallic Ferromagnets Half Metallic?**
Peter Dowben, University of Nebraska
- 4:20 **Wrap-up**

WORKSHOP 2 FUTURE DIRECTIONS IN SYNCHROTRON ENVIRONMENTAL SCIENCE

Organized by Steve Sutton,¹ Ken Kemner,² and Shelly Kelly²

¹CARS, The University of Chicago

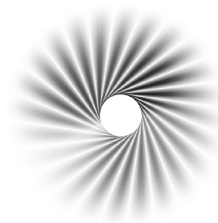
²Environmental Research Division, Argonne National Laboratory

Tuesday, May 4

Building 460 (Argonne Guest House): Conference Room A

Morning	8:45–12:00
Afternoon	1:30–4:45

The purpose of the workshop is to evaluate future directions in environmental science amenable to experimentation with synchrotron radiation. The intent is to focus on frontier science and to evaluate the science-driven requirements for future instrumentation. Although the focus will be on research opportunities at the APS, this will not be a strict limitation. The outcome will be a summary to be submitted to the committee on “Future Scientific Directions for the Advanced Photon Source” chaired by G. Shenoy and S. Sinha. *Abstracts begin on page 26.*



Morning Session

- 8:45 Overview of Synchrotron Environmental Science
Ken Kemner, Argonne National Laboratory
- 9:00 How Do Metal-Reducing Bacteria Deal with Solid Phases?
Jim Frederickson, Pacific Northwest National Laboratory
- 9:30 Surface Complexation Models of Metal Cation Adsorption onto Bacterial Surfaces
Jeremy Fein, University of Notre Dame
- 10:00 Bioremediation of U-Contaminated Subsurface Environments and the
Role of Synchrotron Based X-ray Absorption Measurements
Shelly Kelly, Argonne National Laboratory
- 10:30 Break
- 11:00 Application of Synchrotron-Radiation-Based Techniques to the
Biogenic Oxidation of Manganese
Sam Webb, Stanford University
- 11:30 Environmental Science Using the PNC-CAT Microprobes and Possible Future Directions
Steve Heald, Pacific Northwest National Laboratory
- 12:00 Lunch

Afternoon Session

- 1:30 Mineral-Water Interface Studies
Tom Trainor, University of Alaska
- 2:00 Elemental, Chemical and Structural Characterization of Mineral-Water Interfaces
with X-ray Scattering Techniques
Paul Fenter, Argonne National Laboratory
- 2:30 Correlating Metal Speciation in Soils to Risk
Kirk Scheckel, Environmental Protection Agency–Cincinnati
- 3:00 **Break**
- 3:15 Impact of Redox Disequilibria on Contaminant Transport and Remediation in
Subsurface Systems
Robert Ford, Environmental Protection Agency–Oklahoma
- 3:45 Resolving Biogeochemical Processes of Metals within Physically and Chemically
Heterogeneous Media
Scott Fendorf, Stanford University
- 4:15 Workshop Summary
Paul Bertsch, University of Georgia/SREL

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WORKSHOP 3 THE PROTEIN CRYSTALLOGRAPHY TECHNOLOGY AND LOGISTICS COLLABORATION

Organized by Malcolm Capel,¹ Stephan Ginell,² Lisa Keefe,³ and John Rose⁴

¹NE-CAT, Cornell University

²SBC-CAT, Argonne National Laboratory

³IMCA-CAT

⁴SER-CAT, University of Georgia

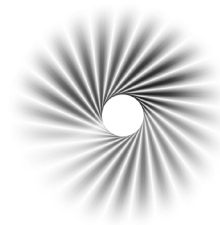
Tuesday, May 4
Building 401: Room A1100

Morning	8:30–12:00
Afternoon	1:00–4:00

The Protein Crystallography Technology and Logistics workshop will focus on the technological advances and goals for operations at the APS macromolecular beam lines. Topics that will be addressed and discussed are:

- Design of a common graphical user interface for users of the macromolecular beamlines.
- Improvement of beam stability at the end-station through application of new white beam position sensing techniques.
- Telepresence and remote data acquisition via network connections.

Abstracts were not available for this workshop.



8:30 Welcome

Development of a Common Graphical User Interface

8:35 Blu-Ice

Tim McPhillips, SLAC, Stanford University

9:05 jdirector

Russ Athay, Rigaku/MSU

9:35 HKL2000 for Data Collection through Structure Solution

Wladek Minor, HKL Research, Inc.

10:05 Refreshments

10:30 Principles of GUI Design

Joseph Yoder, The Refactory, Inc.

11:00 Update from the Protein Crystallography Technical Working Group

Malcolm Capel, NE-CAT

11:30 Discussion

12:00 Lunch

Defining Beam Position

1:00 White Beam Position Monitoring

Gerd Rosenbaum, SER-CAT

1:30 Stability of Delivered Beam

Speaker TBD, Advanced Photon Source

2:00 Access Grid

Ivan Judson, Argonne National Laboratory

2:30 Demonstration of Access Grid at SER-CAT

John Chrzas, SER-CAT

3:00 Refreshments

3:30 Discussion

4:00 Adjourn

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WORKSHOP 4

INELASTIC X-RAY SCATTERING: PRESENT AND FUTURE AT THE APS

Organized by Ercan Alp¹ and John Hill²

¹Experimental Facilities Division, Argonne National Laboratory

²Brookhaven National Laboratory

Tuesday, May 4
Building 362: Auditorium

Morning	8:50–12:40
Afternoon	2:00–5:30

This workshop will focus on the inelastic x-ray scattering work being performed at various beamlines at the APS, including XOR-3, CMC-CAT-9, BESSRC/XOR-11 and BESSRC/XOR-12, GSECARS-13, HP-CAT-16, and UNI-CAT-33. The topics will include collective excitations in high-temperature liquids, phonons in solids at high pressures and temperatures, monitoring the formation of covalent bonds under pressure, phonons in actinides, vibrational study of enzymes, protein model compounds, proteins, and collective electronic excitations in manganites and cuprates. In addition, there will be a session on the near future of IXS at the APS, with a discussion of plans for IXS-CDT, CMC-CAT, HP-CAT, and the proposed actinide elements beamline. *Abstracts begin on page 31.*

8:50 Welcome

Session 1: Current IXS Activities at the APS

9:00 High-resolution IXS in High-temperature Liquids

Harald Sinn, Experimental Facilities Division, Argonne National Laboratory

9:25 Nuclear Resonant Inelastic X-ray Scattering (NRIXS) at the Advanced Photon Source

Wolfgang Sturhahn, Experimental Facilities Division, Argonne National Laboratory

9:50 Magnetic correlations in manganites probed by resonant inelastic x-ray scattering

Stéphane Grenier, Brookhaven National Laboratory

10:15 X-ray Raman Scattering in High-Pressure Research

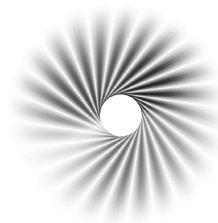
Peter Eng, The University of Chicago

10:40 Break

Session 2: Science Update (I)

11:00 Bonding Charges in Compressed Graphite

Wendy Mao, Carnegie Institution of Washington



-
- 11:20 Formation of sp³ Bonding in BN under Pressure
Yue Meng, Carnegie Institution of Washington
- 11:40 Study of Sound Velocities of the Earth's Core by Nuclear Resonant Inelastic X-ray Scattering
Jung-Fu Lin, Carnegie Institution of Washington
- 12:00 Phonons in Magnetite and VO₂
Robert McQueeney, Los Alamos National Laboratory
- 12:20 Inelastic X-ray Scattering Investigations of Electronic Correlations in Transition Metals and Transition Metal Oxides
Bennett Larson, Oak Ridge National Laboratory
- 12:40 Lunch

Session 3: Science Update (II)

- 2:00 High-Temperature Electronic Instability in Uranium Revealed by Phonons
Michael Manley, Los Alamos National Laboratory
- 2:20 RIXS in Cuprates
M. Zahid Hasan, Princeton University
- 2:40 RIXS in Organic Semiconductors
Clement Burns, Western Michigan University
- 3:00 A New Window into Heme Protein Dynamics: Nuclear Resonance Vibrational Spectroscopy
Stephen Durbin, Purdue University
- 3:20 Vibrational Spectroscopy in Enzymes by NRIXS
Stephen Cramer, University of California, Davis
- 3:40 Break

Session 4: Future Activities at the APS

- 4:00 IXS-CDT and Sector 3: Plans
E. Ercan Alp, Experimental Facilities Division, Argonne National Laboratory
- 4:20 CMC-CAT Plans
John Hill, Brookhaven National Laboratory
- 4:35 Future Prospects of High-Pressure Inelastic X-ray Scattering Spectroscopy at HP-CAT
David Mao, Carnegie Institution of Washington
- 4:50 High-Resolution Inelastic X-ray Scattering Facility for f-Electron Materials Research
Tai-chang Chiang, University of Illinois at Urbana-Champaign
- 5:05 Opportunities for Polarization-Dependent Soft X-ray Resonant Inelastic Scattering at the Advanced Photon Source
John Freeland, Experimental Facilities Division, Argonne National Laboratory

Workshop 5

CHEMICAL AND BIOLOGICAL NANOSCALE MATERIALS

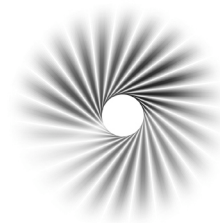
Organized by David Tiede and Andrew Goshe
Chemistry Division, Argonne National Laboratory

Tuesday, May 4

Building 402: Lecture Hall

Morning	9:00–12:00
Afternoon	1:30–5:00

This workshop will explore emergent opportunities for creating new classes of nanocomposite materials that combine the functional properties of inorganic and bio-organic molecular materials. Integration of the normally disparate functionalities of inorganic and bio-organic materials within nanostructured composites holds promise for a wide range of applications in areas that include nano-patterning, signaling, energy transduction, and catalysis. The workshop will highlight advances taking place in the design and realization of supramolecular building blocks with covalent bonded architectures that are tuned for self-directed assembly, molecular recognition, and enhanced chemical reactivity. Biological, bio-mimetic, and chemical design strategies will be examined with a goal of highlighting fundamental molecular mechanisms for function, realization in nanoscale assemblies, successes and challenges for characterization, and opportunities for future exploitation. *Abstracts begin on page 37.*



Morning Session

- 9:00 **Opening Remarks**
David Tiede, Argonne National Laboratory
- 9:15 **Welding Biomolecules to Nanoparticles**
Tijana Rajh, Chemistry Division, Argonne National Laboratory
- 9:45 **Bio-molecules at Interfaces**
Deborah Leckband, University of Illinois at Urbana-Champaign
- 10:15 **Break**
- 10:40 **Design of Nanoscale Architectures and Characterization Using High-Angle X-ray Scattering**
Andrew Goshe, Chemistry Division, Argonne National Laboratory
- 11:00 **Supramolecular Materials Based On High-Valent Metal-Alkylidyne Building Blocks**
Michael Hopkins, University of Chicago
- 11:30 **Foldamer Heterosequences: A Modular Approach to Customizable Molecular Containers**
Jeffrey Moore, University of Illinois at Urbana-Champaign
- 12:00 **Lunch**

Afternoon Session

- 1:30 **Structure and Function in Beta-Peptide Foldamers**
Samuel Gellman, University of Wisconsin–Madison
- 2:00 **Reticular Synthesis of Metal-Organic Frameworks**
Omar Yaghi, University of Michigan
- 2:30 **Functional Supramolecular Assemblies Based on Coordination Chemistry**
Joseph Hupp, Northwestern University
- 3:00 **Break**
- 3:20 **Resolving the Conformational Envelope of DNA in Solution by Wide Angle X-ray Scattering**
Xiaobing Zuo, Argonne National Laboratory
- 3:40 **Solution Phase Structural Analysis of Supramolecular Assemblies by Wide Angle X-ray Scattering**
Jodi O'Donnell, Northwestern University
- 4:00 **Atomic-Scale Structure of Nanoscale Materials by THE Atomic Pair Distribution Function Technique and High-Energy X-ray Diffraction**
Valeri Petkov, Central Michigan University
- 4:20 **Discussion**

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WORKSHOP 6

X-RAY DETECTOR TECHNOLOGIES: PRESENT AND FUTURE DIRECTIONS

Organized by Patricia Fernandez¹ and John Quintana²

¹APS Operations Division, Argonne National Laboratory

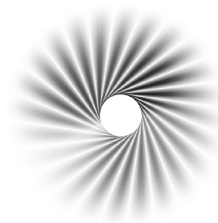
²DND-CAT

Thursday, May 6
Building 402: Lecture Hall

Afternoon 1:30–6:30

The aim of this workshop is to acquaint APS users with x-ray detection technologies and resources available at the APS today, and to present current advances in x-ray detector development that may have an impact on future synchrotron-based research. *Abstracts begin on page 41.*

- 1:30 **APS X-ray Detector Needs: an Issue Covering 15 Orders of Magnitudes**
Jin Wang, Experimental Facilities Division, Argonne National Laboratory
- 2:30 **X-ray Detector and Beamline Instrumentation Development in the APS Operations Division**
Steve Ross, APS Operations Division, Argonne National Laboratory
- 3:00 **New Time-Resolved Detector for Small-Angle X-ray Scattering Studies**
Jan Hessler, Chemistry Division, Argonne National Laboratory
- 3:30 **Break**
- 3:45 **Bent Laue Crystal Analyzers for X-ray Fluorescence Spectroscopy**
Grant Bunker, Illinois Institute of Technology and Quercus X-ray Technologies, LLC
- 4:15 **Protein Crystallographic Detectors of the 21st Century: Where are We Going?**
Edwin Westbrook, Molecular Biology Consortium, Inc.
- 5:00 **Synchrotron Radiation Detectors: Current Status and Where Future Trends Are Taking the UK**
Gareth Derbyshire, CCLRC Rutherford Appleton Laboratory
- 6:00 **Closing Remarks**



WORKSHOP 7

SMALL-ANGLE X-RAY SCATTERING AT THE APS

Organized by David Cookson¹ and Pete Jemian²

¹Australian Synchrotron Research Program and CARS, The University of Chicago

²UNI-CAT, University of Illinois at Urbana-Champaign

Thursday, May 6
Building 401: Room E1100/E1200

Afternoon 2:00–5:20

This workshop will be structured in two segments: (1) Talks highlighting scientific programs currently making use of SAXS capabilities at the APS and (2) A moderated discussion on selected topics of interest to the SAXS community. Attendees will have an opportunity to contribute a presentation of up to five minutes addressing one or more of the following topics:

- Instrumental design enabling simultaneous SAXS and WAXS measurements
- Area detection for small angle scattering at a third generation source
- Liquid sample handling--improving sample throughput and normalization

Please contact the session organizers during the break if you intend to make a presentation during the moderated discussion. *Abstracts begin on page 44.*

2:00	Welcome <i>David Cookson, Australian Synchrotron Research Program and CARS, The University of Chicago</i>
2:05	Overview of APS SAXS Capabilities and Small-Angle Scattering Discussion Group <i>Pete Jemian, UNI-CAT, University of Illinois at Urbana-Champaign</i>
2:20	USAXS Imaging of Materials Deformation <i>Gabrielle Long, X-ray Operation and Research, Argonne National Laboratory</i>
3:00	Time-Resolved X-ray Diffraction Patterns from Indirect Flight Muscles in Living <i>Drosophila</i> during Tethered Flight <i>Tom Irving, Bio-CAT, Illinois Institute of Technology</i>
3:40	Break
4:00	<i>In situ</i> X-ray Scattering: An Ideal Tool to Observe the Fundamental Chemistry and Physics of Particle Nucleation and Growth <i>Jan Hessler, Chemistry Division, Argonne National Laboratory</i>
4:40	Moderated Discussion <i>David Cookson and Pete Jemian</i>
5:20	Adjourn

MAY 3-6, 2004

WORKSHOP 8 ADVANCING ACTINIDE SCIENCE

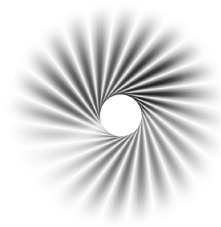
Organized by Lynda Soderholm
Chemistry Division, Argonne National Laboratory

Thursday, May 6
Building 401: Room A1100

Afternoon 1:30–5:00

Actinide science continues to benefit enormously from synchrotron-based techniques. This workshop will highlight recent experiments that offer new insights into long-standing problems. In addition, examples of recent work will be presented that reveal new directions and opportunities for studying structures and speciation in complex systems. Although these examples will pertain to problems in actinide research, their relevance to more mainstream applications will also be addressed. *Abstracts begin on page 46.*

- 1:30 Plutonium Interactions with FeOOH: A Variable Temperature EXAFS Study
Richard E. Wilson University of California, Berkeley, and Lawrence Berkeley National Laboratory
- 2:00 Actinide Redox Speciation through X-ray Absorption Spectroelectrochemistry
Mark R. Antonio, Chemistry Division, Argonne National Laboratory
- 2:30 Mechanisms for the Uranium Accumulation with Aluminosilicate Scale in High-level Waste Evaporators
M.C. Duff, D.B. Hunter, L.N. Oji, W.R. Wilmarth, Westinghouse Savannah River Company
- 3:00 Break
- 3:30 Trace Element Chemistry in Spent Nuclear Fuel Using X-ray Absorption Spectroscopy
Jeffrey Fortner, Chemical Technology Division, Argonne National Laboratory
- 4:00 Aging and Phase Stability in Delta-Stabilized Pu
Luis Morales, A. Lawson, S. Conradson, D. Moore, E. Butler, and M. Ramos, Los Alamos National Laboratory
- 4:30 Chemical Species Formed by Actinide Ions in Solution Identified by Scattering of High-Energy X-rays
Jörg Neuefeind, Argonne National Laboratory



WORKSHOP 1 NANOMAGNETISM

WK 1

Antiferromagnetic Domains and the Spin Flip Transition in Elemental Chromium

P.G. Evans

Dept. Materials Science and Engineering, University of Wisconsin, Madison, Madison, WI 53706-1595

Recent advances in x-ray optics and a new generation of hard x-ray sources have pushed the spatial resolution of diffraction techniques using focused x-ray beams to length scales approaching 100 nm. We have applied these advances to problems in magnetic materials using non-resonant magnetic diffraction as a direct local probe of magnetism. Antiferromagnetic domains in elemental Cr have crucial roles in macroscopically observable mechanical, magnetic, and electronic phenomena and are difficult to observe with conventional experimental techniques. Domains can be distinguished using both the Bragg condition and the anisotropy of the magnetic scattering cross section, which provide complementary information. Using this type of magnetic x-ray microscopy we have imaged these domains with spatial resolution at the micron scale, and connected the domain configuration to the broadening of the magnetic “spin-flip” transition at 120 K.

WK 1

Temperature-Dependent Magnetic Interface Location in Interdiffused NiMn/Ni Epitaxial Bilayers

C. Leighton,¹ M.S. Lund,¹ M.R. Fitzsimmons,² and S. Park²

¹*Chemical Engineering and Materials Science Dept., University of Minnesota, Minneapolis, MN 55455, USA*

²*Lujan Center, Los Alamos National Laboratory, Los Alamos, NM 87545, USA*

Metallic antiferromagnetic (AF) binary alloys are attractive choices for pinning of ferromagnets in applications. However, many of these alloys (as deposited) adopt a chemically disordered paramagnetic phase, which must be annealed to induce a transformation to a chemically ordered AF phase. This annealing induces significant interdiffusion, which reduces the exchange bias. We have deposited epitaxial NiMn (111) / Ni (111) bilayers by MBE and annealed them to examine the effect of interdiffusion on the exchange coupling. Structural characterization by RHEED, x-ray diffraction and reflectometry were combined with conventional magnetometry and polarized neutron reflectometry (PNR). Quantitative analysis of the PNR at 300K and 10K suggests that the effective magnetic thickness of the Ni layers is reduced with decreasing temperature. This remarkable conclusion is supported by magnetometry measurements showing that the magnetization of the Ni layer actually decreases with decreasing temperature, which we believe is due to the increasing dominance of AF interactions in the interdiffused layer. We conclude that the location of the magnetic interface in these systems is temperature dependent due to the competition between AF and F interactions. This competition leads to glassy magnetic phenomena, such as peaks in the zero-field-cooled susceptibility, frequency dependence, memory effects and training. *Work supported by NSF MRSEC.*

MAY 3-6, 2004

WK 1

Synchrotron Radiation Studies of Magnetism in Layered Nanostructures

D. Haskel,¹ G. Srajer,¹ J. Lang,¹ Y. Choi,¹ D.R. Lee,¹ C. Nelson,¹ J. Pollmann,¹ J.S. Jiang,² S.D. Bader,² and J. Meersschaut²

¹Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

²Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

The inherent element specificity of x-ray magnetic circular dichroism (XMCD) and x-ray resonance magnetic scattering (XRMS) techniques continues to enhance our understanding of magnetic phenomena in complex systems with reduced dimensionality. We illustrate the unique advantages of these techniques with a few examples from recent experiments carried out at sector 4 of the Advanced Photon Source. In these examples, circularly polarized x-rays were used to explore interfacial magnetism and spatially inhomogeneous magnetic states in Gd/Fe metallic multilayers and SmCo/Fe spring magnets. *Work at Argonne supported by the U.S. DOE, Office of Basic Energy Sciences, under contract no. W-31-109-Eng-38.*

WK 1

Relaxation Properties of Permalloy Nanoparticles and Their Effect on Superconductivity

M.B. Salamon,¹ Y. Sun,² K. Garnier,¹ and R.S. Averback¹

¹University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA

²Rice University, Houston, TX 77005, USA

We have explored the behavior of monodisperse, 6 nm permalloy nanoparticles produced by gas-phase condensation. The blocking temperature depends strongly on the coverage, indicating that nanoparticle interactions are important. Relaxation studies suggest that the interacting nanoparticles have a hierarchical energy landscape, giving rise to memory effects similar to those of spin glasses. When deposited on Nb, the nanoparticles pin vortices when they are field-cooled below the blocking temperature—but not when allowed to cool in random orientations. The pinning behavior is quantitatively explained by the statistical variation of nanoparticle density on the Nb substrate.

WK 1

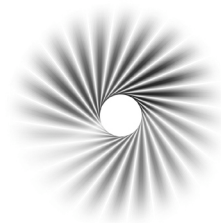
Controlling Ferromagnetic Properties of Ultrathin Ga_{1-x}Mn_xAs Films by Modulation Doping

J.K. Furdyna,¹ X. Liu,¹ and T. Wojtowicz^{1,2}

¹University of Notre Dame, Notre Dame, IN 46556, USA

²Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland

Ferromagnetic (FM) properties of the FM semiconductor Ga_{1-x}Mn_xAs depend on the Mn concentration x , as well as on the concentration of the free holes that mediate the FM interaction between the Mn ions. We illustrate how these properties can be improved by modulation doping (MD) in quantum structures comprised of a 6-nm-thick GaMnAs quantum well (QW) between AlGaAs barriers. When a p-type dopant (Be) is introduced into the barriers, the Curie temperature T_c increases significantly (from about 80K to over 100K), as does the magnetic anisotropy field. This, however, occurs only when the MD is done in the second barrier, deposited *after* the GaMnAs QW is already grown. If one dopes the first barrier (that grown before depositing the GaMnAs layer), T_c actually drops. This indicates that the incorporation of Mn at the Ga sites in GaMnAs is determined primarily by electronic, rather than by



structural, parameters (i.e., by the Fermi level in GaMnAs during the growth). Although the FM properties of GaMnAs can also be improved by annealing, that only applies to *uncapped* GaMnAs. Modulation doping, on the other hand, can be used to improve the FM characteristics of GaMnAs in *heterostructures*, i.e., in geometries most useful for device geometries.

WK 1

Magnetic Nanocrystal Superlattices

C.B. Murray

IBM Corp. T. J. Watson Research Center, Yorktown Heights, NY 10598, USA

Nanometer-scale magnetic nanocrystals with uniform size, controlled crystal shape, structure and surface passivation can now be readily prepared. We combine a high-temperature-solution phase synthesis with size-selective processing techniques to produce organically stabilized, magnetic nanocrystals with size distributions less than 5%. These nanocrystals then form the basis for a combined structural and magnetic study of the evolution of nanocrystal properties with size. I will give a brief introduction to developments in magnetic nanocrystals and in semiconductor dots before focusing on the interesting binary superlattice systems that can be built with these “artificial atoms.” Examples of nanomagnetism will be drawn from work on Co, FePt, Fe₂O₃ and Fe₃O₄ systems. This control has led more recently to the ability to prepare binary nanocrystal superlattices incorporating two different types of particles, for example, semiconductors and magnets. Of particular interest in these binary assemblies will be IV-VI semiconductor building blocks with tunable optical properties in the near IR (e.g., PbSe, PbS, and PbTe). We demonstrate that two sets of monodisperse nanocrystals PbSe and Fe₂O₃ can self-organize during controlled evaporation to produce 2D and 3D superlattices (colloidal crystals, opals) with well-controlled structure. Co-assembly of magnetic nanocrystals and quantum dots into binary superlattices will be described. The resulting systems are found to self-assemble into AB₁₃, AB₂ and AB₅ intermetallic structures under appropriate deposition conditions. The nanocrystals sit on regular close-packed superlattice sites, each separated by a selected organic spacer. The superlattices retain and enhance many of the desirable mesoscopic properties of individual nanocrystals and may permit the first systematic investigation of new collective phenomena.

WK 1

Micromagnetic Simulation of Absorption Spectra

J.B. Ketterson¹ and K. Rivkin²

¹*Northwestern University, Evanston, IL USA, 60201*

²*Dept. Physics and Astronomy, Northwestern University, Evanston, IL USA, 60201*

A new method is developed to numerically calculate the energy absorbed by the various modes excited by a position- and time-dependent external magnetic field in a ferromagnetic body of arbitrary shape in the presence of a uniform static external magnetic field. The method is demonstrated for the case of a two-dimensional disk.

WK 1

Towards Magnetic Storage Media Using a Single Nanocrystal per Bit

F.J. Himpsel

Dept. Physics, University of Wisconsin Madison, Madison, WI 53706, USA

The single-particle-per-bit memory has been the ultimate goal of magnetic data storage for some time. Among the many obstacles to a practical and economical implementation have been irregularities of the magnetic grains and the daunting task of patterning such media at the 10 nanometer level by conventional lithography. This talk discusses the options for producing patterned media via directed self-assembly. Highly perfect magnetic nanocrystals have been synthesized [1], but it has been difficult to assemble them into arrays over macroscopic distances and to orient their anisotropy axes. Synchrotron radiation techniques, such as EUV interference lithography [2, 3] might be able to solve the patterning problem by producing hydrophilic/hydrophobic stripes or circles and selectively attaching the hydrophobic nanocrystals. Near-edge x-ray absorption fine structure (NEXAFS), another synchrotron technique, is able to monitor the bonding between the organic coating of the nanocrystals and the substrate in element- and orbital-specific fashion [4].

[1] Sun, et al., *Science* 287, 1989 (2000).

[2] Kim et al., *Nature* 424, 411 (2003).

[3] Solak and David, *J. Vac. Sci. Technol. B* 21, 2883 (2003).

[4] Pérez-Dieste, et al., *Appl. Phys. Lett.* 83, 5053 (2003).

WK 1

Epitaxial Thin Films, Nanopatterning, and Magnetotransport

R.A. Lukaszew,¹ D. Pearson,¹ and A. Zambano²

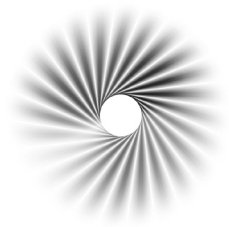
¹*University of Toledo, Toledo, OH 43606, USA*

²*Michigan State University, East Lansing, MI 48824, USA*

The latest results on magnetoresistance in Ni nanocontacts have shown surprising results on excess of several hundred thousands percent [1]. The reported effect has been observed in nanocontacts electrodeposited between Ni wires. There is much controversy surrounding the published data, and therefore, in an attempt to clarify some of the possible processes present, we applied e-beam lithography to epitaxial Ni films. Epitaxial ferromagnetic thin films exhibit narrow domain walls that may favor ballistic transport in a nanocontact as predicted by theory [2]. The magnetic properties were investigated using longitudinal MOKE. Unusually high coercive fields observed along hard axes are due to a second-order-type transition prior reversal with high density of domain walls formation. Thus we expect that a patterned nanostructure with segments parallel to magnetization hard axes will be more likely to experience domain-wall-related effects in magneto-transport. Our preliminary results indicate that magnetic domains do play a role in the magnetoresistance of these nanobridges, but the order of magnitude of the observed effect is considerably smaller than the reported observations for electrochemically prepared nanocontacts.

[1] B. D. Chopra and S. Z. Hua, *Phys. Rev. B.* 66, 020403(R), 2002.

[2] P. Bruno, *Phys. Rev. Lett.* 83, 2425 (1999).



WK 1

Are the Half-metallic Ferromagnets Half Metallic?

P. Dowben

Dept. Physics and Astronomy and Center for Materials Research and Analysis, University of Nebraska, Lincoln, NE 68588, USA

Several classes of materials are currently under investigation as potential high polarization materials. These include the half-metallic systems of the semi-Heusler alloys, the manganese perovskites and the “simpler” oxides such as chromium dioxide and magnetite, but all materials suffer from fundamental limitations. First, the postulated half-metallic systems are probably not half metals at any finite temperature. Second, surfaces, interfaces, and structural inhomogeneities destroy the complete spin polarization of half-metallic systems even at zero temperature. In a strict sense, half-metallic ferromagnetism is limited to zero temperature while magnon and phonon effects lead to reductions in polarization. In this talk I will discuss the influence of such finite temperature effects [1-3].

[1] R. Skomski and P.A. Dowben, *Europhysics Letters* 58 (2002) 544-548.

[2] P.A. Dowben and R. Skomski, *J. Appl. Phys.* 93 (2003) 7948-7950.

[3] P.A. Dowben and R. Skomski, *J. Appl. Phys.* (2004), in press.

WORKSHOP 2

FUTURE DIRECTIONS IN SYNCHROTRON ENVIRONMENTAL SCIENCE

WK 2

How Do Metal-Reducing Bacteria Deal with Solid Phases?

J.K. Fredrickson,¹ A.S. Beliaev,¹ D.A. Elias,¹ A. Dohnalkova,¹ D.W. Kennedy,¹ A.E. Plymale,¹ Y.A. Gorby,¹ M.J. Marshall,¹ J.M. Zachara,¹ and K.H. Nealson²

¹Pacific Northwest National Laboratory, Richland, WA 99352, USA

²University of Southern California, Los Angeles, CA 90089, USA

Shewanella oneidensis MR-1 and other dissimilatory metal-reducing bacteria share the challenge of dealing with products or reactants that are in the solid phase. While these organisms utilize a wide range of more common electron acceptors that are soluble species before and after reduction, they also reduce several insoluble metal species, such as iron or manganese oxides, as well as some soluble species, such as uranium and technetium that precipitate as hydrous oxides upon reduction. Both situations present challenges not dealt with by bacteria that lack these abilities. Recent research has revealed that the microbial reduction of U(VI) and Tc(VII) ions by *S. oneidensis* MR-1 leads to their precipitation as poorly soluble nanoparticles of U(IV) and Tc(IV) hydrous oxides in the periplasm and regions external to the cell. Time-course experiments suggest that the nanoparticles can subsequently be transported to the cell exterior, where insoluble oxides eventually accumulate. A mutant of MR-1 defective in *gspD*, whose gene product is responsible for the OM secretin of the type II secretory pathway, accumulates U and Tc nanoparticles in the periplasm. It is clear that microorganisms have developed a variety of sophisticated mechanisms for dealing with solid phase metals, most of which we know relatively little about.

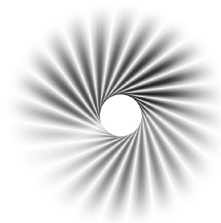
WK 2

Surface Complexation Models of Metal Cation Adsorption onto Bacterial Surfaces

J. Fein

University of Notre Dame, Notre Dame, IN 46556, USA

Bacteria are present in virtually all low-temperature geological settings, and bacterial cell wall adsorption can affect the speciation and fate of a wide range of metals in these environments. Surface complexation modeling offers a means of quantitatively modeling the effect of bacterial metal adsorption, and, in this talk, I will summarize some of the progress made to date in applying surface complexation models to understand bacterial surface adsorption. A number of challenges remain, however, before surface complexation models can be applied to realistic bacteria-bearing systems, and I will discuss a number of these challenges as well. In general, further progress will come from two types of studies: (1) investigations into the molecular-scale mechanisms that underlie bacterial adsorption, and (2) investigations into extrapolation techniques that enable laboratory-based observations of relatively



simple systems to be applied to model the complexity that is present in natural environments. X-ray absorption spectroscopy and other molecular-scale approaches will play an increasingly important role in both types of studies.

WK 2

Bioremediation of U-Contaminated Subsurface Environments and the Role of Synchrotron-based X-ray Absorption Measurements

Shelly Kelly

Argonne National Laboratory, Argonne, IL 60439, USA

Containment of uranium in the subsurface has become the primary focus of remediation efforts. Many containment strategies are based on the link between the valence state of uranium and its solubility. Usually, U(VI) forms soluble complexes in groundwater with high carbonate concentrations, whereas U(IV) forms a uranium-oxide precipitate with very low solubility. Possible mechanisms for reducing uranium in the subsurface include the electron shuttling capabilities of indigenous microorganisms and Fe(II)/Fe(III) oxides. The heterogeneity of environmental systems, including changes in the chemical speciation of uranium, subsurface geochemistry, and hydrodynamics, make the system complex and difficult to predict. Current research is being performed on all length scales, from a seemingly simple one- or two-component system to large-scale field experiments at contaminated sites. X-ray absorption spectroscopy, including both x-ray absorption near edge structure (XANES) and extended x-ray absorption fine structure (EXAFS) spectroscopy have the potential to give critical information regarding the valence state and chemical speciation of U and other trace metals in essentially all current research areas. One of the salient features of XAS is the ability to collect data on pristine samples. However, environmentally relevant samples can have many challenging properties for accurate data collection and interpretation. For example, extremely low trace metal concentrations and non uniformities of concentration and speciation over the sampling area. Experimental methods, including sample series, beamline properties, and data analysis techniques become increasingly important to obtain accurate information from these types of samples. Many of these techniques will be discussed as they apply to environmentally relevant samples containing uranium in a number of different matrices including (1) carbonate solutions with and without calcium, (2) calcium carbonate rock formations, (3) slurries from uranium-contaminated mine sediments, and (4) solutions with microorganisms and humic acids.

WK 2

Application of Synchrotron-Radiation-Based Techniques to the Biogenic Oxidation of Manganese

S.M. Webb

Stanford University, Stanford, CA 94305, USA

Manganese oxides have been shown to be important in many environmental settings as an oxidant of organic matter, as an energy source for microbial metabolism, and as major sources and sinks for heavy metals. Naturally occurring Mn-oxides are believed to be dominantly microbial in origin. Knowledge regarding the nature of biogenic oxides (Mn oxidation state, phase identity(s)) and the mechanisms by

which metals are sequestered therein are fundamental to understanding trace metal cycling in many environments. A variety of synchrotron-based x-ray radiation techniques (WAXS, XAS, μ -XRD, μ -XAS) probe the structure of biogenic manganese oxides produced under various experimental conditions. These studies have shown that the major cation composition has a strong influence on the type of oxide structures observed. Furthermore, the molecular coordination environment of three contaminant metals (Co, Cu, U(VI)) has been examined when the metal is present during Mn oxidation. These results show that each contaminant metal is strongly incorporated into the oxide and displays unique binding properties. The metals also influence the type of oxide ultimately formed

WK 2

Environmental Science Using the PNC-CAT Microprobes and Possible Future Directions

S. Heald

Pacific Northwest National Laboratory, Richland, WA 99352, USA

X-ray microprobes based on Kirkpatrick-Baez mirrors are the most popular facilities at the PNC-CAT beamlines. Microprobes are now available on both the ID and BM lines. On the ID line more than 60% of the time involves microbeams. Most of the work using the microprobes is environmental in nature. In this talk, I summarize the range of applications of the microprobes with some examples concentrating on Hanford site cleanup issues. X-ray fluorescence mapping, micro-XANES, micro-EXAFS, and microdiffraction are combined with electron microscopy to determine the fate of U in sediments collected from below the Hanford tanks. Based on this and other work, it was determined that the uranium phases present could only be explained by considering the microenvironment present within grain fractures. In the second part of the talk, I consider some future directions for improving the performance of the microprobes. These include quick scanning, techniques for easier selection of the beam size, and the ultimate sensitivity possible with improved detectors.

WK 2

Mineral-Water Interface Studies

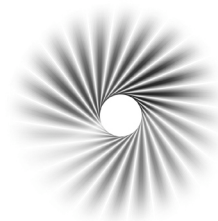
T.P. Trainor,¹ A.S. Templeton,² and P.J. Eng³

¹*University of Alaska Fairbanks, Fairbanks, AK 99775, USA*

²*Scripps Institution of Oceanography, University of California, San Diego, La Jolla, CA 92093, USA*

³*The University of Chicago, Chicago, IL 60637, USA*

Chemical reactions at mineral-water and mineral-microorganism-water interfaces are among the most dominant processes dictating the environmental fate of aqueous compounds. Despite their importance, a detailed molecular scale understanding of environmental interfaces is lacking due to the difficulty in studying heterogeneous systems under realistic chemical and physical conditions. However, the application of synchrotron-based x-ray scattering and spectroscopic techniques provides a novel approach to study chemical and biological processes at the mineral-fluid interface with unprecedented



level of detail under environmental conditions. We will summarize several recent studies utilizing synchrotron-based techniques to investigate molecular and nanoscale structure and reactivity of “model” environmental interface systems. Examples will include the application of *in situ* crystal-truncation rod diffraction to investigate the structure of iron-oxide surfaces and their reactivity with respect to the sorption of aqueous Pb(II); and the application of x-ray reflectivity, long-period x-ray standing waves and grazing angle x-ray absorption spectroscopy to investigate the distribution and speciation of metals at mineral-biofilm-fluid interfaces. The implications for understanding macroscopic reactivity based on these results will be discussed, as well as future scientific directions and required technique developments.

WK 2

Elemental, Chemical and Structural Characterization of Mineral-Water Interfaces with X-ray Scattering Techniques

P. Fenter,¹ Z. Zhang,² C. Park,¹ L. Cheng,¹ J. Regalbuto,³ D. Wesolowski,⁴ M. Machesky,⁵ M. Bedzyk,^{1,2} and N. Sturchio^{1,3}

¹Argonne National Laboratory, Argonne, IL 60439, USA

²Northwestern University, Evanston, IL 60208, USA

³University of Illinois at Chicago, Chicago, IL 60612, USA

⁴Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

⁵Illinois State Water Survey, Champaign, IL 61820, USA

A significant challenge for understanding environmental interfaces is the need to resolve their full complexities, which may include structural, chemical and elemental components. Interfacial chemistry is most widely studied with spectroscopic approaches (e.g., XANES, EXAFS) that are elementally specific and chemically sensitive but typically lack interfacial specificity. X-ray scattering techniques (e.g., crystal truncation rod scattering) have direct structural sensitivity and interfacial specificity, but normally have minimal elemental or chemical sensitivity. I will discuss new opportunities for using advanced scattering techniques that incorporate elemental, chemical and structural sensitivities with surface specificity and Å-scale resolution. Examples will illustrate these capabilities for recent measurements at the mineral-water interface. These include the ability to directly map ion adsorption sites with x-ray standing wave imaging and the use of resonant-anomalous dispersion to provide elemental and chemical sensitivity in probing ion adsorption using surface x-ray scattering. *Supported by the DoE/OBES Division of Chemical Sciences, Geosciences, and Biosciences.*

WK 2

Correlating Metal Speciation in Soils to Risk

K. Scheckel

U.S. EPA, Cincinnati, OH 45224, USA

Understanding the bioavailability of metals from exposure to contaminated soils is a challenging aspect of environmental research. This presentation will highlight three areas of research with respect to metal speciation in soils as it relates to bioavailability: 1) Pb immobilization at a smelter-contaminated site via phosphate amendments, 2) metal speciation and distribution in biosolids amended soils, and 3) thallium hyperaccumulation in *Iberis* spp. The discussion will explore the successes of synchrotron-based research and how the results are advancing new knowledge and research in these areas.

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WK 2

Impact of Redox Disequilibria on Contaminant Transport and Remediation in Subsurface Systems

R.G. Ford and R.T. Wilkin

U. S. Environmental Protection Agency, Ada, OK 74820, USA

Partitioning to mineral surfaces exerts significant control on inorganic contaminant transport in subsurface systems. Remedial technologies for *in situ* treatment of subsurface contamination are frequently designed to optimize the efficiency of contaminant partitioning to solid substrates either through emplacement of reactive solids or manipulation of subsurface chemistry to generate new reactive solid components from parent materials. Current research at the Ground Water and Ecosystems Restoration Division is focused towards developing a process-level understanding of the partitioning reactions that immobilize arsenic and chromium onto natural or engineered subsurface solids. Examples to be discussed include partitioning of arsenic to iron- and sulfur-bearing minerals in a contaminated shallow wetland aquifer system and immobilization of chromium during corrosion of zero valent iron materials installed in subsurface reactive barrier systems. The efficiency and stability of these partitioning processes is governed, in part, by reactions that take place across sharp redox chemical gradients. Coupling system-scale chemical measurements with mineralogical characterization in space and time have been employed to better understand the salient reactions controlling contaminant uptake onto subsurface solids. Synchrotron-based x-ray absorption spectroscopy has recently been employed to better define observed heterogeneity in both trace- and bulk-element oxidation state and structural chemistry.

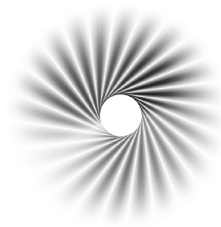
WK 2

Resolving Biogeochemical Processes of Metals within Physically and Chemically Heterogeneous Media

S. Fendorf, B. Kocar, T. Borch, and K. Tufano

Stanford University, Stanford, CA 94305, USA

The fate of metal ions, such as chromium and uranium, are dictated in part by biogeochemical processes within soils and sediments. Owing to both the physical and chemical heterogeneity of such systems, processes controlling the solubility and lability of metal ions will vary even at the submicron scale. As a consequence of the spatial variability, defining operating processes is challenging. Here we explore means to determine the mineralogical and biological heterogeneity relevant to uranium transport within soils and sediments at the micron scale. Using labeled primers specific to classes of bacterial species that metabolize uranium, or other constituents such as iron, in conjunction with x-ray microprobe and micro-XAS analyses, we are able determine the active microbial communities and associated mineralogical constituents that control uranium (and other metals).



WORKSHOP 4

INELASTIC X-RAY SCATTERING: PRESENT AND FUTURE AT THE APS

WK 4

High-Resolution IXS in High-Temperature Liquids

H. Sinn

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

The transport properties of high-melting materials are of interest for a variety of applications, including geosciences, nuclear waste confinement and aerospace technology. While traditional methods of measuring transport properties are often extremely difficult due to the high reactivity of the melts, the combination of containerless levitation and inelastic x-ray scattering offers new insights into the microscopic dynamics of these liquids. Data on the dynamic structure factor of liquid aluminum oxide and liquid boron between 2000-2800 degrees Celsius are discussed and related to several macroscopic quantities like sound velocity, viscosity and diffusion.

WK 4

Nuclear Resonant Inelastic X-ray Scattering (NRIXS) at the Advanced Photon Source

W. Sturhahn

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

In recent years, nuclear resonant scattering techniques that use synchrotron radiation have provided new opportunities for the study of vibrational and magnetic properties of condensed matter under extreme conditions. In particular, the determination of the vibrational density of states with nuclear resonant inelastic x-ray scattering (NRIXS) and the study of magnetic properties with synchrotron Mossbauer spectroscopy (SMS) provided unique results. The basic features of NRIXS will be explained with emphasis on their unique applications in various areas of research. The high brilliance of third-generation synchrotron radiation sources is essential to nuclear resonant scattering techniques because monochromatization to meV levels can be achieved with high spectral flux. The application of NRIXS and SMS also greatly benefits from focusing the x-rays to spot sizes below 10 microns. The measured signal is specific to the nuclear resonant isotope. At the APS ^{57}Fe , ^{119}Sn , ^{161}Dy , ^{151}Eu , and ^{83}Kr have been used. After excitation by a synchrotron radiation pulse, the response of a sample containing the nuclear resonant isotope is delayed in time, a consequence of its narrow line width. Electronic scattering happens almost immediately after excitation and is therefore easily discriminated against. For NRIXS, the time discrimination permits excellent signal-to-noise ratios even for very small amounts of material (<1 ng for iron metal). The small beam sizes available and the minute amounts of material required make these novel nuclear resonant scattering techniques ideally suited for high-pressure measurements using diamond anvil cells, studies of biological materials, and investigations of magnetic nanostructures.

This work is supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

MAY 3-6, 2004

WK 4

Magnetic correlations in manganites probed by resonant inelastic x-ray scattering

S. Grenier

Dept. Physics, Brookhaven National Laboratory, Upton, New York, 11973, USA

We report RIXS studies of a number of manganites with a variety of ground states. Remarkably, we find that in all samples, the excitation spectra show systematic temperature dependencies at energy scales up to 10 eV and further that the integrated spectral weight in the 1 eV to 5 eV energy range scales with the magnetization. This result is reminiscent of, but opposite to, that observed in optical conductivity in the same energy range. On the basis of density functional calculations, we argue that this temperature dependence, which is independent of whether the ground state is insulating or conducting, reflects the temperature dependence of the loss function and that this loss function is sensitive to the nearest neighbor spin-spin correlations.

WK 4

X-ray Raman Scattering in High-pressure Research

P.J. Eng,¹ T.P. Trainor,¹ M. Newville,¹ Y. Meng,² W.L. Mao,^{3,4} H.-k. Mao,^{3,4} C.-c. Kao,⁵ and D.L. Heinz^{3,6}

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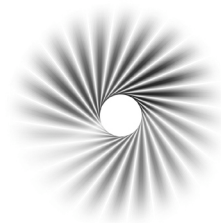
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K-edge absorption spectroscopy of first and second row elements has been limited to soft x-rays and low-energy electrons to date. Due to the high attenuation factors of these probes, such measurements are typically performed in low absorbing environments — samples are contained either in vacuum or at atmospheric pressure with extremely short path lengths, with only the sample's surface region probed. However, through the use of the high-energy inelastic x-ray scattering technique, not only is the bulk accessible but samples can be studied in highly absorbing environments, such as liquid and high-pressure diamond anvil cells. This allows information about chemical bonding to be determined under extreme conditions. We have developed an instrument optimized for inelastic x-ray scattering measurements of samples at high pressure in a diamond anvil cell. To overcome the difficulties of small sample size (typically 100 x 20 microns), high background from the pressure medium, the small inelastic cross section, and the need to probe multiple bond directions, we have combined strong focusing with a multielement analyzer and a multiaxis diffractometer. This combination has allowed us to collect K-edge spectra of compounds containing boron, carbon, nitrogen and oxygen at pressures up to 30 GPa.



WK 4

Study of Sound Velocities of the Earth's Core by Nuclear Resonant Inelastic X-ray Scattering

J.-F. Lin,¹ W. Sturhahn,² J. Zhao,² G. Shen,³ V.V. Struzhkin,¹ Y. Fei,¹ E. Huang,⁴ M.Y. Hu,¹ E.E. Alp,² H.-K. Mao,¹ N. Boctor,¹ and R.J. Hemley¹

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Sound velocities of the Earth's core have been directly measured by seismic wave studies, but experimental measurements of the sound velocities of core materials under core pressures and temperatures are still lacking. Studying the sound velocities of Fe and its alloys under high pressures and temperatures is crucial for geophysical and geochemical models of planetary interiors.

Nuclear resonant inelastic x-ray scattering (NRIXS) has been used to determine the phonon density of states of Fe under high pressures. We have built a double-sided YLF laser heating system to study iron with the NRIXS technique under simultaneously high pressures and high temperatures. Sound velocities of iron have been directly measured up to 58 GPa and 1700K in a laser-heated diamond cell. We found that temperature has a strong effect on the sound velocities; the VP and VS of hcp-Fe decrease significantly with increasing temperature under high pressures. We have also studied the sound velocities and magnetic properties of iron alloys with NRIXS and NFS under high pressures. Addition of Ni slightly decreases the VP and VS of Fe under high pressures. Silicon or S alloyed with Fe increases the VP and VS under high pressures, which provides a better match to seismological data of the Earth's core.

WK 4

Inelastic X-ray Scattering Investigations of Electronic Correlations in Transition Metals and Transition Metal Oxides

B.C. Larson, J.Z. Tischler, P. Zschack, K.D. Finkelstein, A.G. Eguiluz, W. Ku, O. Restrepo, and I. Gurtubay
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The high brilliance of third-generation synchrotron sources has made nonresonant inelastic x-ray scattering (IXS) measurements of the energy-loss spectra in transition metals and transition-metal compounds routine when the ~1 eV energy resolution of the high-heat-load Si monochromator is adequate. We have combined 1 eV energy-resolution IXS measurements on the UNICAT ID-33 beamline with first principles all-electron dynamical electronic response calculations to investigate electronic correlations including crystal local-field and many-body local-field effects in a series of transition metals and transition metal oxides. Direct comparisons of first-principles calculations of the dynamical structure factor with absolute IXS measurements as a function of wave vector and energy loss on Sc, Cr, TiO₂, NiO, and CoO will be used to illustrate the ability to extract spectroscopic information on d-d transitions, local-field effects, novel collective modes, and coulomb repulsion potentials in these materials. The method used for absolute calibration of IXS for arbitrary materials and the removal of quasi-elastic tails will be discussed. *Work at the APS supported by the DOE Office of Science, DMS under contract with ORNL, managed by UT-Battelle, LLC; UNI-CAT is supported by UIUC, ORNL, NIST and UOP Res., Inc.*

MAY 3-6, 2004

WK 4

High-Temperature Electronic Instability in Uranium Revealed by Phonons

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Uranium has three charge-density-wave transitions below 50K, illustrating its potential for electronic instabilities. Our recent phonon results show that electronic instabilities may also occur at higher temperatures. First, neutron measurements on polycrystals show that the interatomic potential landscape of uranium is greatly altered by electronic excitations as the temperature is raised. Observing an apparent shift in a high-energy feature in the phonon DOS, we naturally expected to see phonon frequencies decrease with increasing temperature. Instead IXS measurements show that the structure of the phonon dispersion curves remained fixed, while the scattering intensities shifted between branches. Furthermore, most of the intensity shifts occurred sharply around 473K in the form of an effective depopulation and lifetime broadening of a phonon branch. The phonon must therefore be strongly coupled to another degree of freedom. Earlier work suggests it is electrons, and its discontinuous nature indicates an electronic instability. In light of the phonon results, a technologically significant anomaly in the mechanical behavior coincident with this electronic instability will also be discussed.

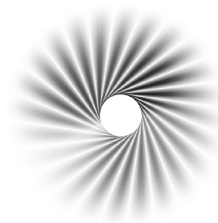
WK 4

A New Window into Heme Protein Dynamics: Nuclear Resonance Vibrational Spectroscopy

S.M. Durbin

Physics Department, Purdue University, West Lafayette, IN

Relatively simple heme proteins like myoglobin, hemoglobin, and various cytochromes have been attractive targets for biophysical investigations of how proteins work because the heme molecule can be probed with resonant Raman spectroscopy and other physical tools, providing clues on how the protein controls the chemistry at the heme Fe atom. Protein structures are now known with exquisite precision, but this has not proved sufficient to understand biological function because structure doesn't automatically tell us how proteins move. We aim to improve understanding of protein function by direct measurement of Fe vibrational spectra, with particular attention to the coupling between the protein backbone and the heme molecule. We utilize the new x-ray synchrotron spectroscopy, nuclear resonance vibrational spectroscopy (NRVS), which provides the complete Fe vibrational density of states. A brief discussion of the basic physics of the process and the unique beamline characteristics will be given, along with results from several systems, including certain heme model compounds, and a comparative study of certain heme proteins.



WK 4

Future Prospects of High-Pressure Inelastic X-ray Scattering Spectroscopy at HP-CAT

H.-K. Mao

HP-CAT and Carnegie Institution of Washington, Washington, DC 20015, USA

Multiple synchrotron x-ray and allied techniques have been developed and integrated at HP-CAT focusing on a unified scientific goal—exploring the rich behavior of materials under extreme pressures and temperatures. A plethora of synchrotron x-ray inelastic spectroscopic techniques has been introduced and applied, many of them for the first time, for high-pressure (HP) applications. Direct probing of the fundamental electron structure and phonon dynamics that were inaccessible for high-pressure research in the past have now been enabled. These include *HP x-ray emission spectroscopy*, which analyzes energies of the x-ray fluorescent photons with sub-eV energy resolution of the emission spectral line shape to provide valuable information on the filled electronic states of the HP samples; *HP x-ray inelastic near-edge spectroscopy*, which opens a wide new field of HP chemical bonding studies of the light elements; *HP electronic inelastic x-ray scattering spectroscopy*, which provides unlimited access to high-energy electronic phenomena, including electronic band structure, Fermi surface, excitons, plasmons, and their dispersions at HP; *HP resonant inelastic x-ray scattering spectroscopy*, which probes shallow core excitations and multiplet structures for highly correlated electronic systems, as well as spin-resolved electronic structures for magnetic samples; and *HP nuclear resonant x-ray spectroscopy*, which reveals phonon densities of state and time-resolved Mössbauer information. The new development is unleashing the full power of high pressure in numerous scientific disciplines, allowing us to tackle a range of grand challenges in various directions. Quests from the early years of the last century, such as the metallization of hydrogen at low temperatures and the mystery of the Earth's inner core, are within reach. Fundamental understanding in electronic structure, from simple electron gas to strongly correlated systems, will be manifested through tuning of the pressure variable. New rules of chemical affinity, reactivity, bonding, and structure will be established across the Periodic Table in each pressure regime. Breakthroughs are expected in applications of high pressure to mineralogy, geophysics, geochemistry, bioscience, as well as specific areas, such as hydrogen storage, stockpile stewardship, and superhard materials.

WK 4

High-Resolution Inelastic X-ray Scattering Facility for f-Electron Materials Research

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²*Lawrence Livermore National Laboratory, Livermore, CA 94551, USA*

A scientific case is made for the development of a fully dedicated high-resolution inelastic x-ray scattering facility at APS for studies of phonons. We are primarily interested in f-electron materials including the 4f and 5f elements, as well as alloys and compounds containing these elements. These systems often exhibit unusual and useful properties that derive from strong electron correlation effects associated with the f electrons. Electronic interactions, including correlation effects, give rise to the novel interatomic forces that govern the thermodynamic and mechanical properties of these materials. Phonon dispersion relations and elastic moduli represent the most sensitive measure of the interatomic

forces, but little data exists to date, even at ambient conditions. The forefront challenge for this project is to extend the experimental capability for making efficient phonon dispersion measurements over a wide range of thermodynamic conditions including extreme conditions of pressure (>GPa), temperature (up to the melting point), and magnetic field (~10 T or higher) for specimens that are typically very small and hard to handle. Recent experimental results on the phonon properties of Pu alloys will be presented and discussed to illustrate the basic scientific issues and the approaches that will be implemented for the investigation.

WK 4

Opportunities for Polarization-Dependent Soft X-ray Resonant Inelastic Scattering at the Advanced Photon Source

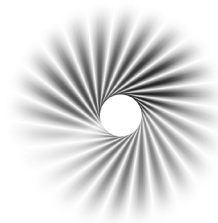
J.W. Freeland,¹ T.A. Calcott,² and D.L. Ederer³

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Resonant inelastic x-ray scattering (RIXS) in the soft x-ray regime offers the ability to map elemental and momentum resolve density of states to better understand the electronic structure of complex materials. Coupling this with the ability to control the polarization of the incident photon beam, as well as the scattering geometry, allows additional selection of spin and orbital contributions. Such information is key to unraveling the role of the interplay of the structural, electronic, orbital and magnetic degrees of freedom in complex oxide systems (e.g., manganites, cuprates, etc.). Here future capabilities and scientific directions for use of RIXS in sector 4 will be presented. *Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, under Contract No. W-31-109-Eng-38.*



Workshop 5

CHEMICAL AND BIOLOGICAL NANOSCALE MATERIALS

WK 5

Welding Biomolecules to Nanoparticles

T. Rajh, J. Liu, Z. Saponjic, N.M. Dimitrijevic, D.M. Tiede, A. Goshe, L. de la Garza, and M.C. Thurnauer
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A new family of semiconductor-based photosynthetic systems that duplicate primary light-induced charge separation mechanisms in natural photosynthesis was created. The synthesis of this new class of functional hybrid materials exploits the finding that the surface of metal oxide nanocrystalline particles is characterized by a highly reactive under-coordinated surface. The coordination sphere of the surface metal atoms is incomplete and exhibits high affinity for oxygen-containing ligands introducing the opportunity for their chemical modification. The shape and size of metal oxide nanoparticles is systematically manipulated in order to control the specific reactivity of facets and metal binding sites. Hybrid nanocomposites were linked to DNA oligonucleotides through bridging enediol ligands, which facilitate hole transfer across the interface of metal oxide nanoparticles. The inherent programmability of oligonucleotides builds recognition properties into the hybrid system allowing selective binding of nanoparticles to targeted molecules. The inorganic nanoparticles are inherently photoresponsive and therefore serve as a source of photogenerated charges, which act as reporters of the electronic properties of the biomolecules. These photoactive bio-inorganic TiO_2 /DA/DNA triads are capable of complex light-induced chemistries, such as light-induced manipulation of biomolecules and their switching functions. As a consequence, light-induced extended charge separation in these systems was found to be a fingerprint of DNA oligonucleotide hybridization and was altered in the presence of mismatches in DNA.

WK 5

Design of Nanoscale Architectures and Characterization Using High-Angle X-ray Scattering

A.J. Goshe

Argonne National Laboratory, Argonne, IL 60439, USA

Methods for the assembly of nanoparticles into spatially well-defined architectures are of interest since properties not found in isolated units emerge from discrete assemblies and arrays of nanoparticles. Supramolecular chemistry acting in concert with defined chemical reactivity at the surface of nanoparticles may provide a new route for organization into precise architectures. Concurrent with the challenges of synthesizing these arrays are the challenges associated with the characterization of the spatial arrangement of organized nanoparticles. We have demonstrated that high-angle x-ray scattering combined with pair distribution function analysis provides a method capable of describing the local structure and long-range order of ordered nanoparticle systems.

WK 5

Foldamer Heterosequences: A Modular Approach to Customizable Molecular Containers

J.S. Moore

University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA

Biological function is largely based on chain molecules that adopt ordered conformations in solution. While ordered solution conformations are commonplace among the biopolymers, only recently have significant strides been made toward folding synthetic chain molecules into unique conformations in solution. This talk will summarize efforts from my laboratory involving m-phenylene ethynylene oligomers that adopt helical conformation in solution, recognize and bind small molecules and subsequently assemble into nanotubular structures in the solid state.

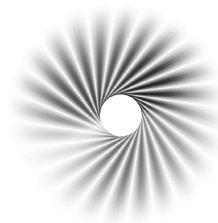
WK 5

Structure and Function in Beta-Peptide Foldamers

S. Gellman

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Proteins perform a wide range of complex functions in biological systems. Nearly all of these molecular operations require the polypeptide chain to adopt a compact and specific folding pattern. We are learning how to induce analogous folding behavior in unnatural oligomers (“foldamers”), with the long-range goal of generating protein-like functions. Much of our effort has focused on beta-amino-acid oligomers (“beta-peptides”). We have found that all three types of secondary structure observed in folded proteins, helix, sheet and turn, are displayed by beta-peptides in organic solvents, when properly selected residues are employed. We have been able to generate beta-peptide secondary structures that are very stable in aqueous solution. Amphiphilic beta-peptides that adopt helical conformations in aqueous solution display potent antimicrobial activity; these beta-peptides are mimics of host-defense peptides, such as the magainins. Other biological functions of interest include disruption of specific protein-protein interactions.



WK 5

Functional Supramolecular Assemblies Based on Coordination Chemistry

J.T. Hupp,¹ J. O'Donnell,¹ K. Splan,¹ A. Massari,¹ J. McGarrah,¹ A. Ramirez,¹ S.J. Lee,¹ R. Sun,¹ S.B. Nguyen,¹ and D. Tiede²

¹Northwestern University, Evanston, IL 60208, USA

²Argonne National Laboratory, Argonne, IL 60439, USA

Metal/ligand directed-assembly techniques can be used to obtain discrete, high-symmetry supramolecular entities featuring well-defined nanoscale cavities. A number of these have been devised at Northwestern based on rhenium coordination chemistry. One demonstrated application is enzyme-like catalysis where isomer-selective rate increases of as much as five orders of magnitude have been obtained in certain cases. In some instances the assemblies have been further elaborated to make functional porous materials. Besides catalysis, appropriately designed materials can: a) mimic natural photosystems and convert sunlight to electrical energy, or b) function as size-selective membranes for molecular transport. A key characterization method is wide-angle x-ray scattering. Of particular interest are solution-phase structures of artificial enzymes intentionally configured in transition-state like conformations.

WK 5

Resolving the Conformational Envelope for DNA Solution with Wide-Angle X-ray Scattering

X. Zuo and D.M. Tiede

Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA

Information on the structure and conformational dynamics of DNA in liquids and other noncrystalline media is critical for understanding its biological and chemical functions. We have begun a program that examines synthetic DNA structure and structural dynamics in solution, and DNA-drug interactions, using synchrotron wide-angle x-ray scattering. Scattering patterns measured to 2 angstrom resolution were found to resolve both nearest neighbor and longer range base-pair distance correlations. Measurements and molecular modeling studies on a series of sequences and temperatures show that scattering peak positions are strongly sensitive to DNA conformation, while configurational dynamics are reflected in peak broadening and attenuation. Significant differences were observed between poly(A)-poly(T) and poly(AT) sequences, and between poly(CG) in low salt and in high salt solution, respectively. X-ray scattering measurements were also done for a few DNA sequences in order to compare solution state conformation with differing structural models obtained from crystallography and NMR methods. In conclusion, wide-angle scattering provides a direct, high-throughput structural approach for quantitative identification of DNA structure and configurational dispersion in functionally relevant solution conditions.

WK 5

Solution Phase Structural Analysis of Supramolecular Assemblies by Wide Angle X-ray Scattering

J.L. O'Donnell,¹ S.-S. Sun,¹ D.W. Tiede,² and J.T. Hupp¹

¹Dept. Chemistry, Northwestern University, Evanston, IL 60208, USA

²Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA

Supramolecular assemblies have been synthesized by joining dipyrindyl ligands with Re(I) coordination chemistry. This synthetic strategy has allowed for the generation of a large library of discrete square, rectangular, triangular and prismatic species. While some of these assemblies have been characterized by x-ray crystallography, crystallization on a large scale for practical applications of these molecules is often difficult or impossible. Many applications can be realized in amorphous or solution phases, but little is known about the configuration and dynamics of these flexible molecules. Wide angle x-ray scattering and pair-distribution analysis of the scattering data are being used to determine the geometries of these molecules in a solution environment.

WK 5

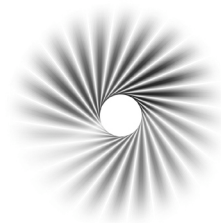
Atomic-Scale Structure of Nanoscale Materials by the Atomic Pair Distribution Function Technique and High-Energy X-ray Diffraction

V. Petkov,¹ S. Shastri,² and Y. Ren²

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²Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA

Knowledge of the atomic-scale structure is an important prerequisite to understand and control the properties of materials. However, materials constructed at the nanoscale do not possess the long-range order and 3D periodicity of conventional crystals and show diffraction patterns with a pronounced diffuse component and few Bragg peaks, if any. This limits the applicability of traditional techniques for structure determination and makes it difficult to obtain precise structural information needed in nanotechnology applications. The challenge can be met by employing the atomic pair distribution function technique (PDF) and high-energy x-ray diffraction. The basics of this non-traditional approach will be introduced and its great potential demonstrated with results from successful studies on vanadium oxide nanotubes, dendritic macromolecules and metallic nanoparticles.



WORKSHOP 6

X-RAY DETECTOR TECHNOLOGIES: PRESENT AND FUTURE DIRECTIONS

WK 6

APS X-ray Detector Needs: An Issue Covering 15 Orders of Magnitudes

J. Wang

Experimental Facilities Division, Argonne National Laboratory, Argonne, IL

With the advent of highly brilliant third-generation synchrotron x-ray sources, such as the Advanced Photon Source (APS), ultrafast structural and dynamical studies on materials using intense x-ray beams become feasible in real time and in a position-sensitive manner. The x-ray probes are used in almost all x-ray techniques involving x-ray imaging (radiography, tomography, phase-contrast imaging), scattering (small-angle x-ray scattering, diffraction) and spectroscopy (extended absorption fine structure, near-edge absorption spectroscopy) of highly transient and dynamic processes including protein folding, laser-matter interactions, and dynamics of polymers and liquids studied by using coherent x-ray beams. It has been recognized that fast, efficient x-ray detectors are one of the most critical technological needs for effective utilization of the intense x-ray beams available at these sources. Unfortunately, despite steady development in this area, there has been the lack of an “ideal” time-resolved, position sensitive and energy-resolved x-ray detector to take full advantage of the extremely intense x-ray beams for a host of experiments. In this presentation, an overview of several important x-ray detector technologies will be briefly reviewed. The time-scale of the applications ranges from sup-picosecond to second. The strengths and the shortcomings of each will be analyzed. To better serve the APS user community, a Detector Pool has been recently established with a mission of supporting users’ general detector needs. An overview of the detector equipment will be presented, from a user point of view, aiming for generating an active discussion on the detector support issues among the vast APS users.

WK 6

X-ray Detector and Beamline Instrumentation Development in the APS Operations Division

Steve Ross

APS Operations Division, Argonne National Laboratory, Argonne, IL 60439, USA

Among other responsibilities, the AOD Experiment Operations Support and Beamline Controls and Data Acquisition groups provide help to APS beamline scientists and users. We report on work on two x-ray detector projects: a low-noise electrometer applied to photodiode x-ray detectors, and fast electronics applied to avalanche photodiode detectors. The first is being used around the APS as a high quality x-ray beam position monitor. The second will aid counting experiments at several sectors by taking advantage of a variety of storage ring fill patterns. We discuss several general purpose electronics modules, including an “EPICS-brick” single board computer to standardize data acquisition and general purpose digital and analog modules. We also mention our future x-ray development plans.

MAY 3-6, 2004

WK 6

New Time-Resolved Detector for Small-Angle X-ray Scattering Studies*

J.P. Hessler,¹ P.M. De Lurgio,² G. Jennings,³ Istvan Naday,² and J. Weizeorick,²

¹Chemistry Division, ²Computing and Instrumentation Solutions Division, and ³Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA

Many new small-angle x-ray scattering (SAXS) studies of nano-sized systems will be possible by improving the time resolution to sub- μ s and beyond. We have designed and are constructing a time-resolved SAXS detector that will have a resolution of ~ 300 ns. Finer time resolution will be possible in a pump-probe mode. From a wafer of $500 \mu\text{m}$ (thick) $\times 15$ cm diameter high-resistivity silicon an annular strip-detector is made. Annular rings on the back of the silicon define the cathodes of the detector. At 12 keV 92% of the X-rays will be absorbed in the silicon. The minimum radial width of the inner rings is designed to be comparable to the size of the incident x-ray beam, $\sim 250 \mu\text{m}$. The outer rings maintain a $\Delta R/R \sim 0.02$, which is comparable to the energy resolution of the incident beam. This produces a detector with only 128 rings or channels. The current from each channel is amplified by a new high-gain high-bandwidth transimpedance preamplifier stage followed by a seven-pole optimized low-pass filter. Each signal is then digitized by a 20 MHz analog-to-digital converter. Up to 72 averaged samples, which form the 12-bit data word, are stored every $3.68 \mu\text{s}$ (the period of the APS). Any combination of these samples may be selected to provide the optimum signal-to-noise and time resolution. Possible experiments and examples from the fields of physics, chemistry, and biology will be presented. *This work and use of the APS have been supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, under contract No. W-31-109-ENG-38.*

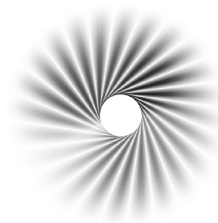
WK 6

Bent Laue Crystal Analyzers for X-ray Fluorescence Spectroscopy

G. Bunker

Illinois Institute of Technology, Chicago, IL 60616, and Quercus X-ray Technologies, LLC, Oak Park, IL 60304, USA

Bent Laue crystal analyzers (BCLAs) are a useful recent addition to the range of detectors available for X-ray Fluorescence Spectroscopy and XAFS. Unlike most conventional energy-resolving detectors, which count each photon and reject those of the wrong energy, the BCLAs block photons that are of the incorrect energy. This greatly reduces or eliminates problems of detector saturation. Other benefits of BCLAs are good collection efficiency and energy resolution on the order of 20-100 eV. In this talk our concept, implementation, performance, benefits, and limitations of BCLAs will be described.



WK 6

Protein Crystallographic Detectors of the 21st Century: Where Are We Going?

E.M. Westbrook

Molecular Biology Consortium Inc., Chicago, IL, USA

Over the past twenty years, x-ray detection technologies for protein crystallographic data collection have been dramatically improved, with each detector advance being based on advances in technology: in electronics and electronic devices, in materials, and in software. Today's "standard," the fiberoptically coupled CCD array, is very good indeed, but it is far from perfect. Emerging new technologies are now out there that we can employ to improve our detectors again, particularly new CCD, CMOS, and silicon pixel-array detector (PAD) technologies. Incorporated into carefully engineered detector systems, these new technologies promise fantastic performance enhancements, in efficiency, dynamic range, size, pixel density, and spatial resolution. *Supported by NIH R01 RR16334 and RR16230, NSF DBI-0116615, and internal MBC funding.*

WK 6

Synchrotron Radiation Detectors: Current Status and Where Future Trends Are Taking the UK

Gareth Derbyshire

CCLRC Rutherford Appleton Laboratory, Chilton, Oxon, UK, OX11 0QX

There is no universal detector solution for synchrotron radiation experiments. This has led to many and varied detector developments. The current state of some of these detector types is described here together with some newer developments on solid state detector systems. This will concentrate particularly on the European and UK perspective. Also the detector development program for the UK's Diamond Synchrotron is also discussed.

WORKSHOP 7

SMALL-ANGLE X-RAY SCATTERING AT THE APS

WK 7

USAXS Imaging of Materials Deformation

G.G. Long¹ and L.E. Levine²

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²National Institute of Standards and Technology, Gaithersburg, MD 20899-4701, USA

When materials are deformed, there is often inhomogeneous deformation microstructure development. Volume-averaged microstructural information from small-angle scattering, while of great value, provides almost no information on how the microstructures are arranged within the sample. In response to the need for both statistical data and positional data, a new transmission X-ray imaging technique using ultra-small-angle X-ray scattering (USAXS) as a contrast mechanism was developed. The technique can be used to provide images at different scattering vectors, where these can highlight different microstructural features within the same sample volume. When used together with USAXS data, the images provide quantitative and qualitative three-dimensional information on the sizes, shapes and spatial arrangements of the scattering objects.

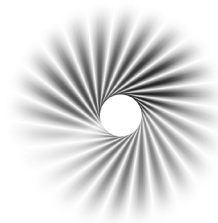
WK 7

Time-Resolved X-ray Diffraction Patterns from Indirect Flight Muscles in Living *Drosophila* during Tethered Flight

T. Irving

Bio-CAT, Dept. BCPS, Illinois Institute of Technology, Chicago, IL 60616, USA

The indirect flight muscles (IFM) of insects provide a particularly well-ordered system for structural studies directed at understanding the molecular mechanisms underlying muscle physiology. The fruit fly, *Drosophila*, with its rich set of tools for genetic manipulation, is a widely used model system for integrative biology. Here I report the results of time-resolved x-ray diffraction experiments on the dorsal longitudinal flight muscles (DLM) of adult flies during tethered flight at the BioCAT beamline at the Advanced Photon Source. Detailed 2D x-ray diffraction patterns were collected at rest and at eight 350 microsecond time frames equally spaced in the ~5 ms wing beat cycle. The results provide convincing evidence for cyclical attachment and detachment of force producing myosin molecules to its binding sites on actin in a living organism. Implications and future directions will be discussed.



WK 7

***In situ* X-ray Scattering: An Ideal Tool to Observe the Fundamental Chemistry and Physics of Particle Nucleation and Growth**

J.P. Hessler, S. Vajda, N. Van Wermeskerken, and R.E. Winans

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Identification of the fundamental chemical and physical processes that induce and control particle nucleation has often eluded researchers. Nucleation has been difficult to study because simultaneous *in situ* observations of multiphase systems are required. Third-generation x-ray sources allow us to perform *in situ* wide-angle, small-angle, and ultra-small-angle x-ray scattering on nucleating systems. Since the signals from x-ray absorption and scattering experiments are line-of-sight and may sample different spatial regions, tomographic inversion is often needed to obtain spatially resolved information. By designing experiments that have cylindrical symmetry, this inversion process may be performed with Abel's inversion transform, which significantly reduces the amount of data needed and the computational requirements. When absolute differential cross sections are measured over a sufficiently large range of reciprocal space, this spatially resolved information may be used to provide a detailed description of the evolution from molecular species to mature particles that may then be used to test the various chemical and physical mechanisms that have been proposed to model particulate nucleation and growth. Examples of the formation of carbonaceous particles in both premixed (minimum influence of fluid dynamics and maximum sensitivity to chemistry) and diffusion flames (controlled by both fluid dynamics and chemistry) will be given along with comparisons to simulations. These examples will demonstrate the need to perform absolute measurements and the detail and limitations of the information that may be obtained from scattering experiments on reacting systems. *This work and use of the APS have been supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, under contract No. W-31-109-ENG-38.*

WORKSHOP 8

ADVANCING ACTINIDE SCIENCE

WK 8

Plutonium Interactions with FeOOH: A Variable Temperature EXAFS Study

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We are currently studying the interactions of actinides with environmentally relevant surfaces. The study of plutonium with the iron-oxide goethite has been studied by batch sorption studies and using x-ray absorption spectroscopy. Plutonium is a chemically complex element from both a solution speciation and redox perspective. The reduction potentials between plutonium's four available oxidation states in aqueous solution are roughly one volt, with all four oxidation states able to coexist in solution. Studying plutonium-mineral surface complexes is complicated by this redox chemistry and any radiation chemistry being performed during the collection of spectra.

We have developed a low-temperature system to freeze wet actinide samples at the APS. By freezing our plutonium-goethite samples to 20 K we can slow or prevent any redox or radiation chemistry from taking place. Freezing the samples also increases data quality by removing thermal disorder from the samples. Batch sorption experiments with Pu(VI) show that plutonium sorbs to the surface of the goethite but do not provide any insight into the reaction mechanisms. Our low-temperature EXAFS studies show that the sorbing surface species is of the originally oxidized Pu(V/VI) form. Experiments in which the samples were allowed to rest at room temperature and then re-frozen show that the oxidized Pu species undergoes a reduction to a Pu(IV) surface complex, suggesting a surface mediated redox reaction.

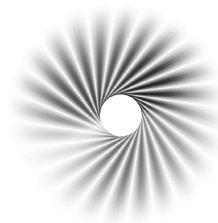
WK 8

Actinide Redox Speciation through X-ray Absorption Spectroelectrochemistry

M.R. Antonio

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We have developed equipment and methods for x-ray absorption spectroelectrochemistry at the APS that are advancing the understanding of the redox reactions of 5f-ions with regard to electronic state structure and electron distribution, as well as speciation and bonding with different ligands, including small, electrochemically inactive ones like water and large, electroactive ones like polyoxometalates. Such information is pivotal to an understanding of basic phenomena in heavy-element sciences involving hydrolysis, disproportionation, complexation, sorption, precipitation, etc., reactions. This talk is intended to highlight selected applications of our spectroelectrochemistry program with examples drawn from U, Np, Pu, and Bk solution experiments, which provide direct insight into redox processes in terms of thermodynamic formal potentials and coordination chemistry. *This work is supported by the U.S. DOE - OBES, Chemical Sciences, under contract W-31-109-ENG-38.*



WK 8

Trace-Element Chemistry in Spent Nuclear Fuel Using X-ray Absorption Spectroscopy

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Knowledge of the chemical states of fission products and transuranium actinides in spent nuclear fuel (SNF) and related materials provides a first line of understanding that may benefit waste disposal in a geologic repository, fuel reprocessing, transmutation/recovery, and development of advanced nuclear fuels. Several x-ray absorption spectroscopy (XAS) measurements from SNF were performed at the Materials Research Collaborative Access Team (MR-CAT) insertion device beamline located at sector 10 of the Advanced Photon Source (APS). The brightness of the APS at high x-ray energy makes it ideal for investigating radionuclide systems, which have relatively high-energy absorption edges and which must be carefully encapsulated for radiological safety. Using a novel “bent-Laue analyzer” (BLA) detection scheme, we have obtained detailed XAS information from trace elements (Pu, Np, Mo, and Tc) in specimens of SNF. The BLA provides unprecedented rejection of the intense uranium fluorescence background, yielding spectral information from elements present at concentrations of 10^{-4} . These data have allowed the first direct observation of oxidation state, coordination environment, and site symmetry of fission product and actinide elements in SNF. *This work was funded by the U.S. Department of Energy (DOE), Office of Civilian Radioactive Waste Management, Yucca Mountain Program, and DOE's Office of Nuclear Energy, Science and Technology, Nuclear Energy Research Initiative, under Contract W-31-109-Eng-38. Work performed at MRCAT is supported, in part, by funding from DOE under grant number DEFG0200ER45811.*

WK 8

Chemical Species Formed by Actinide Ions in Solution Identified by Scattering of High-Energy X-rays

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Actinide ions in aqueous solution do form a large variety of chemically distinct species with its counter ions or as a function of pH. The importance of the environmental chemistry of the actinide elements explains the considerable interest in understanding their complex behavior. Traditionally, information on actinide speciation has been obtained by a combination of optical spectroscopic techniques and more indirect methods like conductivity or pH measurements. High-energy x-ray scattering data are used to determine pair distribution functions (PDFs), which represent ion-ion correlations in solution as a function of distance. Pair distribution functions for individual species permit the determination of the bond distances and coordination numbers in solution. The results are comparable to those obtained from EXAFS experiments, although PDFs provide information to larger coordination distances and coordination numbers can be determined with higher precision. Experiments are presented which combine scattering experiments with spectroscopic information to remove ambiguities that persist in the traditional approach. *This work is supported by the U.S. DOE - OBES, Chemical Sciences, under contract W-31-109-ENG-38.*

2004 USERS



2004 USERS MEETING

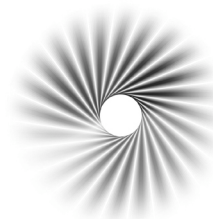
FOR THE ADVANCED PHOTON SOURCE

MEETING

PROGRAM &

POSTER ABSTRACTS

ABSTRACTS

**POSTER S-1****Nuclear Resonant Vibrational Spectroscopy (NRVS) of Iron-Sulfur Cluster Metalloproteins and Model Compounds**

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Iron-containing metalloproteins play key roles in many important biochemical processes. For example, the reduction of dinitrogen to ammonia in biological systems is catalyzed by nitrogenase, which is made up of two unique metal clusters, the P-cluster and the FeMo cofactor [1]. Nuclear resonant vibrational spectroscopy (NRVS) is an important new technique for understanding iron metalloproteins through their vibrational spectra. It is only sensitive to the vibrations of iron atoms, which gives a useful feature of site selectivity [2]. As part of our program to understand biological nitrogen fixation, we have used NRVS to study simple iron-sulfur cluster containing metalloproteins and their model compounds. Measurements were conducted at the XOR-CAT sector 3-ID-D station of the Advanced Photon Source. Detailed Fe vibrational spectra have been obtained for rubredoxin, tetraethylammonium salt, tetraphenylphosphonium salt, $[\text{Fe}_2\text{S}_2\text{Cl}_4]^{2-}$ and $[\text{Fe}_4\text{S}_4(\text{SPh})_4][\text{Bu}_4\text{N}]$. Density of states spectra were extracted from the raw spectra using the PHOENIX program and compared with molecular mechanics calculations. Comparison of measured and simulated spectra shows that Fe-S clusters divide approximately into two main regions: high frequency modes with strong Fe-S stretching motion, and lower frequency bands that are primarily bending modes. The low frequency modes can be further subdivided into simple 3-atom bends, more complex ruffling and twisting motions of the whole cluster, and lattice modes.

[1] O. Einsle, F.A. Tezcan, S.L.A. Andrade, B. Schmid, M. Yoshida, J.B. Howard, D.C. Rees, *Science* **297**, 1696-1700 (2002).

[2] W. Sturhahn, T.S. Toellner, E.E. Alp, X. Zhang, M. Ando, Y. Yoda, S. Kikuta, M. Seto, C.W. Kimball, B. Dabrowski, *Phys. Rev. Lett.* **74**, 3832-3835 (1995).

POSTER S-2**X-ray Crystal Structures of Lipopolysaccharide Biosynthesis Enzymes from *Haemophilus influenzae***

S. Kuruvilla, N. Wetters, A. Chauhan, M. Gajjar, and S. Nair
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The sugar 3-deoxy-D-manno-octulosonate (KDO) is an essential part of lipopolysaccharide (LPS) biosynthesis. Experiments have been used to determine that KDO is required for the maintenance of

LPS. Our focus to this point has been on the enzyme 3-deoxy-D-manno-octulosonate cytidyltransferase (CKS). Cytidine monophosphate (CMP)-KDO is a required substrate in the maintenance of LPS in the outer membrane and overall microbial growth. The CKS helps to convert KDO into CMP-KDO, the required substrate for the next enzyme pathway. The CKS is encoded by the gene *kdsB*. This enzyme has now been crystallized, and an x-ray structure has been determined. This enzyme could be a potential target for the design of inhibitors of this pathway.

Environmental & Geology

POSTER S-3

Spontaneous Magnetostriction in $R_2Fe_{14}B$ ($R=Y, Nd, Gd, Tb, Er$)

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Thermal expansion anomalies of $R_2Fe_{14}B$ ($R=Y, Nd, Gd, Tb, Er$) stoichiometric compounds were studied by x-ray diffraction with high-energy synchrotron radiation using a Debye-Scherrer geometry in temperature range of ~10K to 1000K. Huge and similar invar effects and corresponding large temperature dependence of lattice parameters up to ~10K to 15K above their Curie temperatures (T_c) were observed. The a-axis showed relatively larger invar effects than c-axis in all of the compounds. The spontaneous magnetostrain of the lattice and bonds were calculated. The thermal expansion above T_c is isotropic for $Y_2Fe_{14}B$ but anisotropic for all the other compounds studied. The iron sublattice was shown to dominate the volumetric spontaneous magnetostriction of the compounds and the contribution from the rare-earth sublattice is roughly proportional to the spin magnetic moment of the rare earth. The bonds length change is fully consistent with the theoretical spin density calculation.

Materials Science

POSTER S-4

High-Energy X-ray Diffraction of Al/ Al_2O_3 Composites with 3-D Regular Architecture

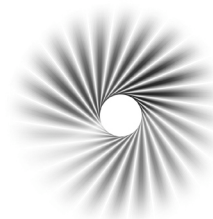
M.L. Young,¹ J.D. Almer,² U. Lienert,² D.R. Haeffner,² R. Rao,³ J.A. Lewis,³ D.C. Dunand¹

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Interpenetrating Al_2O_3/Al composites were created by liquid metal infiltration of alumina structures with three-dimensional periodicity produced by a robotic deposition method. Volume-averaged lattice strains in the alumina phase were measured by synchrotron x-ray diffraction at various uniaxial compression stresses up to 200 MPa. Load transfer was observed to occur from the aluminum to the alumina phase, and the average state of stress in the alumina is close to uniaxial compression. No change in load transfer was observed upon unloading from 200 MPa, indicating that damage (by alumina fracture or interface decohesion) was absent.



POSTER S-5

Small-Angle X-ray Scattering: Binary Fluid Universal Adsorption Surface Scaling Functions

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Local phase separation can occur above the critical temperature of a binary fluid near the free surface. This happens when the surface-tension difference between the two components is large. The component with the lowest surface tension preferentially adsorbs at the liquid-vapor interface. With increasing depth, the composition profile returns to the uniformly mixed state. The variation of the local volume fraction with depth should be governed by a universal function called a surface scaling function. We have used x-ray specular reflectivity to measure the surface composition profile in a binary fluid of dodecane and tetrabromoethane at critical composition. Measurements were made over a wide range of temperatures in order to test the scaling hypothesis. The composition profile extracted from x-ray reflectivity is not in agreement with that predicted from earlier ellipsometry measurements. Instead, there appears to be the formation of a distinct surface monolayer and a separate longer range composition variation. Some preliminary models for the composition profile, as well as comparison with the expectations of scaling theory will be presented.

Environmental & Geology

POSTER S-6

Novel Synchrotron-Based Techniques to Explore the Connection between Metal Speciation in Soils and Plants

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We investigated the speciation of Ni in refinery-contaminated soils and attempted to discern what impact soil metal speciation had on plant metal availability. Using a combination of macroscopic and microscopic techniques including SEM, and micro-x-ray absorption fine structure (XAFS) and x-ray fluorescence (XRF) spectroscopies, it was found that the primary phases present in the soil were NiO and Ni(OH)₂ with mixed metal surface precipitates as minor phases. Similar techniques were applied to ascertain the species and location of Ni in *Alyssum murale*, a known Ni hyperaccumulator grown in the above soils. Micro-XAFS analysis identified Ni-citrate and Ni-histidine as the primary Ni complexes in plant shoots. Fluorescence tomography indicates that there is a concentration of Ni in the epidermis and vascular tissues of the leaf, as well as indicating an association of Ni with Mn at the base of the trichome. The novel application of absorption edge tomography revealed that this association was consistent throughout the entire leaf, as well as indicating an apparent concentration of nickel in the older portions (tip) of the leaf. Understanding how soil metal speciation impacts metal accumulation and

the mechanisms of plant translocation will help improve the efficiency and effectiveness of phytoremediation technologies.

Time-Resolved Studies

POSTER S-7

A Structural Pathway for Signaling in the E46Q mutant of Photoactive Yellow Protein

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Knowledge of the mechanism of action of a protein requires determination not only of its ground-state structure—but also of its intermediate structures and the pathways by which these structures interconvert. In a photoreceptor, such as photoactive yellow protein (PYP), absorption of light by its chromophore leads to a conformational change in the protein associated with differential signaling activity as it executes a reversible photocycle. Time-resolved crystallography allows short snapshots (as low as 150 picoseconds) of high crystallographic resolution ($\sim 1.6 \text{ \AA}$) to be taken of a protein as it functions. Here we analyze a comprehensive time-resolved crystallographic data set comprising thirty time delays of the E46Q mutant of PYP spanning 10 nanoseconds to 100 milliseconds by singular value decomposition. We refine the structures of five distinct intermediates and identify a plausible chemical kinetic mechanism for the photocycle in the crystal. A clear structural progression is visible through these intermediates, allowing the identification of a distinct structural pathway through conserved residues by which a signal generated at the chromophore results in structural changes over 20 \AA away from it.

SAXS

POSTER S-8

***In situ* Studies of Nucleation and Nanoparticle Growth in Flame Aerosol Processes**

N. Agashe,¹ G. Beaucage,¹ H. Kammler,² R. Jossen,² P. Jemian,³ T. Narayan,⁴ and S. Pratsinis²

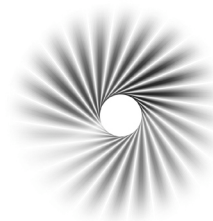
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Ultra-small-angle x-ray scattering (USAXS) is used as an *in situ* technique to characterize titania particles made by a high-temperature flame method. Due to the extremely fast nature of the reaction and low concentrations associated with this process, it is difficult to accurately observe the formation of nuclei and their growth to form aggregated nano-particles by thermophoretic sampling. The high brilliance of synchrotron radiation provides a method to study *in situ* particles at low concentrations. The Bonse-Hart camera of the USAXS instrument at UNICAT (APS, Argonne) can measure a wide range of sizes from nanometer (10^{-9} m) to micrometer (10^{-6} m) and make it possible to simultaneously examine evolution and morphology of these particles. Particle characteristics like the titania volume fraction, primary particle size, polydispersity in this particle size, number concentration, aggregate size and mass



fractal dimension (df.) are presented along the flame axis. The aggregates have complex mass fractal shapes that are joined by partial coalescence, ionic bonds and van der Waals forces.

High Pressure

POSTER S-9

(Mg,Fe)SiO₃ Perovskite to 120 GPa Using Synchrotron Mössbauer Spectroscopy

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Iron-bearing magnesium silicate perovskite, coexisting with small amounts of (Mg,Fe)O ferropericlasite and CaSiO₃ perovskite, is suggested to be the most abundant phase in Earth's lower mantle (670 - 2900 km depth). Knowledge of physical and chemical properties of such phases under conditions approaching those of the lower mantle is therefore essential to our understanding of the deep Earth. The electronic environment of the Fe atoms in two silicate perovskite samples, Fe_{0.05}Mg_{0.95}SiO₃ and Fe_{0.1}Mg_{0.9}SiO₃, have been measured to 120 GPa and 75 GPa, respectively, at room temperature using diamond anvil cells. Such investigations of extremely small and dilute ⁵⁷Fe-bearing samples have become possible through the development of synchrotron Mössbauer spectroscopy. Our results show that, at pressures extending into the lowermost mantle, the fraction of Fe³⁺ remains essentially unchanged, indicating that pressure alone does not alter the valence states of iron in (Mg,Fe)SiO₃ perovskite. The quadrupole splittings of all sites first increase with increasing pressure, which suggests an increasingly distorted (noncubic) local iron environment. Around 70 GPa, a change in the volume dependence of the isomer shift could be indicative of the endpoint of a continuous transition of the Fe³⁺ from a high-spin to a low-spin state.

High Pressure

POSTER S-10

The Equation of State of Al-H-Bearing SiO₂ Stishovite to 60 GPa

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³Institute of Mineralogy, Petrology and Economic Geology, Tohoku University, Sendai 980-8578, Japan

We determined the equation of state of hydrous aluminium-bearing SiO₂ stishovite by x-ray powder diffraction to 60 GPa using synchrotron radiation. Our sample contains 1.8wt% Al₂O₃ and up to 500 ppm H⁺, which is a typical composition for stishovite in subducting slabs. Fitting a 3rd-order Birch-Murnaghan equation of state to our compression data, the isothermal bulk modulus K_T = 281 (±7) GPa with K' = 5.8 (±0.5). When K' is fixed at 4, K_T = 304 (±3) GPa. Our results show that Al³⁺ and H⁺ have a smaller effect on the elastic properties of stishovite than previously claimed. Changes of peak intensities and decreased quality of a tetragonal fit at P = 44(±1.5) GPa may indicate the rutile ↔ CaCl₂

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transition. The absence of a change in cell volume before and after the experiment indicates that stishovite is an agent for transporting water to the lower mantle.

Materials Science

POSTER S-11

EXAFS Examination of the Mn Site in $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ and $\text{Ga}_{1-x-y}\text{Mn}_x\text{Be}_y\text{As}$

A.M. Stuckey, I. DeVasconcelos, X. Liu, T. Wojtowicz, J.K. Furdyna, and B.A. Bunker

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For the last number of years, $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ has been of great interest as it pairs the electrical properties of GaAs with a magnetic spin from Mn. However, the Curie temperature of these materials is below room temperature, and this has led to a large body of work attempting to raise the Curie temperature. Varying the Mn concentration, annealing the material, co-doping with Be and other methods have been tested as methods to raise the Curie temperature. This study examined the local structure about the Mn by x-ray absorption fine-structure spectroscopy in $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ and $\text{Ga}_{1-x-y}\text{Mn}_x\text{Be}_y\text{As}$. The $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ was examined as a function of Mn concentration, and the $\text{Ga}_{1-x-y}\text{Mn}_x\text{Be}_y\text{As}$ was studied at several Be concentrations (while the Mn concentration was held steady). Manganese was found to exist in several local structures and the percentage of Mn in the various local structures has been tied to the concentration of Mn in the $\text{Ga}_{1-x}\text{Mn}_x\text{As}$ materials and to the concentration of Be in the $\text{Ga}_{1-x-y}\text{Mn}_x\text{Be}_y\text{As}$ materials.

Imaging

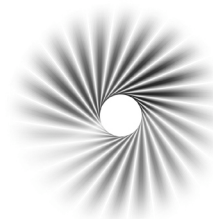
POSTER S-12

Noise Immunity of Iterative Algorithms Used in Coherent Diffractive Imaging

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Coherent x-rays diffracted by a small crystal can be used to image the 3D density of the crystal if the phase information of the diffracted wave can be recovered. Brilliant sources like the Advanced Photon Source provide an x-ray beam that is sufficiently coherent with enough brightness to make possible the measurement of micron-sized crystals by coherent x-ray diffraction (CXD) in a reasonable amount of time. Since the phase information of the diffracted wave is lost in the measurement of intensity, we must rely on computational methods to overcome this “phase problem.” The computational methods commonly employed include the Error Reduction and Hybrid Input/Output algorithms and Elser's Difference Map. In this poster, we will investigate the resilience of the preceding algorithms to the noise present in our data by means of a simulated CXD pattern with various types and degrees of noise. It is found that occasional shot noise is not as detrimental to the reliability of these algorithms as is the presence of bias.



POSTER S-13

3D Mapping of Strain Using Coherent X-Ray Diffraction

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Strain in a crystal can be represented as displacements of the atoms from ideal lattice sites. Any crystalline defects will have associated strain; knowledge of this strain inside a crystal can give insight into interfacial interactions and the deformation history of the crystal. It is therefore desirable to visualize the distribution of strain in a crystal. By measuring the coherent x-ray diffraction (CXD) patterns from individual micron-sized Pb crystals and applying phase-retrieval algorithms with suitable constraints, we have successfully recovered both the full 3D shapes of those crystals and internal strain projected onto the scattering vector with a resolution of better than 200 angstroms. The strain is manifested as a complex real-space density. By measuring CXD patterns from a single crystal as a function of temperature near its melting point, we have been able to observe thermal evolution of the particle's strain and equilibrium crystal shape.

Environmental & Geology

POSTER S-14

X-ray Tomography of Bubbles in Silicate Foams

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The growth of bubbles and exsolution of water from molten rocks is responsible for most volcanic eruptions on Earth. To better understand the mechanisms of volcanic eruptions, we investigated the formation of, and bubble size distributions in foamed glasses using x-ray microtomography. Hydrous glasses were prepared by dissolving water into silicate melts at high temperatures and high pressures followed by quenching; chips of these glasses were heated in our lab at 1 atm to exsolve water and create foams. A few chips were degassed in a furnace on GSECARS beamline BM-13 and observed during vesiculation. All foams were imaged by x-ray microtomography on BM-13. Microtomography data was collected at less than 10 microns spatial resolution and one-quarter degree increments; x-rays were converted into visible light by a YAG phosphor screen and imaged with a CCD camera. Bubble sizes in 3-D were measured from the tomographic data using Blob3D software; bubble sizes in 2-D image slices were measured with ImageJ software. Bubble size distributions follow power laws whose slopes support coalescence as the mechanism for the growth of large bubbles. Some bubble size distributions also agree with theoretical models for random packing of randomly sized spherical objects.

POSTER S-15

Preferential Adsorption of Au Adatoms to the Faulted Half of the Si(111)7x7 Unit Cell

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Surface x-ray diffraction measurements have been used to reveal the structure of physisorbed Au atoms on the Si(111)7x7 reconstructed surface. Experiments were performed using the bending magnet beamline X16A at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. We found that the surface space group symmetry of Si(111)7x7 surface (p6mm) is broken by adsorption of Au atoms at very low coverage (~0.06 ML) at room temperature on Si(111). The translational symmetry remains 7x7. A 3D difference Fourier map gives the real space image of the structural change at the S(111)7x7 surface after Au adsorption. Taking the difference map as a guide to model the Au structure, we were able to fit the data with a model. We found there are nine Au atoms bounded to the faulted half side of the Si(111)7x7 unit cell and none to the unfaulted side. The bonding sites of the Au adatoms are well matched to some of the potential sites predicted earlier using Density Functional Theory (DFT). Extensions of the same structural model also explain changes seen on the fitted (1 0 L) crystal truncation rod for different coverage of Au starting from ~0.06 to ~0.20 ML. Our current results address the question of formation of metallic nanoclusters. Au nanoclusters of this dimension (~ 3 nm) have applications as single-electron devices.

This work is supported by Air Force Office of Scientific Research MURI grant, # F49620-01-1-0336. The X16A beamline is supported by the DOE at UIUC MRL under DEFG02-91ER45439, and NSLS is supported by DOE under DEAC02-98CH10886.

POSTER S-16

X-ray Surface Scattering for the Structural Analysis of Adsorbed Peptides at Hydrated Interfaces

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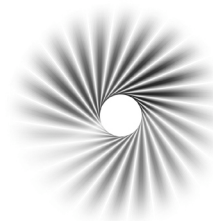
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Protein adsorption onto solid surfaces is a significant process in a wide variety of applications including biomaterials, tissue engineering, biosensors, immunoassays and protein arrays. As soon as a biomaterial is brought into contact with a biological fluid, synthetic and naturally occurring molecular adsorbates alter the surface properties of the biomaterial. Given that protein adsorption takes place well before a cell will interact with the biomaterial offers incentive to study the protein-surface interface. Specifically, we are interested in probing the structural conformation of adsorbed peptides at this aqueous-solid interface on several biomaterial surfaces, including amine-functionalized Si wafers and polymer films. We employ x-ray photoelectron spectroscopy and atomic force microscopy to study the chemistry and structure of the dry surface. The majority of surface-analysis techniques require ultrahigh vacuum



conditions, creating difficulties in examining the peptide in its natural hydrated conformation. The conformation of bromine-labeled peptides adsorbed at liquid-solid interfaces is examined by variable period x-ray standing wave fluorescence spectroscopy (XSW) and x-ray reflectivity. By monitoring the bromine fluorescent yield profile as a function of incident angle provides information about the distance of the bromine layer with an accuracy of several angstroms.

Condensed Matter Physics

POSTER S-17

Resonant Inelastic X-ray Scattering Study of $\text{HgBa}_2\text{CuO}_{4+\delta}$

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The characteristics of the elementary excitations in high-temperature superconductors (HTSC) and their Mott-insulating parent compounds remain a controversial issue after many years of extensive study by different spectroscopic methods. Energy- and momentum-resolved resonant inelastic x-ray scattering (RIXS) is gaining in importance as a powerful tool in the study of elementary charge excitations in HTSC. Here, we report a RIXS study of charge excitations in optimally doped $\text{HgBa}_2\text{CuO}_{4+\delta}$ (Hg1201) ($T_c = 97\text{K}$). Hg1201 is perhaps the structurally simplest of the HTSC, with the highest recorded value of T_c among all single-layer HTSC. We have succeeded in growing sizable high-quality Hg1201 single crystals (up to 10 mm^3 in volume) and begun to map out the charge excitations at the Advanced Photon Source (APS). We are able to resolve several distinct excitation modes in the 2-8 eV energy range and compare our data with results for related materials.

Condensed Matter Physics

POSTER S-18

Quantum Beating Patterns in the Surface Energy of Pb Film Nanostructures

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We have studied the nanoscale structural evolution of Pb films grown at 110K on a Si(111) substrate as they are annealed to increasingly higher temperatures. Surface x-ray diffraction was used to observe the morphology evolve from an initially smooth film through various metastable preferred-height island states before reaching a state of local equilibrium, at which point the coverage of different height Pb structures was analyzed and related to the thickness-dependent surface energy. Rich patterns are seen in the resulting energy landscape similar to the beating patterns heard from the interference of two musical notes of similar pitch. Such patterns are consistent with a calculation of a free-electron gas confined to a quantum box.

MAY 3-6, 2004

POSTER S-19

Coherent GISAXS Investigation of Granular Microstructure in Thin Metal Films

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Coherence is one of the exciting new opportunities available at third-generation synchrotron sources, such as the NSLS-2. We have investigated polycrystalline gold films in the GISAXS geometry using a coherent x-ray beam at sector 34-ID-C of APS [1]. We find a strongly speckled diffraction pattern that shows both an off-specular ridge and doubly off-specular diffuse scattering. Both features are enhanced by the Yoneda effect. The shapes of the speckles are used to report the changing geometry associated with different parts of the pattern. Analytical studies are beginning to show that the origin of the pattern is in the granular microstructure of the film. Annealing shows a dramatic change as the grains of the film start to coarsen.

[1] “Coherent Grazing Exit X-ray Scattering Geometry for Probing the Structure of Thin Films”, F. Pfeiffer, W. Zhang and I. K. Robinson, accepted in Applied Physics Letters (2004)

Condensed Matter Physics

POSTER S-20

Surface of Lead-Free Solder Alloys: X-ray Studies of BiSn

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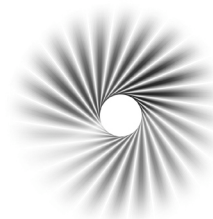
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The widespread use of lead-based soldering materials in the electronic industry is generally believed to lead to a major environmental hazard, and massive efforts are now underway around the world searching for alternatives. A second motivation for studying these materials is to better understand the fundamentals of the competition between the pairing interactions that stabilize the low-temperature liquid eutectics and surface energetics. We present our recent x-ray studies of the surface structure of the low-melting-temperature BiSn eutectic alloy. The resonant x-ray reflectivity technique allows for distinguishing between the two species at the surface. Our preliminary analysis indicates that the reflectivity data are consistent with Bi enrichment of the surface, in accordance with Gibbs absorption rule.



POSTER S-21

Are All Liquids Intrinsically Layered? Surface Studies of Water

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A number of measurements within the last approximately ten years have shown that the free surfaces of liquid metals and alloys are always layered, regardless of composition and surface tension. This was predicted nearly three decades ago and has been supported by other simulations and theory.

Unfortunately, it has not proven practical to experimentally test the absence of layering at the free surface that is characteristic of surface structure simulations of nonmetallic liquids. One open question has been whether or not the layering of liquid metals arises from the fact that their surface tensions (i.e., Hg 500 mN/m, Ga 750 mN/m and In 550 mN/m) are often much larger than for nonmetallic liquids, such as water (73 mN/m). On the other hand, recent measurements show atomic layering at the surface of liquid K (110 mN/m). In order to examine this question, we have extended x-ray reflectivity measurements from the surface of water to the maximum practical wave-vector transfer along the surface normal. We show that there is no observable surface-induced layering in water at T=298K, thus highlighting a fundamental difference between dielectric and metallic liquids. The implications of this result for the question in the title are discussed.

Condensed Matter Physics

POSTER S-22

Surface of Liquid Sn: Anomalous Layering

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Understanding the liquid surface structure of pure metals is important for both fundamental surface science and nanotechnology applications. We present x-ray reflectivity measurements from the liquid-vapor interface of liquid Sn. In addition to a peak in the reflectivity that arises from surface-induced atomic layering that is similar to what has been found for other pure metals (K, Hg, Ga and In), the reflectivity exhibits a low-wave-vector peak feature that has not been found for other pure metallic systems. Analysis indicates that this feature requires an electron density at the surface that is higher than the bulk value. The only electron density model consistent with the data requires that the distance between the first and second atomic layers at the surface is ~10% less than the spacing between subsequent layers. The possibility that this is an artifact due to contamination by other chemical species has been ruled out by a number of different methods.

POSTER S-23

Time-Resolved Crystallographic Studies of the Bacterial Reaction Center

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Light-induced structural changes in the bacterial reaction center were studied by a time-resolved crystallographic experiment. Crystals of protein from *Blastochloris viridis* (formerly *Rhodospseudomonas viridis*) were reconstituted with ubiquinone and analyzed by monochromatic and Laue diffraction, in the dark and 3 ms after illuminating the crystal with a pulsed laser (630 nm, 3 mJ/pulse, 7 ns duration). Refinement of mono-chromatic data shows that ubiquinone binds only in the “proximal” Q_B binding site. No significant structural difference was observed between the light and dark datasets; in particular, no quinone motion was detected. This result differs from crystallographic freeze-trapping of the reaction center from *Rhodobacter sphaeroides*, suggesting that Q_B binds in the “distal” position in the dark and moves to the “proximal” position upon transfer of an electron from the primary donor. Attempts have been made to resolve this discrepancy by obtaining a cryogenic dark dataset for the *B. viridis* reaction center, room temperature dark datasets of the *R. sphaeroides* reaction center using monochromatic and Laue diffraction, and using near infrared microspectrophotometry to gauge the extent and kinetics of photoactivation of in crystals of the *B. viridis* reaction center.

Other

POSTER S-24

Quantitative Vibrational Dynamics of Iron in Nitrosyl Porphyrins

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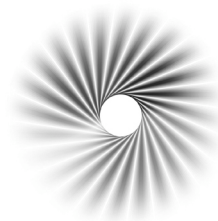
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We use quantitative experimental (nuclear resonance vibrational spectroscopy - NRVS) and theoretical (density functional theory - DFT) approaches to characterize the vibrational dynamics of the ⁵⁷Fe atom in a series of iron nitrosyl porphyrins designed to mimic the active site of heme proteins. NRVS yields the frequencies, amplitudes, and directions of the Fe vibrations, and thus provides a quantitative test of DFT-based normal mode predictions. With the exception of the predicted Fe-NO bond length and frequency for Fe(TPP)(NO), quantitative comparison of calculated and measured Fe dynamics on an absolute scale reveals good agreement. This suggests that DFT can be a reliable guide to the character of observed Fe vibrational modes, including a series of modes involving Fe motion in the plane of the heme, Fe-ligand modes, and reactive modes. In contrast to expectations from a simple three-body oscillator, mode energy remains localized on the FeNO group for only two modes. Bending of the FeNO



unit contributes to several in-plane modes, but no primary FeNO bending mode is identified for Fe(TPP)(NO). Vibrations associated with hindered NO rotation and heme doming are predicted at low frequencies, where Fe motion perpendicular to the heme is experimentally identified at 73 and 128 cm^{-1} .

Condensed Matter Physics

POSTER S-25

X-ray Studies of the Growth of Smooth Ag Films on Ge(111)

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We have performed reflectivity measurements using synchrotron radiation of Ag films deposited on Ge(111) over the thickness range of 3-12 atomic layers. The films deposited at a substrate temperature of 110K are not well ordered but become well ordered upon annealing, as evidenced by substantial changes in the x-ray reflectivity data. The thickness distribution for each annealed film, deduced from a fit to the reflectivity data, is remarkably narrow, with just two or three adjacent discrete thicknesses present, despite the large lattice mismatch between Ag and Ge. In some cases, the film thickness is nearly atomically uniform. The results are discussed in connection with recent models and theories of electronic effects on the growth of ultrathin metal films.

Time-resolved Studies

POSTER S-26

Time-Resolved Crystallographic Studies of the Heme-Based Sensor Protein FixLH

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The protein FixL is a heme-based oxygen sensor found in nitrogen-fixing *Rhizobia*, which is responsible for the regulation of nitrogen-fixation genes in response to molecular oxygen. It is a modular protein composed of two domains; an N-terminal PAS domain, which contains covalently bound heme (FixLH), and a C-terminal histidine kinase domain, whose activity is regulated by the N-terminal sensor domain in response to heme-bound ligand. FixLH is a member of the emerging family of heme-PAS gas sensor proteins, distinct from globins in structure and function. In order to investigate the structural process of ligand recognition in FixLH, we have conducted static and time-resolved crystallographic experiments on the photolabile carbon monoxide complex of FixLH from the organism *Bradyrhizobium japonicum*. Structures of CO-FixLH determined at room temperature reveal a shift of the residues distal to the heme pocket in the H and I beta strands of the protein upon CO binding. Time-resolved experiments and subsequent analysis of time-resolved difference Fourier maps by singular value decomposition (SVD) show relaxation of CO induced change in the molecule to the deoxy state following CO photolysis.

MAY 3-6, 2004

POSTER S-27**Time-Resolved GISAXS Studies of the Bio/Inorganic Interface during Cell-Directed Self-Assembly**H. Baca,¹ E. Branson,¹ C. Ashley,¹ and C.J. Brinker^{1,2}¹Dept. Chemical and Nuclear Engineering, University of New Mexico, Albuquerque, NM 87131, USA²Sandia National Laboratory, Albuquerque, NM. 87185, USA

Templated self-assembly of inorganic mesophases has been well investigated and can be adapted for whole-cell biocompatibility. However, introducing a living cell into an inorganic/surfactant system capable of self-assembly adds both a metabolically active entity and a heterogeneous, dynamically controlled surface to the system. We used evaporation-induced self-assembly (EISA) and biocompatible phospholipid templates to immobilize cells within periodic, porous, silica nanostructures. We find that the interface plays an important role in both promoting cell viability and dictating the structure of the inorganic phase, with the cell actively influencing its microenvironment. We used time-resolved GISAXS and confocal microscopy to follow *in situ* structure development at both the abiotic/biotic interface and in the surrounding silica mesophase. Phospho-cholines, with zwitterionic headgroups and short double hydrocarbon tails allow the predictable development of specific inorganic mesophases, depending on both the structure and concentration of the lipid. In the presence of cells, however, a multilayered, organized interface develops, which determines the organization of the silica mesophase. The living cell's ability to manipulate its surroundings through a well-defined cell-phospholipid-silica interface suggests that cells may serve as tools in the construction of hierarchical architectures that incorporate the directed organization of biomolecules of interest

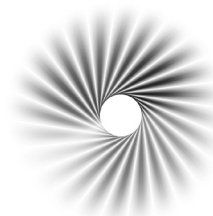
Biology

POSTER S-28**Structural Biology of HU-DNA Complexes**

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Generating sharp bends or kinks in short segments of DNA is an energetically unfavorable process. This large energetic cost must be paid in order to package DNA into cells. The energetic penalty is also exploited to regulate cellular processes, such as replication initiation, site-specific recombination, and transcription, when sharp bends or DNA loops are required for assembly of multicomponent nucleoprotein complexes. Proteins are required to induce/stabilize severe DNA bending, and one of the most abundant bacterial DNA-bending proteins is HU. We have solved HU-DNA cocrystal structures using BioCARS 14BMC and SBC ID19 beamlines and have measured binding affinities of HU with different DNA substrates. The structures and binding data help to pinpoint strain in the DNA and suggest why HU prefers certain DNA structures. HU stabilizes different DNA bend angles ($\sim 105^\circ$; -140°) and is better modeled as a hinge, not a rigid bend as previously thought. Furthermore, the overall bend in the DNA is not planar, which may explain the observation that HU restrains negative supercoils in bacterial chromosomes. The ability to induce/stabilize varying bend angles is consistent with HU's role as an architectural cofactor in various systems that may require differing geometries.

**POSTER S-29****Solution Phase Structural Analysis of Supramolecular Assemblies by Wide-Angle X-ray Scattering**J.L. O'Donnell,¹ S.-S. Sun,¹ D.W. Tiede,² and J.T. Hupp¹¹Dept. Chemistry, Northwestern University, Evanston, IL 60208, USA²Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA

Supramolecular assemblies have been synthesized by joining dipyrindyl ligands with Re(I) coordination chemistry. This synthetic strategy has allowed for the generation of a large library of discrete square, rectangular, triangular and prismatic species. While some of these assemblies have been characterized by x-ray crystallography, crystallization on a large scale for practical applications of these molecules is often difficult or impossible. Many applications can be realized in amorphous or solution phases, but little is known about the configuration and dynamics of these flexible molecules. Wide-angle x-ray scattering and pair-distribution analysis of the scattering data are being used to determine the geometries of these molecules in a solution environment.

Diffraction

POSTER S-30**X-ray Standing Wave Imaging of Ion Adsorption at the Rutile-Aqueous Solution Interface**Z. Zhang,¹ P. Fenter,² N.C. Sturchio,³ M.J. Bedzyk,¹ M.L. Machesky,⁴ and D.J. Wesolowski⁵¹Dept. Materials Science and Engineering, Northwestern University, Evanston, IL 60208, USA²Environmental Research Division, Argonne National Laboratory, Argonne, IL 60439, USA³Dept. Earth and Environmental Sciences University of Illinois at Chicago, Chicago, IL 60612, USA⁴Illinois State Water Survey, Champaign, IL 61820, USA⁵Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

An atomistic understanding of ion adsorption at the rutile (110)-aqueous solution interface is obtained by using the x-ray standing wave (XSW) imaging method. The Sr^{2+} , Zn^{2+} , and Y^{3+} ion distributions are probed with the XSW measurements of a full set of Bragg reflections from the rutile crystal. The XSW data is directly processed with a model-independent Fourier synthesis approach to image the ion distribution. The ion 3D distribution maps reveal that Sr^{2+} and Y^{3+} adsorb in the tetradentate site between the surface terminal oxygen (TO) and bridging oxygen (BO) sites, while Zn^{2+} adsorbs mostly as a monodentate species to the BO sites, with some Zn^{2+} bridging the TO sites. Precise positions of the ion sites (both laterally and vertically) were obtained with XSW triangulation. The measurements of Zn^{2+} ions at different solution conditions, e.g., at different pH and/or solution ionic strengths, yield no significant change of ion distribution at the rutile (110)-aqueous solution interface. The ion adsorption structure obtained from these x-ray measurements is proven to be very important for understanding this interfacial phenomenon, as a representative example of the century-old problem of the electrical double layer (EDL).

POSTER S-31**Ultrafast Strain Propagation in Epitaxial Thin Films**

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We report on the generation and propagation of ultrafast coherent acoustic wave packet in (100) AlGaAs/GaAs heterostructure. Differential thermal expansion in the GaAs substrate is driven by ultrafast laser excitation and generates an elastic response consisting of a unipolar compression wave traveling into both media along [100] directions. Transient changes to the multilayer structure due to coherent acoustic phonons propagation and thermal expansion are measured by picosecond time-resolved x-ray diffraction. This technique is sensitive to the frequency dependent reflection and transmission coefficients of acoustic phonons at the interface and thus should be useful in testing models for the Kapitza resistance.

SAXS

POSTER S-32**Amorphous-Crystalline Transition in Ferritin Solution**

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The protein ferritin from horse spleen displays intriguing behavior when a solution of it is frozen. A new state of aggregation is found to exist, and this state displays a strikingly large thermal compressibility. This state appears more ordered than amorphous yet is not crystalline. Furthermore, once pressure is applied to the system, this state is seen to transform into the well-known FCC crystal structure of ferritin. This transformation is seen to be reversible.

*Materials Science***POSTER S-33****Quantitative Measurements of Piezoelectric Displacement and Polarization Switching in PZT Thin-Film Capacitors by Using Time-Resolved X-ray Microdiffraction**

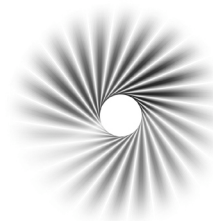
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The piezoelectric response of ferroelectric materials has been widely investigated for applications in fields such as micromechanical actuators, microsensors, and ultrasonic devices. The polarization switching and piezoelectric response to external electric fields are closely linked and can be inhomogeneous on micron and smaller scales. We used time-resolved x-ray microdiffraction with a submicron x-ray beam focused by a Fresnel zone plate to study the fundamental local piezoelectric properties of epitaxial Pb(Zr,Ti)O₃ (PZT) thin-film capacitors. By measuring the time dependence of the PZT (002) Bragg angle during an electrical field pulse, we determined the lattice spacing change and computed the piezoelectric response. The ferroelectric layer was patterned using a focused ion beam



(FIB) to vary the mechanical boundary conditions on different areas of the devices. We have found that the longitudinal piezoelectric coefficient, d_{33} , was 45 pmV^{-1} for the center of the top electrode. The longitudinal piezoelectric displacement can be strongly constrained by either substrate or PZT film around the edge of a capacitor. Near areas where the top electrode and film were removed with the FIB, the piezoelectric coefficient value was larger than near the edge of a shadow-masked electrode.

Polymers

POSTER S-34

Motions of Colloidal Gold Nanoparticles as Studied by X-ray Standing Waves

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Complex nanocomposites of metal nanoparticles dispersed in polymer matrices are of great interest in connection with the fabrication of novel electronic, magnetic and photonic devices. The use of polymers as a matrix material allows increased control of the particle dispersion, while recent advances in colloidal chemistry allow control of the metal particle size. A model metal nanoparticle/polymer system was studied using x-ray standing waves generated by total external reflection above a mirror surface. Colloidal gold nanoparticles, with a diameter of $\sim 15 \text{ \AA}$, were sandwiched between two layers of PVP (poly-2-vinylpyridine). The experiment was able to probe the particle motions *in situ* with subnanometer spatial resolution when annealed above the glass transition temperature of PVP. These studies demonstrate the motion of the nanoparticles on time scales comparable to the relaxation time of the entanglement network.

EXAFS & XAFS

POSTER S-35

Cu K-edge EXAFS Study: CdCl_2 Treatment Effect on CdTe Film

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High-performance CdS/CdTe thin-film solar cells are usually completed with a low-resistance Cu back contact. The copper appears to be critical for achieving heavy *p*-type doping of the CdTe at the contact. It is also known that Cu doping can increase the open-circuit voltage. However, copper is also a fast diffuser, which can accumulate at the CdS/CdTe junction and is suspected of playing a role in cell performance deterioration under certain conditions. High-temperature CdCl_2 treatment is a critical step needed to improve the performance of CdTe thin-film cells. However the process is not yet well understood. Extended x-ray absorption fine structure (EXAFS) is a powerful technique for investigating the short-distance local structure around certain elements in interesting material. In this study, EXAFS on copper-diffused CdTe films with different treatments are studied, particularly the effect of CdCl_2 treatment. We find copper sites in an oxidized environment in CdCl_2 -treated CdTe film, while such sites are bound with tellurium in CdTe film without CdCl_2 treatment.

MAY 3-6, 2004

Other

POSTER S-36

Beam-Splitting Mirror Design

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In order to maximize utilization of a synchrotron radiation beam, an x-ray beam could be split into two by partially inserting a mirror into the incoming beam. The mirror reflects the intercepted beam, while the rest of beam is allowed to continue undisturbed. Such a beamline arrangement is considered for the APS sector 2-ID. A mirror is inserted halfway in a two-collinear-undulator beam to reflect soft x-rays into one branch. The nonintercepted beam continues into another branch where hard x-rays are utilized. The design of the insertion mirror will be discussed. The mirror is side cooled. Several mirror regimes are considered. Two substrate materials are compared. Cooling and mirror geometry are optimized.

Work supported by the U.S. Department of Energy under Contract No. W-31-109-ENG-38.

High Pressure

POSTER S-37

Vibrational Modes in Nanocrystalline Iron at High Pressure

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The phonon density of states (DOS) of nanocrystalline ⁵⁷Fe was measured using nuclear resonant inelastic x-ray scattering (NRIXS) at pressures up to 28 GPa in a diamond anvil cell. The nanocrystalline material exhibited an enhancement in its density of states at low energies, which persisted throughout the entire pressure range. At higher energies, the van Hove singularities observed in both samples were coincident in energy and remained so at all pressures.

Materials Science

POSTER S-38

XANES and EXAFS Study of a Fully Operating Direct Methanol Fuel Cell

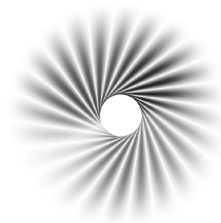
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We report the first *in situ* synchrotron x-ray spectroscopy study of a direct methanol fuel cell. XANES and XAFS data were taken at both Pt and Ru edges of catalyst in transmission mode on the anode side of the fuel cell. The cathode side was prepared using Pd to avoid interference with anode measurements. Multiphase flow at the anode side was controlled by the application of a small back pressure to the fuel



side. XANES data were decomposed by nonlinear squares fitting, and EXAFS data were fit using structural models generated with FEFF. A most striking finding is that the methanol-containing environment maintains the Pt atoms in a nearly metallic state, while the environment of the Ru atoms can only be described well by including a noticeable amount of oxygen near neighbors. Preliminary data also show that the concentration of methanol in the fuel may affect the amount of oxygen near the Ru atoms. These experiments are a first step in addressing the long-standing question of what the state of a DMFC catalyst is within the working potential and environment of a real fuel cell.

Materials Science

POSTER S-39

Application of X-ray Phase-Contrast Imaging and Stereometry to the 3-D Study of Fatigue Cracks

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X-ray phase-contrast imaging is a novel method that offers increased sensitivity compared to methods based on x-ray absorption imaging. In this work, synchrotron phase-contrast radiographs (propagation method) of fatigue cracks in a small, compact tension specimen of aluminum alloy were recorded, and, by taking images at different sample orientations and applying a stereo reconstruction technique, a crack surface map was obtained. Because of the high aspect ratio of the compact tension-specimen geometry, multiple-angle stereometry produces volumetric data with higher resolution and sensitivity than data reconstructed with conventional tomographic methods. *In situ* measurements of the specimen under load were carried out, and changes in the opening at a given position in the crack were also measured and compared with the crack-opening data obtained with absorption tomography.

POSTER 1**Atomic-Scale Structure of Nanoscale Materials by the Atomic Pair Distribution Function Technique and High-Energy X-ray Diffraction**V. Petkov,¹ S. Shastri,² Y. Ren,² and T. Vogt³¹Physics, Central Michigan University, Mt. Pleasant, MI 48858, USA²APS, Argonne National Laboratory, Argonne, IL 60439, USA³Dept. Physics, Brookhaven National Laboratory, Upton, NY 11973, USA

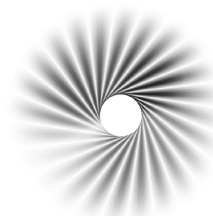
Knowledge of the atomic-scale structure is an important prerequisite to understand and control the properties of materials. However, materials constructed at the nanoscale do not possess the long-range order and 3D periodicity of conventional crystals and show diffraction patterns with a pronounced diffuse component and a few Bragg peaks, if any. This limits the applicability of the traditional techniques for structure determination and makes it difficult to obtain precise structural information needed in nanotechnology applications. The challenge can be met by employing the atomic pair distribution function technique (PDF) and high-energy x-ray diffraction. The basics of this nontraditional approach will be introduced and its great potential demonstrated with results from successful studies on vanadium oxide nanotubes, dendritic macromolecules, nanomagnets and metallic nanoclusters.

*Environmental & Geology***POSTER 2****The Examination of Mercury Interactions with Iron in the Presence of Chemical Agent Simulant Using EXAFS and XANES**L. Skubal,¹ S.G. Biedron,¹ M. Newville,² J.F. Schneider,¹ S. Milton,¹ P. Pianetta,³ and H.J. O'Neill¹¹Argonne National Laboratory, Argonne, IL 60439, USA²Consortium for Advanced Radiation Science, The University of Chicago, Argonne, IL 60439, USA³Stanford Synchrotron Radiation Laboratory, Stanford Linear Accelerator Facility, Menlo Park, CA 94025, USA

Under the Chemical Weapons Convention [1], the U.S. is to destroy its current stockpiles of chemical weapons. Two types of destruction techniques are commonly used: incineration and chemical destruction. Oftentimes, metal catalysts/contaminants such as mercury and arsenic (from synthesis processes) are present in containers holding the agent. Before destruction processes via incineration can commence, contaminant metals must be removed by chemical means to prevent their entry into the atmosphere. To choose an appropriate and successful chemical removal method, the valence state of the metals present in the agent must be known.

The work presented here examines the changes of state that occur in mercury and arsenic as they are introduced into a mustard simulant, thiodiglycol. Metal characterizations were performed using extended x-ray absorption fine structure spectroscopy (EXAFS) and x-ray absorption near edge spectroscopy (XANES).

[1] Technical Secretariat for the Organisation for the Prohibition of Chemical Weapons, Convention on the Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and on their Destruction (1993).

**POSTER 3****Geometric and Spectroscopic Substructures of $\text{Pt}(\text{NH}_3)_4^{2+}$ Adsorbed at the Quartz (100)-Water Interface determined by Resonant Anomalous X-ray Reflectivity**C. Park,¹ P.A. Fenter,¹ N.C. Sturchio,² and J.R. Regalbuto³¹*Environmental Research Division, Argonne National Laboratory, Argonne, IL 60439, USA*²*Dept. Earth and Environmental Sciences, University of Illinois at Chicago, Chicago, IL 60607, USA*³*Dept. Chemical Engineering, University of Illinois at Chicago, Chicago, IL 60607, USA*

Adsorption of the aqueous platinum-tetraammine complex ($\text{Pt}(\text{NH}_3)_4^{2+}$, or PTA) on silica surfaces at high pH is a primary step in noble metal catalyst impregnation. Previous studies have found that this process is electrostatically driven and appears comparable to the “outer-sphere” adsorption of many geochemically relevant species. However, the physical structure and the spectroscopic properties of the adsorbed PTA species on a well-defined homogenous oxide surface have never been directly probed. High-resolution (nonresonant) specular x-ray reflectivity revealed changes to the total interfacial structure due to adsorption of PTA (at pH~10, 200 ppm PTA concentration), but the Pt subprofile is not readily resolved from these data due to the small Pt occupancy, which is obscured by the electron density profile of the interfacial water. To resolve the Pt-specific interfacial profile, resonant anomalous x-ray reflectivity (RAXR) as a function of incident photon energy near the Pt LIII edge was measured at fixed momentum transfer values. Since RAXR uses the elastic coherent scattering channel, it reveals element- and interface-specific information about the physical and spectroscopic properties of the adsorbed species. The RAXR results reveal an unexpectedly complex adsorbate geometric structure, which consists of two discrete PTA layers above the quartz (100) surface. The same data simultaneously reveal the spectroscopic structure of the adsorbed species (i.e., the x-ray absorption spectra of the interfacial PTA) and show a substantial white-line enhancement with respect to that of the solution species. We expect this approach will be valuable as a general approach to understanding complex adsorption phenomena at aqueous-mineral interfaces.

*Polymers***POSTER 4****Depth Profiling of Density Fluctuations within a Polymer Film Using Diffuse X-ray Scattering**

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The properties of polymer thin films, such as glass transition temperature, can differ from those of the bulk due to interfacial effects, such as free ends at the surface layer and confinement of the chain conformation at the polymer-substrate interface. While the surface structure and dynamics of polymer films have been studied, little information about the interior of the film is available. In the present study, we have used grazing incidence x-ray illumination to measure diffuse scattering from the density fluctuation inside a polystyrene thin film supported on Si substrate. At wave vectors small relative to the inverse of the intermonomer spacing, this diffuse scattering is independent of wave vector and proportional to the isothermal compressibility of the film. At angles below the critical angle, the diffuse scattering is dominated by the top layer, which corresponds to the penetration depth of the evanescent

wave. Above the critical angle, the diffuse scattering is mainly from the interior layer where a standing wave is formed. The measurements were performed as a function of incident angle and temperature. Consequently, we are able to investigate the depth and temperature dependence of the compressibility of the film.

SAXS

POSTER 5

Influence of Heavy Metal Elements and Thermal Treatment on Nanophase Growth in Vitreous Silica

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Conventional and anomalous small-angle x-ray scattering (ASAXS) studies have been used to investigate some of the factors that influence formation of heavy metal phosphate nanophase inclusions in vitreous silica at temperatures as high as 1200°C. ASAXS studies near the L3 edges of most lanthanides as trivalent ions and selected higher valent actinide ions provided definitive evidence, in the case of the 4f elements, that the observed nanophases contain heavy metal ions. X-ray scattering studies on similarly prepared actinide oxide nanophase samples provide a basis for interpreting both our present ASAXS data, as well as our past small-angle neutron-scattering data (recorded at IPNS), from samples that contained actinide phosphate phases embedded in vitreous silica. Powder x-ray diffraction and laser-induced fluorescence studies (where appropriate) provided additional probes of the local and extended structure surrounding the embedded heavy metal ions. Both thermal treatment and the nature of the heavy metal were found to strongly influence the size of the observed nanophases and the structure of such nanophases. *Work, including use of the APS, supported by the U. S. Department of Energy, Office of Science, Basic Energy Sciences under Contract W-31-109-ENG-38.*

Other

POSTER 6

Double K-Shell Photoionization of Neon

S.H. Southworth,¹ E.P. Kanter,¹ B. Kraessig,¹ L. Young,¹ G.B. Armen,² J.C. Levin,² D.L. Ederer,³ and M.H. Chen⁴

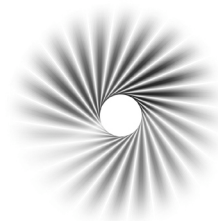
¹Argonne National Laboratory, Argonne, IL 60439, USA

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In the photoionization process, the photon interacts with a single electron, so double photoionization is a manifestation of electron-electron interactions. Theoretical treatments include electron correlation in the ground state, “shakeoff” of the second electron due to relaxation of the ionic wave function, and “knockout” of the second electron by collision with the photoexcited electron. Double K-shell photoionization as a function of atomic number Z is particularly interesting for experimental and theoretical studies. In the high-energy limit, the double-to-single K-shell photoionization cross-section ratio R can be accurately calculated for helium and helium-like ions, because the knockout interaction is



negligible and only shakeoff with ground-state correlation is needed. Accurate K-shell ground-state wave functions can be constructed, and the Z dependence of R can be calculated. At lower energies, R depends on both shakeoff and knockout, but the knockout interaction is more difficult to calculate. The K-shell electrons of neutral atoms are localized near the nucleus, and their interactions with outer electrons can be neglected to first approximation. Double K-shell photoionization measurements on high-Z atoms can therefore be compared with calculations on helium-like ions of the same Z. We used 5000 eV x rays on beamline 12-ID to study double photoionization of neon (Z=10) by recording Auger-electron spectra. The Auger-electron spectrum is dominated by strong diagram lines produced from single-K vacancies, but “hypersatellite” lines were also recorded due to double-K vacancies. The Auger spectrum is complicated by contributions from several multivacancy states, so shake calculations and Auger calculations were performed to help interpret the spectrum. The relative intensities of hypersatellite and diagram lines were used to determine $R = 0.32(4)\%$ for neon at 5000 eV. This ratio is roughly four times larger than the calculated high-energy-limit ratio. We conclude that the knockout interaction is strong compared with shakeoff for neon at this energy.

Chemistry

POSTER 7

Actinide Chemistry at the APS

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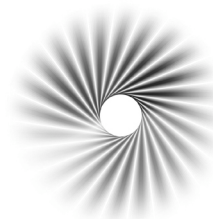
The Actinide Facility is an infrastructure that couples the sample preparation and handling capabilities of ANL's Chemistry Division with the third-generation synchrotron, the APS, located at the same site. This facility is focused on two objectives: (1) to promote, and assist with, synchrotron experiments involving the 5f-elements at any of the beamlines at the APS, and (2) to design, develop, and demonstrate new synchrotron experiments that can address fundamental questions of interest to the actinide community. The first objective is realized through the availability of fully HEPA-filtered hot laboratory space for prepackaged handling of radioactive samples. Also available are safety protocols for working with radioactive samples on the APS floor. The second objective is realized through the specific development of spectroelectrochemistry, small-angle x-ray scattering (SAXS) and high-energy (>60 keV) scattering experiments for problems germane to the actinide community. Some recent examples of technique development will be presented, including formal potential determination and electron distributions in uranyl solutions. *This work is supported by the U.S. DOE, BES, under contract W-31-109-ENG-38.*

POSTER 8**Photoexcited Local Structures of Metal Complex Building Blocks for Nanoscale Supermolecules Probed by X-ray Pulses from the APS**L.X. Chen,¹ G.B. Shaw,¹ B. Cohen,² M.D. Hopkins,² L. Yu,³ J.S. Lindsey,³ D. Holten⁴¹Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA²Dept. Chemistry, The University of Chicago, Chicago, IL 60637, USA³Dept. Chemistry, North Carolina State University, Raleigh, NC 27695, USA⁴Dept. Chemistry, Washington University, St. Louis, MI 63130, USA

Metal complexes have been used as building blocks of nanoscale supermolecules. These metal complexes not only serve as joint points to integrate building blocks into nanoscale supermolecules but also function as light-harvesting chromophores and electron donors/acceptors. These functions are intimately connected to the structures of the excited states. We will present our recent work on determination of two photoexcited metal complex structures using the laser-pulse pump, x-ray-pulse probe XAFS developed in our facility at the APS. A tungsten complex, ligated by four phosphorous ligands and containing a triple bond with a carbon atom, is an electron donor when it is photoexcited to an excited state. Within the time resolution limited by the synchrotron x-ray pulse, i.e. 30-100 ps, we have observed bond elongation due to the metal-to-ligand charge transfer induced by light. The excited state structure of a Zn complex with 2 ns lifetime has been determined, which undergoes structural rearrangement after the light excitation. The local structural changes determine the branching ratio for this molecule to convert light energy to luminescence and to heat. Such structural information is valuable in the rational design of the nanostructured materials with targeted functions.

*Time-resolved Studies***POSTER 9****Exciplex Formation and Structure of Photoexcited Copper(II) Octaethylporphyrin Revealed by Time-Resolved XAS**G.B. Shaw,¹ L.X. Chen,¹ T. Liu,¹ G. Jennings,² and K. Attenkofer²¹Chemistry Division, Argonne National Laboratory, Argonne IL, 60439, USA²Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA

The triplet excited structures of Cu(II) octaethylporphyrin (CuOEP) in toluene and in 1:1 mixture of toluene and tetrahydrofuran (THF) were investigated with 100 ps resolution by time-domain laser-pulse pump, x-ray-pulse probe x-ray absorption spectroscopy (pump-probe XAS) at room temperature using x-rays from the Advanced Photon Source. Transient optical absorption measurements indicated a strong solvent dependency of the triplet-excited-state lifetime due to the presence of oxygen-containing solvent molecules. While the ground state CuOEP molecules remain square-planar in both solvents, the attenuation of a peak attributed to the $1s \rightarrow 4p_z$ transition at the Cu K edge for the laser-excited CuOEP in the THF/toluene mixture revealed the penta-coordinated exciplex formation, which is responsible for the shortening of the triplet-excited-state lifetime. Meanwhile, the average Cu-N distance in the triplet excited state is lengthened by 0.035 Å due to ligation with a THF solvent molecule, which agrees with a domed coordination structure for copper in the penta-coordinated exciplex. *Work at ANL was supported by the U.S. Dept of Energy, Office of Basic Energy Sciences, Division of Chemical Sciences, under contract W-31-109-Eng-38.*



POSTER 10

EPICS Support for the Turbo PMAC2 Motion Controller

O. Makarov and S. Stepanov

GM/CA-CAT at the APS, Argonne National Laboratory, Argonne, IL 60439, USA

Motion control is the key component of any beamline control system and greatly affects the capabilities of beamline automation and throughput. We have developed EPICS support for one of the most advanced controllers on the market—the Turbo PMAC2-VME Ultralight motion controller by Delta Tau. The predecessor of this controller called PMAC1 was successfully used at the SBC and BioCAT lines. Its main advantage was the capability to synchronize on-the-fly motion of several drives thus allowing for fast on-the-fly scans of motor assemblies. The controller was however limited to 8 axes and servo drives only. The new Turbo PMAC2-VME Ultralight motion controller has a modular design, which allows it to drive virtually any type of motor and control up to 32 axes. We have derived our EPICS driver for Turbo PMAC2 from the PMAC1 prototype; it was refined and ported to EPICS-3.14. The databases also derived from PMAC1 have been simplified and reduced in size by the factor of 10 as compared to that at SBC. This resulted in an easily maintainable and configurable control system. It is important to point out that the user interface part of the databases was essentially preserved, and the capabilities were extended, for example, speed controls were added.

Data Analysis

POSTER 11

80,000 X-ray Experiments: Can the APS Beat This?

S. Stepanov

GM/CA-CAT at the APS, Argonne National Laboratory, Argonne, IL 60439, USA

The poster presents a status report on the X-ray Server that has been operational at the APS since 1997 and performed around 80,000 numerical x-ray experiments. X-ray Server is a noncommercial project not supported by any grants. The goal of this project is to explore novel technologies for establishing scientific collaborations, sharing personal research results, and refining scientific software. The server provides web-based access to a number of programs developed by the author for simulating x-ray diffraction and scattering. The software code is not shared but operates directly on the Server. This philosophy brings numerous benefits to both the developer and the users. Currently seven programs are operational. They provide the calculations of (1) the Bragg curves from strained crystals and multilayers, (2) the specular reflectivity curves from multilayers with interface roughness, (3) the diffuse scattering patterns from correlated interface roughness in multilayers, (4) the resonant x-ray specular reflection from magnetic multilayers, (5) the multiple Bragg diffraction of x-rays in arbitrary diffraction geometries, (6) the dispersion corrections and scattering factors for various materials in the x-ray range, and (7) the orientations of the Bragg planes in crystals satisfying given conditions. Programs (4) and (5) were added in the last year and more additions are on the way.

POSTER 12

Going Closer to the Beam Center at SAXS Measurements by Damping Slit Scattering

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*2*GM/CA-CAT at the APS, Argonne National Laboratory, Argonne, IL 60439, USA

Measuring SAXS in close vicinity to the direct beam is often complicated by low useful signal as compared to that from the scattering of beamline elements, especially the collimator slits. This is often the case for studying biological macromolecules in solutions that provide low scattering. On the other hand, undulator beamlines with focused beams are characterized by excessive intensity for static SAXS measurements. It is possible to install some additional x-ray optics into the standard SAXS setup that can suppress the slit scattering at the expense of a small reduction in the direct beam intensity. The first choice solution for improving the beam shape and reducing the intensity of its wings would be multiple diffraction of the beam inside a channel-cut crystal. However, in the case of a focused beam, it is not trivial and requires some examination of the intensity and focusing parameters behavior. We have shown that a two-bounce Si (111) crystal installed onto the focused x-ray beam behind the collimator slits in the standard SAXS setup damps the beam wings by ~ 70 times relatively to the peak. At that the beam intensity reduces by ~ 7 times and the focusing parameters are preserved. *The work was supported by NIH grant RR08630.*

Chemistry

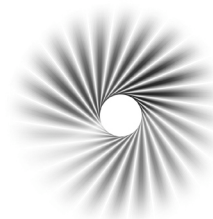
POSTER 13

Oxidation States of Pt in Ceria-Supported Pt Water-Gas Shift Catalysts

M.J. Castagnola S.Y. Choung, C.C. Rossignol, and T.R. Krause

Chemical Engineering Division, Argonne National Laboratory, Argonne, IL 60439, USA

Ceria-supported platinum has received considerable interest as a catalyst for the water-gas shift ($\text{CO} + \text{H}_2\text{O} \leftrightarrow \text{CO}_2 + \text{H}_2$) reaction. The changes in the oxidation state of platinum supported on a gadolinium-doped ceria ($\text{Pt}/\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9}$, Pt/CGO) and a zirconia-doped ceria ($\text{Pt}/\text{Ce}_{0.8}\text{Zr}_{0.2}\text{O}_2$, Pt/CZO) during temperature-programmed reduction were monitored by *in situ* x-ray absorption spectroscopy. Using principal component analysis of the Pt L_3 edge in the XANES region of the spectra in combination with fitting of the EXAFS region revealed that Pt(IV) oxide was initially present on both doped-cerias, and that the Pt(IV) was reduced to Pt(II) before being completely reduced to Pt(0). Pt/CGO underwent reduction from Pt(IV) to Pt(II) at a lower temperature (100°C) than Pt/CZO (150°C). In the case of Pt/CGO, the reduction of Pt(IV) to Pt(II) occurred at a lower temperature (50°C) if the sample was reduced in hydrogen and then reoxidized after the initial calcination. This effect was not observed with Pt/CZO. Analysis of the Ce L_3 edge of a sample of Pt/CGO showed that only a small fraction ($<5\%$) of cerium was reduced from Ce(IV) to Ce(III) at 250°C , with no further reduction observed below 500°C , which suggested that only cerium located in the surface layers was reduced.

**POSTER 14****Synchrotron X-ray Characterizations of Styrene-Butadiene-Styrene Tri-Block Copolymers Modified with Polyhedral Oligomeric Silsesquioxane**

A. Lee

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Tri-block copolymers of styrene-butadiene-styrene (SBS) grafted with varying amount of polyhedral oligomeric silsesquioxane (POSS) were synthesized using the hydrosilation method. The POSS molecule was designed to contain a single silane function group, which was used to graft onto the dangling 1,2 butadiene in the polybutadiene block. Unlike other studies of POSS grafted hybrid polymers, this synthesis method enabled us to make a series of polymers with varying amounts of POSS without any change to the overall degree of polymerization or structure of the main SBS backbone. This approach offers a unique opportunity to study the influence on polymer properties and morphologies due solely to the POSS grafted on the backbone of block copolymers. X-ray diffraction revealed that the grafted POSS molecules were well dispersed, which is in contrast to the strong segregation if the POSS-silane was simply blended into SBS. Small-angle x-ray scattering (SAXS) indicates that, although the block copolymer morphology (cylindrical or lamellar) was not altered by the presence of POSS, the attachment of POSS to the butadiene block affects the order and periodicity of phases. SAXS was also used to examine the behavior of the order-disorder transition, and results were compared to standard rheological measurements. Dynamic mechanical analysis showed POSS attachment hindered the motion of the polybutadiene block. More interestingly, the presence of POSS grafted to the soft block also resulted in a tapered damping factor associated with the glass transition of the polystyrene block and improved the tensile performance at temperatures near the glass transition of polystyrene, while maintaining the mechanical performance of SBS at temperatures about the glass transition of polybutadiene. This observed high-temperature tensile performance in SBS was attributed to the delayed re-orientation process in the styrene block, as suggested by the in situ SAXS measurements.

Biology

POSTER 15**Wide-Angle X-ray Solution Scattering as a Probe of Ligand-Induced Conformational Changes in Proteins**R.F. Fischetti,¹ D.J. Rodi,¹ D.B. Gore,² and L. Makowski¹¹Biosciences Division, Argonne National Laboratory, Argonne, IL 60439, USA²Bio-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

A chemical genetics approach to functional analysis of gene products utilizes high-throughput target-based screens of compound libraries to identify ligands that modulate the activity of target proteins. Candidate compounds identified via binding assays are further screened using functional assays designed specifically for the protein—and function—of interest. This approach suffers from the need to customize the assay to each protein. An alternative strategy is to utilize an *in vitro* biophysical probe to detect the structural changes that usually accompany the binding of a functional ligand. Wide-angle x-ray scattering (WAXS) from proteins in solution provides a means to identify a broad range of ligand-

induced changes in secondary, tertiary and quaternary structure. Although WAXS data is significantly weaker than SAXS data, it can be collected using less than 200 μl of solution volume with protein concentrations of 5 to 10 mg/ml in less than 30 seconds with the high flux available at a third-generation synchrotron x-ray source. The speed and accuracy of data acquisition, combined with the broad range of label-free targets and binding conditions achievable with this technique, indicate that WAXS is well suited as a moderate-throughput assay in the detection and analysis of protein-ligand interactions.

Instrumentation

POSTER 16

Stabilization of Beam Position at Energy Scans by Synchronous Corrective Motions of Monochromator and Mirror

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²GM/CA-CAT at the APS, Argonne National Laboratory, Argonne, IL 60439, USA

Double-crystal monochromators are supposed to provide fixed exit of the x-ray beam during the energy changes. However, if the two crystals of the monochromator have different lattice spacing due to a difference in their material or temperature, then the fixed exit property may be broken and the beam moves along the sample surface at energy scans. Considering the same material of the crystals, the difference in their temperature corresponding to cryo- and water-cooling of the first and the second crystal, respectively, gives the $dD/D = 2.4 \times 10^{-4}$. At the energy of 7.112 keV (Fe K edge) this results in the beam motion of $\sim 300 \mu\text{m}$ per 1 keV scan at the distance of 15 m from the monochromator. The motion can be separated into linear and angular components. The former can be eliminated by correcting vertical displacement of the second crystal of the monochromator during the scan and the latter by rotating the mirror. Importantly the linear correction is aimed to keep the beam at constant height at the mirror so that the mirror focusing parameters are preserved. Recently we have implemented these corrections in the On-the-Fly Scan software at BioCAT and experimentally proved the stabilization of the beam position. *The work was supported by NIH grant RR08630.*

Chemistry

POSTER 17

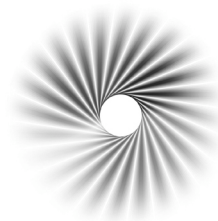
Studying DNA Conformational Dynamics in Solution with Wide-Angle X-ray Scattering

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Information on the structure and conformational dynamics of DNA in liquids and other noncrystalline media is critical for understanding its biological and chemical functions, such as a building block for molecular electronics, recognition, and a linker for light-activated donor/acceptor assemblies. We have begun a program that examines synthetic DNA structure and structural dynamics in solution using synchrotron wide-angle x-ray scattering. Scattering patterns measured to 2 angstrom resolution were found to resolve both nearest-neighbor and longer range base-pair distance correlations. Measurements and molecular modeling studies on a series of sequences and temperature show that scattering peak positions are strongly sensitive to DNA conformation, while configurational dynamics are reflected in



peak broadening and attenuation. Significant differences were observed between poly(AT) and poly(CG) sequences that resolve characteristic differences in both base composition and base-pair rigidity. Wide-angle scattering provides a direct, high-throughput structural approach for quantitative identification of DNA structure and configurational dispersion in functionally relevant solution conditions.

Condensed Matter Physics

POSTER 18

Single-Crystal Diffuse Scattering: Measuring Volumes of Reciprocal Space

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²Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA

Traditionally, crystallography has focused on measuring the average structure of materials, examining only Bragg diffraction intensities and making the assumption that the system is translationally invariant. This assumption often overlooks subtle structural details important in, for example, strongly correlated electron systems and ionic conductors. Diffuse scattering is a powerful probe of defect structure, including defect-defect correlations, and comprises all coherent scattering that does not arise from the average crystal lattice. It is typically weak compared to Bragg scattering and extends broadly throughout Q space. Applying the rotation method and coupling high-energy x-rays with area detectors allows volumes of diffuse scattering data to be measured both efficiently and quantitatively. The application of high-energy x-rays is a necessity for these measurements as it eliminates the need for absorption corrections and allows high values of the scattering vector, Q, to be probed. We present preliminary results of measurements we have made at beamline 1-ID at the Advanced Photon Source, illustrating the application of the method to systems with intrinsic disorder.

Condensed Matter Physics

POSTER 19

Spin-Resolved Core Level Photoemission of Ferromagnetic Thin Films

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We present spin-resolved 2p core level photoemission results for thin films of Fe, Co, and Ni. All films have an in-plane magnetic easy axis. We observe spin polarization in the main photoemission peaks consistent with trends in the bulk magnetic moments of the transition metals. In addition, Ni and Co have satellite peaks due to electron correlation effects, and we see spin polarization in the Ni 6 eV satellite peak and much weaker spin polarization in the 4 eV Co peak. The existence of a satellite peak in the Co 2p spectrum is somewhat controversial, and these data mark the first observation of spin-polarization in that peak. In addition, in the Ni/Co/Cu(001) system, we have preliminary results for very thin films of Ni where charge transfer from the Co to the Ni affects the electronic and magnetic properties of both films, and we see differences in the photoelectron spin polarization of both Co and Ni compared to results for thicker Ni and Co films. These results emphasize the importance of spin-

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resolved photoemission in understanding the combined Coulomb, spin-orbit, and exchange interactions in the presence of interatomic electron correlation effects and configuration mixing that affect the photoemission process.

Condensed Matter Physics

POSTER 20

Surface Oxidation of Liquid Sn

A. Grigoriev,¹ O. Shpyrko,¹ C. Steimer,¹ P. Pershan,¹ B. Ocko,² M. Deutsch,³ B. Lin,⁴ J. Gebhardt,⁴ T. Graber,⁴ and M. Meron⁴

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Interfacial chemical reactions will often exhibit both unusual kinetics and new reaction products that are either unknown or unstable in the bulk state. We report on an x-ray scattering investigation of the oxidation of the liquid Sn surface. The pure liquid Sn surface does not appear to react with molecular oxygen below a threshold pressure of about 5.0×10^{-6} Torr. Above that pressure a rough solid Sn oxide grows over the liquid metal surface. On the other hand, once the threshold pressure has been exceeded and oxides have been formed, further oxidation proceeds slowly at pressures below the activation threshold. The observed Bragg peaks from the surface oxide can only be fit by a cubic rock salt (NaCl) structure with lattice parameters are close to one of the parameters of SnO. This phase does not match any of the known Sn oxide phases at normal conditions.

Condensed Matter Physics

POSTER 21

Surface Structure Study of Liquid Eutectic Alloys: AuSi and AuGe

A. Grigoriev,¹ O. Shpyrko,¹ C. Steimer,¹ P. Pershan,¹ B. Ocko,² M. Deutsch,³ B. Lin,⁴ J. Gebhardt,⁴ T. Graber,⁴ and M. Meron⁴

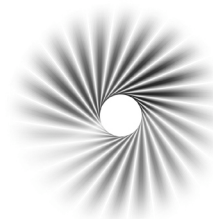
¹Dept. Physics, Harvard University, Cambridge, MA 02138, USA

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Knowledge of surface structure and surface properties is crucial to the rapidly developing field of nanotechnology, especially when speaking of such technologically important materials as silicon, gold and germanium. We report on a study of the liquid surface structure of AuSi and AuGe eutectic alloys. These eutectics have a melting temperature of about 360°C, which is far below the melting temperature of Au (1064.42°C), Ge (937.4°C) or Si (1409.84°C), and are already being widely and successfully used as solders in electronics. In spite of the clear similarity between the two eutectics, we found that their surface structure is remarkably different. Surface layering has been observed in both liquids, but, for the AuSi eutectic, the layering peak in reflectivity is at least 20 times stronger than in any of the liquid metals and alloys studied previously. This effect is being currently analyzed; however, there are open questions that can only be addressed by experiments that are proposed.



POSTER 22

Measurement of the Valence Electron Form Factor of Simple Metals Using Inelastic X-ray Scattering

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In order to understand the nature of the metallic bonding, it is crucial to obtain the valence electron charge density. The Fourier transform of the charge density, the atomic form factor, can be split into two parts: namely a rigid part, which moves together with the nucleus (core electrons), and the valence electrons, which are not as strongly bound to the nucleus as the rigid part. The atomic form factor can, in principle, be derived from x-ray scattering experiments. In earlier experiments, the form factor was obtained from the intensities of Bragg reflections, limiting the accessible Q-range to above $\sim 2.5 \text{ \AA}^{-1}$.

In contrast to previous experimental methods (Bragg intensities), the inelastic x-ray scattering (IXS) technique can be applied to this small Q-region by measuring phonon intensities. We used IXS with a very high resolution of 2.2 meV to measure the phonon intensities along the [0 0 L] direction in aluminium and beryllium and the [0 L L] direction in lithium crystals. From the relative intensities of phonons, the atomic form factors for these samples have been calculated. Comparing to Hartree-Fock calculations, the form factor deviates significantly in the case of lithium and beryllium.

Materials Science

POSTER 23

Resonant Scattering Studies of the Pb/Bi Distribution in the Thermoelectric Material $\text{Pb}_5\text{Bi}_6\text{Se}_{14}$ at K Edges

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The compound $\text{Pb}_5\text{Bi}_6\text{Se}_{14}$ is an efficient thermoelectric material, in which the Pb and Bi atoms are distributed over eleven crystallographically distinct sites. The determination of this distribution is needed in order to fully evaluate what determines the thermoelectric properties of the materials. There is very little contrast between Bi and Pb when using a common laboratory x-ray source, and neutrons also provide poor contrast. However, considerable contrast can be achieved in resonant scattering experiments at the Bi and Pb L_{III} edges, but the measurement of reliable powder-diffraction data at these edges is greatly hindered by the strong sample absorption due to the Pb, Bi and Se (the L_{III} edges are above the Se K edge) and a strong tendency towards preferred orientation when using reflection-geometry powder samples. Resonant scattering measurements employing the Pb and Bi K edges, at ~ 90 keV, get around the absorption problem and facilitate the use of a Debye-Scherrer geometry to solve the preferred orientation problem.

The recently developed high-resolution high-energy x-ray optics at the 1-ID beamline of the Advanced Photon Source achieve a narrow energy bandwidth (~ 6 eV at 90 keV) and excellent energy stability, making them well suited for resonant scattering studies at high energies. Resonant powder-diffraction data were collected using a Debye-Scherrer geometry using a MAR345 image plate area detector. Data were recorded at three energies, 87.968 keV (just below the Pb K edge), 90.493 keV (just below the Bi K edge) and 86.014 keV. The resonant scattering factors were obtained by Kramers-Kronig transformation of absorption spectra for $\text{Pb}_5\text{Bi}_6\text{Se}_{14}$. The image plate data were integrated with the FIT2D program. Rietveld refinement using the GSAS program with combined data sets gave convincing Pb/Bi occupancy values. This experiment demonstrates that very high energy resonant scattering can be a useful tool for site occupancy determinations in complex materials when lower energy edges cannot be used due to sample absorption or sample container requirements. Even though the high-energy K absorption edges do not provide as much scattering contrast as the lower energy edges, the data quality is much better than those at the lower energy L_{III} edges.

Acknowledgment: The work at the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Basic Energy Sciences, under contract W-31-109-Eng-38. APW is grateful for partial support from the NSF under DMR-0203342. Financial support from the Office of Naval Research (contract N00014-02-1-0867 for M.G.K.) is gratefully acknowledged.

Imaging

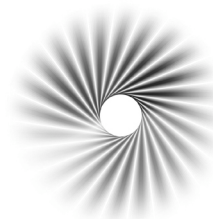
POSTER 24

Imaging of Quantum Array Structures with Coherent and Partially Coherent Diffraction

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Recent achievements in experimental and computational methods open the possibility to measure and invert the diffraction pattern from a finite object of submicron size. In this paper, the possibilities of such experiments for 2D arrays of quantum dots are discussed. The diffraction pattern corresponding to coherent and partial coherent illumination of a sample was generated. Test calculations based on the iterative algorithms were applied to reconstruct the shape of the individual islands in such a quantum structure directly from its diffraction pattern. It is demonstrated that, in the case of coherent illumination, the correct shape and orientation of an individual island can be obtained. In the case of partially coherent illumination, the correct shape of the particle can be obtained only when the coherence of the incoming beam is reduced to match the size of the island.



SAXS

POSTER 25**Dynamics of a Monolayer Nanocrystal Self-Assembling Process in Liquid Film Studied by in situ Small-Angle X-ray Scattering**X.-M. Lin,¹ S. Narayanan,² X. Li,² and J. Wang²¹Materials Science Division, Chemistry Division and Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA²Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Hierarchical self-assembly is an important process nature adopts to form the physical world around us. In this scheme, atoms are used to form nanometer scale objects, which are subsequently used as building blocks for more complex structures. Self-assembling chemically synthesized monodispersed colloidal nanocrystals provide a simple model system to mimic this process. It is also a key step for making future electronic and optical devices. However, even for the simplest case of drying a nanocrystal colloidal droplet, a variety of self-assembled structures can be formed. Presumably, this is due to different experimental conditions, such as particle sizes, ligand chain length, solvent polarity, evaporation rate, etc. On the other hand, the physical properties of different assemblies can vary dramatically because of the structural differences. Therefore, understanding how self-assembly depends on the experimental conditions is crucial to controlling the self-assembling process. In situ experiments to study the dynamics of the self-assembly can delineate the effect of various experimental conditions. We have performed the first in situ small-angle x-ray scattering (SAXS) experiment of the formation of a highly ordered nanocrystal superlattice monolayer in liquid film. We have shown that assembly of the nanocrystal occurs preferentially at the liquid-air interface under the fast initial evaporation conditions. This is in sharp contrast with the traditional notion that the self-assembling process occurs predominantly at the liquid-substrate interface. Our experiments also indicate that kinetics can play an important role in formation of nanostructures. If the initial evaporation rate is fast, nanocrystals can accumulate near the liquid-air interface, and formation of 2D superlattices occurs. On the other hand, if the liquid evaporation is slow enough, the diffusion of nanocrystals into the bulk part of the liquid becomes important, then 3D nanocrystal superlattices can be formed.

*High Pressure***POSTER 26****Quenchable High-Pressure Phase of Al(OH)₃**H. Liu,¹ J. Hu,² Z. Liu,² D. Häusermann,^{1,2} H.-K. Mao,² J. Chen,³ and D.J. Weidner³¹HP-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA²Geophysical Laboratory, Carnegie Institution of Washington, Washington DC 20015, USA³Mineral Physics Institute, State University of New York at Stony Brook, NY 11794, USA

In situ high-pressure synchrotron x-ray diffraction and infrared absorption experiments for gibbsite were performed at room temperature up to 53 GPa and 25 GPa. A phase transition was confirmed at about 2.5 GPa. The high-pressure phase is indexed as an orthorhombic structure, rather than a triclinic structure as reported in previous studies. The compressibility of gibbsite and its high-pressure polymorph were

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studied, and their bulk moduli were determined to be 49 GPa and 75 GPa, respectively, with K' as 4. The *in situ* high-pressure infrared absorption spectra revealed the gradual disordering of hydrogen substructure above 15 GPa in quasi-hydrostatic compression.

High Pressure

POSTER 27

High-Pressure Crystal Structure of CdO: From Theoretical Calculations to *in situ* Synchrotron X-ray Diffraction Experiments up to 176 GPa

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²Geophysical Laboratory, Carnegie Institution of Washington, Washington DC 20015, USA

The 3d and 4d transition metal monoxide ZnO and CdO were both predicted to undergo a NaCl (B1) to CsCl (B2) type structural transformation under high pressure based on first principle total energy calculations. We will present experimental research on the compressibility and phase transition of CdO up to 176 GPa at room temperature using *in situ* high-resolution angular dispersive x-ray diffraction from a synchrotron source combined with the diamond anvil cell technique. The phase transition from a B1- to a B2-type structure for CdO was observed at a pressure of about 90.6 GPa, which is in good agreement with the prediction. The bulk moduli of the B1 and B2 phases of CdO at zero pressure were estimated as 147 and 169 GPa, respectively. The difference between the experimental and simulation results for the bulk modulus and the possibility of the B1-to-B2 phase transition in ZnO will be discussed.

Imaging

POSTER 28

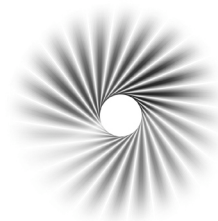
X-ray Fluorescence Imaging and Micro-XAFS in Biological Samples

R. Barrea,¹ D. Gore,¹ M. Davidson,² E. Kondrashkina,¹ K. Zhang,¹ R. Heurich,¹ M. Vukonich,¹ and T.C. Irving¹

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²University of Florida, Materials Science and Engineering, Microfabritech, Gainesville, FL 32611, USA

The Biophysics Collaborative Access Team (BioCAT) at the APS is dedicated to the study of partially ordered and disordered biological materials. Recently, microfocus capabilities have been incorporated at BioCAT that will allow x-ray fluorescence imaging, microdiffraction, micro-XAFS, microtomography and microfluorescence tomography on biological samples. The microfocus optics comprises a pair of XRADIA KB mirrors and a zone plate, 10 cm focal length. The platinum-coated silicon KB mirrors are 4 × 0.5 × 0.2 inches, and each individual KB mirrors has two separate bending picomotors, a tilt and a translation picomotor (four picomotors per mirror). Each of the picomotors has a LVDT position-sensitive device to determine the absolute position. The cut-off energy is somewhat structured due to the Pt L edges, but the mirrors are functional out to ~17 KeV. The low-energy limit of 3.5 keV is imposed by the beamline monochromator. High-precision positioners for sample positioning, a KeteK Si drift



detector and a Ge detector complete the setup. The combination of microbeams and the existing fast-scanning capabilities of the BioCAT beamline allow performing x-ray fluorescence mapping and micro-XAFS measurements. This paper reports the commissioning results of the KB mirrors and the first results of x-ray fluorescence mapping and micro-XAFS at the BioCAT 18ID undulator beamline. A $3.5 \times 3.5 \mu\text{m}^2$ beam at 10 keV was measured by means of a knife-edge Ni thin film. Comparison of standard 2D step scans and fast continuous scans will be presented. Examples of x-ray fluorescence mapping on brain cells with three levels of resolution, 100 μm , 20 μm and 3.5 μm beam size, will be shown.

High Pressure

POSTER 29

Microstructure Evolution of NaMgF_3 under Quasi-Hydrostatic Pressure Conditions

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²Mineral Physics Institute, State University of New York at Stony Brook, NY 11794, USA

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The Earth's lower mantle is thought to be dominated by $(\text{Mg}, \text{Fe})\text{SiO}_3$ perovskite, and the physical properties of this mineral are, therefore, of great interest within the Earth sciences. However, the lower mantle conditions (high pressure and high temperature) are not easily investigated. Due to the similarities between their crystal and electronic structures, NaMgF_3 is an ideal analogue model for silicate perovskite. The weaker bonding features of NaMgF_3 grant us the opportunity to simulate the behavior of silicate perovskite at the lower mantle, i.e., under high-pressure and high-temperature conditions, at relatively lower P-T conditions. Here we report the results from high-pressure *in situ* angle-dispersive x-ray diffraction experiments, and the microstructure evolution of the sample studied under the high-pressure and high-temperature conditions.

Materials Science

POSTER 30

High-Voltage $\text{Li}[(\text{Ni}_{0.5-x}\text{Co}_x)\text{Mn}_{1.5}]\text{O}_4$ Spinel Oxide Cathode for Lithium Batteries

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Electrochemical Tech. and Basic Sciences Program, Chemical Engineering Division, Argonne National Laboratory, Argonne, IL 60439, USA

In the $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{1.5})\text{O}_4]$ spinel electrode, which provides approximately 4.7 V against metallic lithium, the nickel and manganese ions are formally divalent and tetravalent respectively. Analogous to the charge-compensation mechanism in the layered $\text{Li}[(\text{Ni}^{\text{II}}_{0.5}\text{Mn}^{\text{IV}}_{0.5})\text{O}_2]$ electrode, a Ni(II/IV) two-electron redox couple also occurs on cycling the spinel electrode (between 5.0 and 3.7 V) during which the Mn(IV) remains electrochemically inert. In this work, a series of Co-substituted spinel compounds $\text{Li}[(\text{Co}_x\text{Ni}_{0.5-x}\text{Mn}_{1.5})\text{O}_4]$ ($x=0, 0.1, 0.2, 0.4, \text{ and } 0.5$) were synthesized by solution-precipitation methods and characterized. Rietveld profile refinements of powder XRD patterns for the cubic, cation-ordered spinels (space group P4332) were used to calculate the lattice parameter, a , and non-lithium (Ni, Co) occupancy of the 8c tetrahedral site. The lattice parameter decreases linearly with the amount of substituted Co in the spinel structure, consistent with solid-solution behavior and the direct substitution of the slightly smaller Co(II) ions for Ni(II) ions. Ni, Co and Mn XANES data were collected to

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determine the nominal oxidation states of the transition metal ions in each of the compounds. For $x=0.1$, the cobalt ions are divalent, the Mn ions tetravalent, and Ni ions divalent. For higher values of x , charge-transfer to Mn occurs, creating Mn(III) with Co(III), while Ni(II) persists. First principles calculations were performed at various values of x to compare the theoretically predicted charge-ordering in $\text{Li}[\text{Co}_x\text{Ni}_{0.5-x}\text{Mn}_{1.5}]\text{O}_4$ spinels with that determined experimentally.

Financial support from the Office of Basic Energy Sciences and the Office of FreedomCar and Vehicle Technologies of the U.S. Department of Energy under Contract No. W31-109-Eng-38 and DE-AC03-76SF00098 subcontract no. 6517749 is gratefully acknowledged. Use of the Advanced Photon Source was supported by the US-DOE, Office of Science, Basic Energy Sciences. Work performed at MRCAT is supported, in part, by funding from the US-DOE.

Instrumentation

POSTER 31

Thin Films/Multilayers You May Get from the APS Deposition Lab

C. Liu, R. Conley, and A.T. Macrander

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

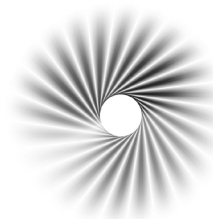
In the past 8 years, we have developed some advanced thin-film and multilayer products that you, as an APS user, may get free of charge for scientific collaborations and development.

1. Routine thin-film and multilayer coatings, substrates up to 1.5 m \times 15 cm \times 14 cm.
2. Laterally graded multilayers, maximum graded length: 100 mm.
3. Depth-graded multilayers, up to a total thickness of 12 microns.
4. Narrow-bandpass multilayers, 54% reflectivity with a bandpass of 0.46% at 10 keV and ~45% reflectivity with a bandpass of 0.67% at 25 keV of undulator x-rays.
5. Gold profile-coated elliptical KB mirrors, monolithic, available Si substrates: 70 \times 9 \times 4.5 mm³, maximum Au thickness: ~12 micron, maximum mirror length: 100 mm, expected focus spot: ~0.4 to 1 micron [1].
6. Bendable KB mirror coatings, Rh, Pd, or Pt with Cr undercoating.
7. Structure-coatings using masking techniques.
8. Other coatings and experimental samples [2].

[1] "From flat substrate to elliptical KB mirror by profile coating," C. Liu et al., Eighth International Conference on Synchrotron Radiation Instrumentation, San Francisco, CA, 2003, to be published.

[2] "Multifarious activities at the APS Deposition Lab," C. Liu, J. Erdmann, and A. Macrander, the APS User Activity Report 2000, p. 274, ANL-01/03, Dec. 2001. *The work at the Advanced Photon Source is supported by the U. S.*

Department of Energy, Office of Science, Basic Energy Sciences, under Contract No. W-31-109-ENG-38.



POSTER 32

Optics Fabrication and Metrology Group of APS/XFD

A. Macrander

Argonne National Laboratory, Argonne, IL 60439, USA

The OFM group can provide services to the APS user community in these areas:

- Crystal optics fabrication (R. Khachatryan)
- Metrology (L.A. Assoufid, J. Qian)
- Thin film deposition (C. Liu, R. Conley)
- X-ray characterization (S. Krasnicki, J. Maj)
- Optics design and development (A. Khounsary)

These areas and a new web-based work request system to access the services will be detailed.

This work supported by the U.S. DOE, Basic Energy Sciences, under contract No. W-31-109-ENG-38.

Biology

POSTER 33

Structure Determination of a Family of Cytochromes c7 from a Metal-Reducing Organism *G. sulfurreducens*

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Biosciences Division, Argonne National Laboratory, Argonne, IL 60439, USA

Geobacter sulfurreducens is important for bioremediation of toxic metals in subsurface environments. Its genome encodes over 100 cytochromes c, many of which function in electron transport to metal ions. We have previously determined the structure of a small three-heme cytochrome c7, PpcA (71 residues), which reduces soluble Fe(III) and U(VI) *in vitro* and plays a role in electron transfer in reduction of soluble Fe(III) by the organism. The genome encodes four additional cytochromes c7 with protein sequence identities ranging from 57% to 77% to PpcA. We have produced all four homologs of PpcA in *E. coli* and determined their structures by the MAD method using data collected at the Fe K edge (19BM, APS). The different homologs diffract in the range of 2.15 Å to 1.3 Å resolution, and their structures are being refined. The structures of the four homologs are more similar to each other than to PpcA. Although the sequences and structures of the three-heme cytochromes c7 are similar, their surface electrostatic potentials are different, suggesting that these molecules probably interact with different partners in the Fe(III) reduction pathway(s) used by the organism. *This work was supported by the U.S. DOE, NABIR program.*

POSTER 34

ccdApp: Area Detector Support for EPICS

M. Rivers

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ccdApp provides a uniform interface for controlling area detectors (CCD, online image plates) from EPICS. Any EPICS client (e.g., spec, IDL, scan record) can control (at a bare minimum):

- Exposure time
- File name
- Start collection, wait for completion

Much more control is provided for most detectors.

Current status of the software:

- MAR 165 CCD (complete, in use)
- Roper CCD detectors (complete, in use)
- Bruker CCD detectors (soon)
- MAR 345 online image plate (soon)

The implementation uses manufacturer's software for the primary user interface. This minimizes the amount of new code, uses existing file formats, unwarping algorithms, etc. These programs include:

- marccd for MAR165
- Winview/Winspec for Roper cameras
- SMART for Bruker cameras (can also use WinView)
- scan345 for MAR 345 image plate

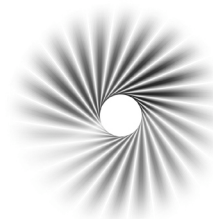
ccdApp provides control of these programs from EPICS.

Each of these programs has a "remote control" interface, typically using TCP/IP sockets

Using EPICS means each client (e.g., spec) does not have to know how to talk to each type of detector; it only has to know how to talk to EPICS. EPICS software consists of

- Database of records (PVs), identical for all detectors
- State-notation-language (SNL) programs, unique for each detector. Reads/writes PVs and communicates with remote control interface over sockets.

The database and SNL programs are typically run on the same machine on which the user interface software runs (e.g., Linux box for MAR detectors, Windows for Roper and Bruker). No VME crate is required.



POSTER 35**Picosecond Time-Resolved XAFS of Laser-Excited Thin Ge Films**D.L. Brewes,¹ E.A. Stern,² S.M. Heald,^{1,3} and Y. Feng⁴¹PNC-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA²Physics Dept., University of Washington, Seattle, WA 98195, USA³Pacific Northwest National Laboratory, Richland, WA 99352, USA⁴James Franck Institute, The University of Chicago, Chicago, IL 60637, USA

A facility at PNC-CAT in the Advanced Photon Source measures with 100 picosecond (ps) time resolution both x-ray absorption fine structure (XAFS) and diffraction on femtosecond laser-excited samples. XAFS is measured with two orders more efficiency by laser-pump and a 95 ps FWHM x-ray-probe pulse each cycle of the ring. XAFS allows the determination of: the time for the laser excitation to couple to the lattice, the sample temperature after reaching thermal equilibrium, any ablation of the sample with time, and, in many cases, allows distinction between amorphous and crystalline states. Preliminary measurements on 200-nm-thick polycrystal Ge films indicate that the laser excitation is transferred to thermal heating of the lattice with a time constant of roughly 0.5 ns.

*Instrumentation***POSTER 36****Design of a Scanning Stage System with Nanometer Resolution for a X-ray Nanoprobe Instrument**D. Shu,¹ J. Maser,^{1,2} B. Lai,¹ S. Vogt,¹ Y. Han,¹ B. Tieman,¹ R. Winarski,¹ C. Roehrig,¹ A. Smolyanitskiy,¹ and G.B. Stephenson^{2,3}¹Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA²Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA³Materials Sciences Division, Argonne National Laboratory, Argonne, IL 60439, USA

A hard x-ray nanoprobe beamline is being developed at the Advanced Photon Source (APS) [1]. The beamline will house a hard x-ray nanoprobe, one of the centerpieces of the x-ray characterization facilities of the Center for Nanoscale Materials (CNM), which is under construction at Argonne National Laboratory (ANL). The instrument will operate with photon energies between 3 keV and 30 keV. The focal length of the nanofocusing optics will typically be in the range of 10-30 mm.

We have developed a prototype instrument with a novel interferometrically controlled scanning stage system [2]. The system consists of nine DC-motor-driven stages, four picomotor-driven stages, and two PZT-driven stages. An APS-designed custom-built laser Doppler displacement meter system provides two-dimensional differential displacement measurement with subnanometer resolution between the zone-plate x-ray optics and the sample holder. The entire scanning system was designed with high stiffness, high repeatability, low drift, flexible scanning schemes, and possibility of fast feedback for differential motion. Designs of the scanning stage system, as well as preliminary mechanical test results, are presented in this paper.

[1] J. Maser, G. B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider, to be published in Proc. SRI 2003, Aug. 25-29, 2003, San Francisco, CA.

[2] D. Shu, J. Maser, B. Lai, and S. Vogt, to be published in Proc. SRI 2003, Aug. 25-29, 2003, San Francisco, CA

Work supported by the U.S. Department of Energy under contract No. W-31-109-Eng-38.

Instrumentation

POSTER 37

Progress in the Robotic Sample-Changer System Development for SBC-CAT

D. Shu,¹ C. Preissner,¹ D. Nocher,¹ Y. Han,¹ P. Lee,¹ W-K. Lee,¹ Z. Cai,¹ S. Ginell,² R. Alkire,² K. Lazarski,² R. Schuessler,² and A. Joachimiak²

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We are continuing to develop the robotic sample-changer system for the Argonne Structural Biology Center (SBC) beamlines 19-BM and 19-ID with improved cryosample temperature control and system integration. Unique to the SBC system is the ability to mount and retrieve samples from a kappa configuration goniostat. The option of loading samples by hand using cryotongs or liquid sample vials is always available to users by putting the robot in the park position. Key features of the automation system include: a direction-sensitive, force-detecting handset; crystal thermal protection; easy-to-load, open-top storage dewar; the use of commercial sample magazines; and efficient beamline hardware/software integration. Recent tests confirm a storage dewar lifetime of over 24 hours and the ability to mount and dismount a sample up to 7 times without degradation in crystal quality.

This work was supported by the U.S. Department of Energy, Office of Science, Office of Biological and Environmental Research, under Contract No. W-31-109-Eng-38.

Biology

POSTER 38

SAXS Study of the Phase Separation of Liquid ³He-⁴He Mixtures in Aerogel

L. Lurio,¹ N. Mulders,² M. Paetku,³ and M. Hold⁴

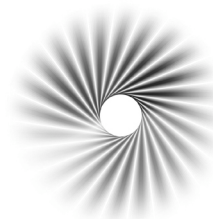
¹Northern Illinois University, DeKalb, IL 60115, USA

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Mixtures of liquid ³He and ⁴He will spontaneously separate into a ⁴He-rich superfluid and a ³He-rich normal fluid at temperatures below 0.8K. The nature of this phase separation is modified when the liquid is placed within an aerogel matrix. In this case there exist three distinct low-temperature phases, a ⁴He-rich superfluid, a ³He-rich superfluid and a ³He-rich normal fluid. The nature of the ³He-rich superfluid is believed to be related to wetting of the aerogel by the ⁴He component and possibly to capillary condensation of the ⁴He. We present measurement of the microphase separation of helium mixtures in aerogel using small-angle x-ray scattering.



POSTER 39**Dynamics at a Buried Polymer Interface**X. Hu,¹ S. Narayanan,² X. Xiao,² A.R. Sandy,² L.B. Lurio,³ and J. Lal¹¹IPNS, Argonne National Laboratory, IL 60439, USA²APS, Argonne National Laboratory, IL 60439, USA³Dept. Physics, Northern Illinois University, DeKalb, IL 60115, USA

Here we present a further development of x-ray photon correlation spectroscopy (XPCS) at grazing incidence to study polymer interfaces. We have probed capillary wave dynamics not just at a free surface but also at a buried polymer/polymer interface within a bilayer film. The bilayer was chosen so that the critical angle for total external reflection for the top layer is smaller than that for the bottom layer. When x-rays are incident below the critical angle of the top layer, only the structure and dynamics of the top layer are probed. When x-rays are incident above the critical angle of the top layer but below that of the bottom layer, a standing wave is set up. The phase of this standing wave can be adjusted to have a high intensity at the polymer-polymer interface and simultaneously a node at the polymer-air interface. Consequently, one can isolate, for the first time, the static scattering and XPCS from a single buried layer. Results on a system consisting of a 120-nm polystyrene film on top of a 100-nm poly(4-bromo styrene) film, supported on a Si substrate, will be discussed.

*Materials Science***POSTER 40****Evidence of Layered Ordering in Hybrid Silica-Oligo (p-phenylene vinylene) Nanocomposite Thin Films by X-Ray Reflectivity**

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Well-ordered hybrid silicate-surfactant self-assembled nanocomposite thin films have attracted much interest both scientifically and technologically mainly because of the possible use of their mesoporous features, which have potential applications. In this work, oligo p-phenylene vinylene (OPV) molecules were used as a surfactant to synthesize hybrid silicate-OPV layered films in a spin-coating process. OPV has been demonstrated as a material that can be used as the active element in large-area light-emitting diodes. A series of silicate-OPV films with thicknesses varying from 143 Å to 314 Å were spin-coated on float-glass substrates and studied using the specular x-ray reflectivity technique at DND-CAT. Periodic layering of silicate-OPV in these films along the surface normal was observed by the presence of “Bragg” peaks in the x-ray reflectivity data. The normalized reflectivity data was Fourier inverted to produce 1D direct space Patterson maps that showed periodic secondary maxima corresponding to periods inside the film that ranged from 26.2 to 29.4 Å, with the thicker the films having the more contracted internal structure. The surface of the films showed porous features observed by atomic force microscopy (AFM).

POSTER 41**Amorphous-Amorphous Transitions in Y_2O_3 - Al_2O_3 Liquids: Structural Changes from Combined Neutron and X-ray Diffraction**M. Wilding,¹ M. Wilson,² P. McMillan,² and C. Benmore³¹University of California at Davis, Davis, CA 95616, USA²University College London, London WC1H0AJ, UK³IPNS, Argonne National Laboratory, Argonne, IL 60439, USA

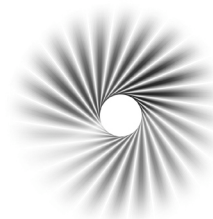
Supercooled Y_2O_3 - Al_2O_3 liquids show transition between a high-density amorphous phase (HDA) and a low-density amorphous phase (LDA). This transition is believed to be polyamorphic, a first-order transition between different structured amorphous phases. This transition is one of the few in polyamorphic systems that can be observed, and characteristic two-phase samples comprising the juxtaposed, compositionally identical HDA and LDA phases can be produced.

Combined neutron and x-ray diffraction data can be used to evaluate the structural changes accompanying this transition. These results show that the transition does not involve a change in short-range order; there is, for example, no change in the first neighbour coordination of Al(III) or Y(III). The main differences are in mid-range order. Weighted differences between the x-ray and neutron structure factors and their real-space transforms indicate that HDA-LDA transition reflects a reduction in entropy associated by changing the connectivity of Al-O and Y-O structural units.

Instrumentation

POSTER 42**Single-Pulse Measurement of Synchrotron Radiation with a X-ray Streak Camera**J. Liu,¹ E. Landahl,¹ J. Wang,¹ and Z. Chang²¹Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA²J. R. Macdonald Lab, Kansas State University, Manhattan, KS 66506, USA

For ultrafast synchrotron experiments, there is increasing interest in obtaining shorter beam bunches and higher peak current for storage rings of synchrotron radiation sources. A streak camera is one of the commonly used detectors for ultrafast laser-pump-x-ray-probe measurements with both high temporal and spatial resolutions. With the recent development of third-generation synchrotron radiation sources, intense hard x-rays having a pulse duration of a few tens of picoseconds have become available; single shot measurements are also possible. We have demonstrated that a newly developed x-ray streak camera can be effectively used to measure a single x-ray pulse from synchrotron radiation of the Advanced Photon Source. In order to obtain high sensitivity, CsI was used as a photocathode. With this streak camera, the single bunch x-ray pulse width is measured to be 105 ps (full width at half maximum) at photon energy 8.0 keV. After optimizing all aspects of the detector and the beamline optics, it will be possible to perform single x-ray-pulse experiments in a manner of a single shot without resorting to a laser pump. *This work and the use of the APS are supported by the U.S. Department of Energy under contract W-31-109-ENG-38 and the work is also partially supported by DOE DEFG02-00ER15082.*



POSTER 43

Combined Experimental and Theoretical Investigation of Electronic Correlations in Simple and Complex Metals and Insulators

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Inelastic x-ray scattering (IXS) measurements have been made of the energy-loss spectra for simple and transition metals and transition metal oxide materials, including Al, Sc, Cr, TiO₂, and NiO. The measurements made on UNICAT 33ID with ~ 1 eV energy resolution have been reported in absolute units of 1/(eV*Å**3) so that direct correlation with first principles computations can be made with no arbitrary scaling factors. Detailed comparison of nonresonant IXS measurements with first-principles time-dependent density functional theory (TDDFT) calculations have shown that the basic dynamical electronic physics is already contained in the random phase approximation (RPA) even for correlated electron systems, such as transition metals, if crystal local-field effects are included. On the other hand, the impact of dynamical electronic exchange-correlation effects cannot be ignored and must be addressed in order to proceed toward a fundamental understanding of the dynamical electronic structure of materials as probed by IXS measurements of the electronic response.

Time-resolved Studies

POSTER 44

Directly Visualizing Global Structural Changes in Biological Molecules

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Biological molecules are dynamic entities that are often required to undergo a wide range of structural motion in order to perform their activities in the cell. Time-resolved crystallography is an invaluable tool in which to directly observe these structural transitions, but the technique itself can potentially perturb results. The blue light photoreceptor, photoactive yellow protein (PYP), exhibits a light-induced photocycle that ultimately produces large-scale tertiary structural changes in solution. Although it has been established that the structural changes occurring on and around the light-absorbing chromophore are not significantly altered by embedding PYP in a crystalline lattice, there is considerable question as to whether the large-scale tertiary motion is possible. We have demonstrated with extremely redundant, high-quality, time-resolved crystallographic data on PYP that the global changes are maintained in the crystal. Although the magnitude of motion is clearly muted, the nature of the structural change is fully consistent with that observed in solution.

POSTER 45

Effect of Ethylene Glycol on the Conformation of Bovine β -Lactoglobulin Studied by Solution X-ray Scattering

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We have paid special attention to the use of ethylene glycol (EGOH) for cryogenic study, as it moderately affects protein structure and function. Recently, the effect of hydration on protein conformation has also been extensively studied, and the effect of EGOH on the hydration shell should be re-examined under the light of recent knowledge of hydrated water surrounding proteins, which is the first aim of the present study. EGOH is also expected to be an effective radical scavenger as well, which is the second aim of this paper.

As a target protein, we used bovine beta-lactoglobulin (BLG). Results show that ethylene glycol may affect the hydration shell, but no significant conformational changes were observed. A radiation effect was also investigated in the presence and absence of EGOH. Exposure to x-rays of 10^{10} photons/s for as long as 300 s induced a radiation effect, judging from the radius of gyration in the absence of EGOH; whereas no significant changes were observed even for 1 hour exposure in the presence of 45% EGOH.

Instrumentation

POSTER 46

Lithium X-ray Refractive Optics: Performance and Limitations

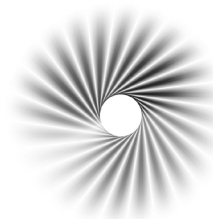
N.R. Pereira,¹ E.M. Dufresne,² R. Clarke,² and D.A. Arms³

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For refractive x-ray optics, the most promising material is lithium metal. The two-lens geometries implemented in lithium to date, multiprisms and parabolic lenses, have close to the predicted 50% throughput for x-rays at 10 keV. However, the intensity gain in the focus is an order of magnitude lower than expected from an ideal lens because the focal region is larger than predicted. This poster highlights the performance of the lithium lenses that are available now and discusses how the optical quality can be improved.



POSTER 47

Impact of Interfacial Disorder and Magnetism on Spin Injection through an Fe/AlGaAs Schottky Barrier

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²Materials Physics Branch, Naval Research Laboratory, Washington, DC 20375, USA

³Dept. Physics, State University of New York, Buffalo, NY 14260, USA

High-quality interfaces are key to the efficient transport of spins across the ferromagnet/semiconductor. The electronic and magnetic of Ga and As at the interface may also play an important role in ways that are not well understood. Here we present a study of both the interface magnetism and morphology of an Fe/AlGaAs/GaAs/AlGaAs quantum well structure. X-ray magnetic circular dichroism (XMCD) and x-ray magnetic resonant scattering (XRMS) provide an element-selective insight into both the magnetism of the Fe layer, as well as that of the AlGaAs interface underneath. Since only the first few monolayers of AlGaAs at the interface are spin polarized, it is only possible to measure the induced moments of a buried interface using XRMS. Interface morphology can also be determined in an element- and therefore interface-selective manner via this technique. Here we present results from (100) and (110) oriented device structures to correlated interface morphology and magnetism with the spin-dependent transport.

Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, under Contract No. W-31-109-Eng-38. The work at NRL was funded by ONR and DARPA and work at SUNY was funded by the NSF.

Time-resolved Studies

POSTER 48

X-ray Photoionization in the Presence of Strong Optical Fields

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We are investigating how a high-field/ultrafast laser modifies the x-ray photoionization and vacancy decay of an isolated atom. Since many proposed experiments for the next-generation x-ray sources, e.g., the Linac Coherent Light Source (LCLS), involve laser/x-ray pump-probe techniques on the ~ 100 fs timescale, it is interesting and important to understand how the x-ray physics of an atom is perturbed due to the presence of a high-power laser. Two effects have been theoretically predicted and observed for valence electrons: (1) a ponderomotive shift of the ionization threshold and (2) the appearance of sidebands in the photoelectron spectrum. The shifts may be sizable with readily available laser intensities; at 10^{14} W/cm² ($1\text{mJ}/100\text{ps}/10\mu\text{m}^2$) for 800 nm light, the ponderomotive energy is 6 eV. These effects have never been observed in the x-ray region or at an inner shell threshold. These studies are being conducted at MHATT-CAT at the Advanced Photon Source, where the output of an amplified Ti:sapphire laser system, (~ 1 kHz, ~ 1 mJ/pulse), has been overlapped with focused x-ray pulses (2×10^5 x-rays/pulse, <10 μm spot size) in an effusive krypton jet. X-ray emission and ion/electron time-of-flight are used to monitor the x-ray absorption and decay.

POSTER 49**GISAXS Studies of Temperature-Induced Growth of Supported Metal Nanoparticles**

S. Vajda, R.E. Winans, S. Riley, G. Tikhonov, S. Seifert, B. Lee, and N. VanWermeskerken

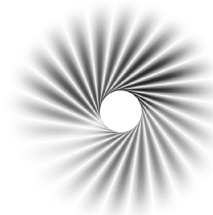
Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA

Thermal stability of supported nanoparticles is a central issue, for example, in catalysis, because the increased mobility of nanoparticles at elevated temperatures may cause their coalescence leading to the loss of catalytic activity and selectivity. In our studies, we addressed the growth of supported nanoparticles as a function of temperature and time by employing GISAXS technique on the BESSRC 12-ID beamline at the Advanced Photon Source of ANL. Platinum clusters were generated in a laser vaporization source and deposited on silicon substrates. In order to determine the temperature region of cluster growth, the samples were gradually heated from room temperature up to above 400°C, and x-ray scattering data were collected during the heat treatment. The analysis of GISAXS data shows that the nanoparticles preserve their original size up to approximately 350°C, at which an abrupt onset of agglomeration takes place. The kinetics of cluster growth was studied by heating up a sample to 400°C and recording scattering data as a function of time. The obtained data are indicative of a two-step agglomeration process. These experiments demonstrated the powerful combination of cluster deposition technique with x-ray techniques, which can aid in characterization and design of novel nanoparticle-support combinations with potential use in catalysis. *This work has been supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.*

Condensed Matter Physics

POSTER 50**Is Colossal Magnetoresistance (CMR) Just a By-product of Magnetostructural Effects in the Manganites?**Y. Ren,¹ O. Yu. Gorbenko,² and N.A. Babushkina³¹*Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA*²*Dept. Chemistry, Moscow State University, Moscow 119899, Russia*³*Kurchatov Institute, Moscow 123182, Russia*

Magnetic field (MF) dependence of the phase separation (PS) in some manganites was studied using high-resolution high-energy x-ray powder diffraction. At room temperature, all compounds in the investigation are single phase. But below a certain ordering temperature T_c , they all show intrinsic inhomogeneities in the form of coexisting competing phases. Application of MFs not only eliminates the multiple phases for the benefit of the ferromagnetic phase below T_c but also significantly affects the structure above T_c . The MF dependence of superstructural reflections, due to charge/spin/orbital ordering, clearly indicates a much more complicated PS. This MF-induced homogeneity should play a key role in the colossal magnetoresistance (CMR) effect. The results imply that the CMR effect is probably just a by-product of the magnetocrystallographic effect in these manganites. *Work at Argonne is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.*

**POSTER 51****Comparison of Neural Tissue Preparation Methods for X-ray Fluorescence Microanalysis**S. Vogt,¹ T. Paunesku,² B. Lai,¹ J. Maser,¹ G. Woloschak,² and E. Bigio³¹Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA²Dept. Radiology, Northwestern University, Chicago, IL 60611, USA³Dept. Pathology, Northwestern University, Chicago, IL 60611, USA

Metals likely have a role in the development of neurodegenerative disease—if not directly causative, at least because of their involvement in free radical production. For example, iron levels are high in brains of individuals with Alzheimer disease and Parkinson disease. X-ray fluorescence to determine metal concentrations in tissue sections is the most sensitive technique available for this type of analysis and we intend to use it to establish a firm correlation between metals in brain tissue and dementia. Since sample preparation and resulting quality of preservation of (trace) metal content is a critical issue, we compared several different methods of neural tissue preparation including an approach identical to the one used most commonly on human samples. We compared unfixed brain tissues to tissues fixed in glutaraldehyde, paraformaldehyde, and formalin, in combination with freezing and cryosectioning (with and without freeze drying), or paraffin embedding. The data suggest that the tissues from human brain banks will provide us with samples of acceptable quality for x-ray fluorescence analysis, and therefore will potentially allow the use of the full range of tissues that are available in brain banks to study neurodegenerative diseases by x-ray fluorescence microscopy.

*High Pressure***POSTER 52****High-Pressure Structures of Sulfur Dioxide by Vibrational Spectroscopy and X-ray Diffraction**Y. Song,¹ M. Somayazulu,² Z. Liu,³ D.R. Herschbach,⁴ H.-K. Mao,¹ and R.J. Hemley¹¹Carnegie Institution of Washington, Washington DC 20015, USA²HP-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA³National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11793, USA⁴Dept. Chemistry and Chemical Biology, Harvard University, Cambridge, MA 02138, USA

Gaseous sulfur dioxide has been investigated by extensive means due to its fundamental importance in atmospheric chemistry. However, its behaviors and structures in condensed phase, especially under high pressures, are basically unknown. In this study, we used diamond anvil cell techniques to study sulfur dioxide in a broad pressure and temperature range. Characterized by Raman and infrared spectroscopy, as well as x-ray diffraction measurement, several new phases of sulfur dioxide are identified under high pressures. At pressures above 17.5 GPa and room temperature, we observed wealthy and peculiar behavior of several Raman modes of sulfur dioxide, indicating the transformation into new phases and profound changes of molecular structures. Low-temperature Raman measurement, however, revealed totally different responses to pressure than did room temperature measurement, thus contributing to the construction of the phase diagram of sulfur oxide. Synchrotron infrared absorbance spectra suggest consistent pressure-induced phase transitions. Angle-dispersive x-ray diffraction measurement as a function of pressure further provides ambiguous evidence of new phases. The patterns with wealthy

reflections in the pressure region of 5.1 GPa to 10.2 GPa allow the determination of crystal structures of sulfur dioxide at high pressures and possible structural refinement.

Other

POSTER 53

Fluctuation X-ray Microscopy: A Novel Approach for Structural Study of Disordered Materials

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Fluctuation x-ray microscopy is an x-ray extension of an electron-scattering approach developed previously. It offers quantitative insight into medium-range correlations in materials at nanometer and larger length scales. X-ray speckle patterns are measured from a large number of similar locations in the sample under study. From an examination of the sample-illumination size dependence of the speckle variance, the correlation length can be estimated. We studied a distribution of polystyrene latex spheres to demonstrate this new technique. The medium-range order in the system was determined and analyzed quantitatively.

Instrumentation

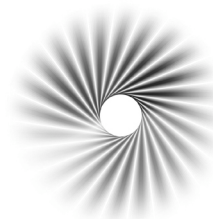
POSTER 54

Use of Robotics for High-Throughput Screening and Data Collection at IMCA-CAT

K.P. Battaile, A.M. Mulichak, I.Koshelev, J.L. Muir, K.S.M. Favale, A. Bertling, and L.J. Keefe

IMCA-CAT, Argonne, IL 60439, USA

Pharmaceutical research and development, as well as structural genomics projects, are starting to rely on high-throughput methods for data collection. A limitation to high-throughput use of the synchrotron is the manual mounting and centering of samples. With this in mind, IMCA-CAT has moved to implement robotics on 17-ID to mount samples, automatically center cryoloops and collect data. The system we have installed is a Rigaku/MSM ACTOR robot controlled by JDirector software. Use of the robot for these functions results in significant time savings by eliminating the need to repeatedly enter and exit the end station. With this system, we are able to screen up to 60 crystals in about 90 minutes. The JDirector software is laid out in a tabbed-notebook format that is user customizable, which allows a new user to quickly start collecting data. JDirector is also integrated with beamline controls, making it ideal for MAD data collection. Data collection jobs can be edited while running, which allows a great deal of user flexibility to prioritize experiments. Overall, the integrated robotics on 17-ID can reduce the number of people required to collect data and increase the amount of data that can be collected during an experiment cycle.



POSTER 55

Telecrystallography: Remote Control and View Experiments

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We are developing remote control and viewing capability at BioCARS. A prototype has been implemented, and users can simply link to a specified website where a Java Applet user interface will appear. The user interface's layout attempts to achieve the look and feel of BLU-ICE (<http://smb.slac.stanford.edu/blu-ice/intro.html>). The software consists of the following parts: a Java Applet that interacts with a user, a Java Server that interacts with the Applet as well as EPICS and a MySQL database, and a Sequence Engine that interacts with EPICS and the MySQL database. Once the user fills in fields such as rotation axis, exposure time, file directory, and image prefix, a push of the Run button triggers the Sequence Engine to start data collection. Diffraction patterns, run status, and beamline status are reported back to the user interface. This telecrystallography software provides users with remote control and viewing capability. The separation of the software into these parts permits developers to use a variety of tools to develop the user interface with access controlled through the CARPS administrative database (<http://carps.sourceforge.net>).

Materials Science

POSTER 56

Ag-Ag Correlations in Silver Sulphide Glasses Studied Using High-Energy X-ray Scattering, Neutron Diffraction with Isotopic Substitution and RMC Modeling

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Ionic conductivity and silver tracer diffusion coefficient increases by 7 to 10 orders of magnitude with increasing Ag content from 30 ppm to 35 at.%, also changing dramatically the ion transport regimes and the Haven ratio from $H_R = 1$ (uncorrelated) to $H_R = 0.2-0.3$ (strongly correlated ion motion) [1, 2]. The essential role of short Ag-Ag correlations at $\sim 3 \text{ \AA}$ in determining the ionic mobility and the preferential conduction pathways in a disordered glass network will be shown using high-energy x-ray scattering, neutron diffraction with isotopic substitution, and RMC modelling for $\text{Ag}_2\text{S-GeS-GeS}_2$ glasses with the silver content ranging between 5.9 and 30.8 at.%. A fully connected Ag-related subnetwork consisting of chains and chain fragments formed by edge- and corner-sharing AgS_3 pyramids is a characteristic structural feature of the superionic $40\text{Ag}_2\text{S-40GeS-20GeS}_2$ glass. Disintegration of the silver subnetwork into isolated species with decreasing Ag content leads to the observed remarkable change in the ion transport.

[1] E. Bychkov et al., J. Non-Cryst. Solids **208** (1996) 1.

[2] E. Bychkov, D.L. Price, C.J. Benmore, and A.C. Hannon, Solid State Ionics **154-155** (2002) 349.

POSTER 57

Viral Assemblies at High Pressure

K. Brister,¹ R. Fourme,² H.-K. Mao,³ J.E Johnson,⁴ and T. Lin⁴

¹The University of Chicago, Argonne, IL 60439, USA

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⁴The Scripps Research Institute, La Jolla, CA 92037, USA

Diffraction from the icosahedral virus Cowpea Mosaic Virus (CPMV) can be dramatically improved when pressure is applied to the crystals: when the pressure is raised to 3.5 kbar a resolution of 2.1 Å has been seen from a sample that gives 3.0 Å resolution at lower pressures. This method, besides having an intrinsic scientific interest, would be of great value in x-ray crystallography if shown to be generally applicable.

Using the facilities of HP-CAT (APS sector 16), we have reproduced these results previously obtained at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, and have extended these studies with data sufficient for the structural study of the pressure effects on the CPMV structure. This opens the way for in-depth studies for application of this method to virus and protein crystallography.

POSTER 58

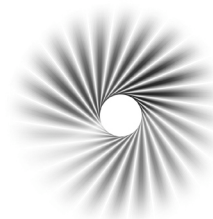
X-ray Study of Monolayers of N-Alcohols and Carboxylic Acids at Oil-Water Interface

A.M. Tikhonov,¹ M.L. Schlossman,² and S.V. Pingali²

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N-alcohols and carboxylic acids are slightly soluble in n-hexane and adsorb at the oil-water interface as a monolayer. As temperature increases, the monolayer undergoes 2nd-order phase transition. X-ray reflectivity is used to study the structure of the low- and high-temperature phases. It appears that for the C30-alcohol, this transition is from the liquid phase to the gas. On the other hand, it is from solid phase to liquid phase for the C30-acid. Temperature dependence of the reflectivity allowed reconstructing the interfacial coverage by surfactant and establishing the parameters of the Marchenko's theory for the critical transition at the liquid-liquid interface.



POSTER 59

Development of Submicron-Resolution 2D and 3D X-ray Structural Microscopy

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We have developed 2D and 3D x-ray structural microscopy techniques with submicron point-to-point spatial resolution and high angular resolution on the UNICAT beamline (sector 34). Polychromatic x-rays are focused using elliptical Kirkpatrick-Baez mirrors, Laue patterns are collected using a CCD area detector, and subsequent computer analysis provides structural maps of the phase, grain orientations (texture), and the local stress/strain tensor. These microdiffraction capabilities have enabled 2D and 3D structural studies of materials on mesoscopic length scales of tenths-to-hundreds of microns. For thin samples, such as deposited films, 2D structural maps are obtained by step-scanning the area of interest. Two-dimensional x-ray microscopy has been applied in studies of the epitaxial growth of oxide films on metal substrates [1]. For bulk samples, a 3D differential-aperture x-ray structural microscopy (DAXM) technique has been developed that provides the full diffraction information from each submicron volume element [2]. The utility of 3D x-ray microscopy has been demonstrated with measurements of intra- and intergranular orientations and morphology in polycrystalline aluminum. This capability provides the previously lacking, direct link between the experimentally measured 3D microstructure and the results of theory and modeling of materials processes on mesoscopic length scales.

[1] Budai et al., *Nature Mat.* **2**, 487 (2003).

[2] Larson et al., *Nature* **415**, 887 (2002).

Work at the APS supported by the DOE Office of Science, DMS under contract with ORNL, managed by UT-Battelle, LLC; UNICAT is supported by UIUC, ORNL, NIST and UOP Res., Inc.

POSTER 60

Microstructure and Evolution of Materials on Mesoscopic Length Scales

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Essentially all technological and biological materials are inhomogeneous on mesoscopic length scales of tenths-to-hundreds of microns, and features, such as grain sizes or precipitates, often control critical materials properties. Three-D x-ray structural microscopy techniques have been developed recently to probe the lattice structure, orientations and elastic and plastic strain within bulk polycrystalline materials [1]. This polychromatic x-ray microdiffraction approach uses a differential-aperture x-ray microscopy (DAXM) technique similar to a traveling pinhole to achieve submicron 3D point-to-point spatial resolution. Results of two recent DAXM studies at the UNICAT beamline (sector 34) will be presented. First, measurements of the 3D deformation microstructure introduced by nano- and microindents in Cu single crystals reveal the local lattice rotations and the geometrically necessary dislocation (GND)

density patterns. Results for different tip-shape geometries will be compared. Second, *in situ* studies of 3D grain growth in polycrystalline aluminum have been initiated, and results showing the evolution of grain boundaries during thermal annealing will be presented. In the future, experimental information provided by these studies will be used in collaboration with materials theory and large-scale computer modeling efforts to obtain a fundamental understanding of microstructural evolution during materials processing.

[1] Larson et al., Nature **415**, 887 (2002).

Work at the APS supported by the DOE Office of Science, DMS under contract with ORNL, managed by UT-Battelle, LLC; UNICAT is supported by UIUC, ORNL, NIST and UOP Res., Inc.

Condensed Matter Physics

POSTER 61

X-ray Microdiffraction from Ferroelectric Domains in BaTiO₃

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We present the first results from the x-ray microprobe at station 8-ID-E of the Advanced Photon Source, Argonne National Laboratory. Using the high coherence of the 8-ID line and a Fresnel zone plate, a focal spot as small as 0.4 microns in the vertical and 3 microns in the horizontal is obtained. This combined with a feedback-controlled XYZ translation stage with 40 nm step size over a 25 mm travel range allows x-ray microdiffraction measurements with a high degree of precision and efficiency. As an illustration, we present results imaging the (100)/(001) Bragg peak from 90 degree ferroelectric domains in barium titanate. The domain pattern consists of alternating stripes of a and c domains with an average domain separation of ~10 microns. This agrees well with lower resolution tomographic data taken in parallel with a near-field CCD setup using the unfocused beam to image the diffracting region. With these data, we discuss strain fields, dislocation, and pinning near the domain walls.

Materials Science

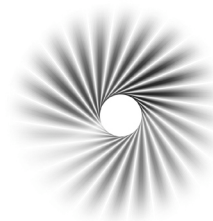
POSTER 62

Yield Strength and Strain-Hardening of the Deformed Sample under High Pressure

T. Uchida, Y. Wang, N. Nishiyama, M. Rivers, and S. Sutton

CARS, The University of Chicago, Argonne, IL 60439, USA

Yield strength of material is of fundamental importance in many fields, including mechanical engineering, material science, and mineral physics. Combining a newly developed high-pressure deformation module with high-resolution monochromatic x-ray diffraction, quantitative deformation experiments can be performed under high-pressure and high-temperature conditions. Elastic lattice strains measured from distortion of the diffraction Debye rings can be used as a stress gauge. Axial strain, which contains both elastic and plastic components, is obtained from sample axial length in radiographic image of the cell assembly. By carefully examining the relation between lattice strain and



axial strain, initial yield point can be identified as the deviation from linearity. Peak width of diffraction profile also provides information about yield point at which the width saturates. Results obtained for cylindrical polycrystalline MgO samples revealed that MgO yielded at low axial strains ($< 1\%$) and the yield strength (ca. 0.5 GPa) was insensitive to pressure up to 7 GPa. Beyond the yield point, MgO showed a strong strain-hardening behavior, characterized by an initial rapid increase in flow stress (differential stress beyond yield point), followed by a linear stage with a constant hardening parameter.

Diffraction

POSTER 63

Combined Angle- and Energy-Dispersive Structural Analysis and Refinement

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²INPS, Argonne National Laboratory, Argonne, IL 60439, USA

We have developed a new step-scan diffraction technique, which collects angle-dispersive data using an energy-dispersive setup. By scanning a well-calibrated Ge solid-state detector with 2θ angles at 0.01 degree steps, a series of 1-dimensional (1-D) energy dispersive data (intensity vs. energy) are obtained as a function of 2θ . The entire data set is then regrouped according to photon energy, which is defined in the multichannel solid-state detector as individual channels, yielding 2048 1-D angle-dispersive datasets (intensity vs. 2θ), each of which corresponds to a given photon energy or wavelength. The entire dataset or selected partial datasets can be used for Rietveld refinement. This technique is useful for any diffraction studies that have limited access to the sample. Advantages involve (1) this technique does not require a monochromator and high-energy photons can be used to refine angle-dispersive data, (2) collimators help to eliminate background noise, and (3) data from several energies can be binned to increase 2θ coverage, hence scan steps can be significantly increased, enabling less time consuming step-scan. We will discuss the details of these advantages using the data collected on Al_2O_3 (a NIST diffraction standard) at ambient condition, with varying schemes for data treatment.

Materials Science

POSTER 64

In situ Studies of Early-Stage Copper Oxidation

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Using a new environmental chamber at BESSRC sector 12-ID, we are investigating the early-stage oxidation behavior of the Cu (001) surface. Using our ability to monitor oxide growth and reduction at temperature, in controlled $p\text{O}_2$, we find several strain- and size-induced effects on oxidation that differ from expectations based on macroscopic theory and previous ex situ, later stage experimental studies. For example, we find that the critical oxygen partial pressure delineating the thermodynamic limit between oxide growth and reduction is more than nine orders-of-magnitude larger for thin-film samples than predicted by bulk phase equilibria. We also observe large reversible changes in the oxide lattice parameter of up to 0.5% in response to controlled variations in oxygen partial pressure in the chamber. While to date we have focused on studies of oxidation, in this poster we will also briefly discuss the

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potential uses of our environmental chamber in future studies of many other types of gas-solid interactions, including studies relevant to hydriding behavior, gas sensing, surface photochemistry, and heterogeneous catalysis.

Chemistry

POSTER 65

X-ray Absorption Spectroelectrochemistry of f-Element Coordination and Redox Behavior in Heteropolyoxoanion Complexes

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Inorganic nanoanions with P-W-O framework structures known as hetero-polyoxometalates are of current practical interest and of future promise in catalysis, electroanalytical, and separations sciences. Unlike more familiar ligands (e.g., water, halides, carbonates), many heteropolyoxoanions are electroactive and, notably, function to stabilize otherwise reactive oxidation states of f-block (i.e., lanthanide and actinide) ions. Despite considerable research, there is a dearth of information about this useful behavior and about general aspects concerning the coordination and electrochemical response of f-ions in molecular clusters with the Wells-Dawson and Preyssler anions. We have utilized the combination of specialized facilities available at Argonne, including the laboratory infrastructure of the Actinide Facility and the Basic Energy Sciences Synchrotron Radiation Center, to address these issues. Through the multifaceted use of electrochemistry and in situ x-ray absorption spectroscopy, we are advancing the understanding of f-ion redox speciation in solution complexes with heteropolyanions a task that is not possible with conventional experiments. Our approach, purpose-built equipment, and results will be presented in light of their implications to studies of other systems of fundamental and functional interest in which electrons are involved. *This work is supported by the U.S. DOE OBES, Chemical Sciences, under contract W-31-109-ENG-38.*

Time-resolved Studies

POSTER 66

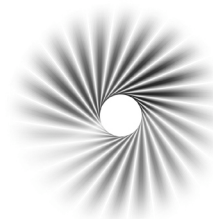
Fast Modulation of a Synchrotron X-ray Beam with Standing Acoustic Waves

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Time-resolved x-ray diffraction measurements from a quartz crystal under a 19.6 MHz acoustic wave excitation were performed by synchronizing the acoustic wave and the APS x-ray burst periodicity. We used the fact that, twice per period, the acoustic standing wave produces a zero net deformation across the crystal thickness. By introducing an oscillating delay to the acoustic excitation, we were able to effectively change the phase of the wave relative to the x-ray burst periodicity. The x-ray diffraction intensity was strongly affected by tuning the timing of the x-ray arrivals to the minimum or maximum of the acoustic deformation. A deep modulation of x-ray intensity was observed in a wide frequency range between 0.1 Hz and 1 Mhz, which demonstrates that acoustically excited quartz crystals can potentially be used as slow and fast x-ray modulators and choppers with high duty rate cycles.



POSTER 67

Optimizing Channel-Cut Monochromator Designs for Undulator Sources

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Northwestern University, Argonne, IL 60439, USA

Hard x-ray monochromators on synchrotron beamlines often employ a double-crystal design in order to return the monochromatic beam to a direction parallel to the white beam. Monolithic channel-cut and separated crystal designs are used, and both methods have their advantages and disadvantages. While a channel cut excels in simplicity, as well as thermal and mechanical stability, its compromises include potential contamination of the output beam from harmonics and Laue spots that propagate through the device. This study focuses on optimizing the parameters of channel-cut construction under the constraints given by a typical undulator and propose design choice solutions that would be appropriate for a beamline that uses a post-monochromator-defining slit and a mirror for Laue spot and harmonic rejection.

Instrumentation

POSTER 68

Adaptive Optics for a High-Resolution X-ray Fluorescence Analyzer

B. Adams, K. Attenkofer, M. Wiedenhoef, and M.A. Beno

Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA

A bent-crystal x-ray fluorescence analyzer based upon adaptive optics was developed and tested. Currently, its energy resolution is ca. 6 eV but, eventually, better than 1 eV should be reached. This resolution is achieved by bending a silicon crystal into the shape of a logarithmic spiral, using a pair of main benders and up to 8 correction actuators. The shape can be readily adjusted to match any x-ray wavelength, and other shapes (such as elliptic) are also possible. The analyzer was developed for the purposes of ultrafast laser pump, x-ray probe spectroscopy of carrier dynamics in GaAs. It should, however, be useful in x-ray near-edge spectroscopy in general.

Imaging

POSTER 69

Phase Contrast Microscopy

D. Paterson, Y. Chu, S. Vogt, B. Lai, and I. McNulty

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We continue to develop powerful new phase contrast techniques in the X-ray Microscopy Group that uniquely capitalize on the high brilliance of APS x-ray sources. Recent work includes absolute determination of the phase of materials science specimens, such as AFM tips, using coherent full-field imaging, differential phase contrast of weakly absorbing biological samples using configured detectors in scanning x-ray microscopes, and phase contrast enhancement of defects in weakly diffracting matter, such as protein crystals. These sensitive x-ray phase contrast methods, in addition to being advantageous

for imaging weakly absorbing or scattering features with minimum attendant radiation dose, are also useful for fundamental measurements of wave fields.

Chemistry

POSTER 70

Scattering Experiments with High-Energy X-rays on Actinide Ion Solutions

J. Neufeind, L. Soderholm, S. Skanthakumar, M-H. Chiang, and M. Jensen

Argonne National Laboratory, Argonne, IL 60439, USA

This poster presents information obtained by scattering experiments on the environment of actinide ions in aqueous solution as a function of parameters, such as the counter ions present or the pH of the solution. Under these conditions actinide ions are known to form a variety of chemically distinct species. The importance of the environmental chemistry of the actinide elements explains the considerable interest in understanding its complex behavior. Traditionally information on the speciation of actinides has been obtained by spectroscopic methods but also more indirect methods like conductivity or pH measurements. Pair distribution functions determined by scattering experiments are a measure of the probability to find a neighbor atom at a particular distance. The pair distribution functions expected for individual species likely to form during hydrolysis (for example) of an actinide ion are fixed by the bond distances and coordination numbers. Pair distribution functions can provide information for coordination distances up to about 20Å, and coordination numbers can be determined with high precision. Combination of scattering experiments with spectroscopic information can remove ambiguities that persists in the traditional approach. *This work is supported by the U.S. DOE, OBES - Chemical Sciences, under contract W-31-109-ENG-38.*

Other

POSTER 71

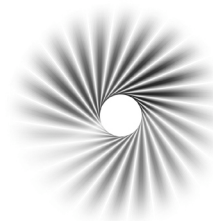
Optimization of the APS Storage Ring Parameters for XPCS Experiments at Beamline 8-ID

A. Sandy,¹ M. Borland,¹ P. Ilinski,¹ S. Narayanan,¹ and L. Lurio²

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²Dept. Physics, Northern Illinois University, DeKalb, IL 60115, USA

Studies to optimize APS storage ring (SR) source parameters for x-ray photon correlation spectroscopy (XPCS) experiments at beamline 8-ID were performed in cooperation with the APS Accelerator Systems Division. In general, the signal-to-noise ratio in an XPCS experiment depends linearly on the speckle contrast, which is proportional to the ratio of coherent-to-incoherent flux and the number of coherent modes illuminating the sample; the contrast is therefore a key figure of merit for XPCS experiments. X-rays were produced by undulator A using, first, the current standard storage-ring lattice and, second, a trial 8-ID-specific low horizontal beta lattice. Speckle patterns were obtained for the different SR lattices by measuring the scattered intensity produced by a disordered aerogel sample. From the speckle patterns, we have extracted the speckle contrast, the number of coherent modes illuminating the sample and the ratio of the coherent-to-incoherent incident fluxes. The contrast measured with the custom lattice increased approximately 8 percent over the value measured with the standard lattice. This is considerably less than the expected increase of approximately 20 percent based on source-size considerations. We attribute this difference to the large, measured ratio of coherent-to-incoherent flux



resulting from imperfect beamline optics. This work demonstrates both the importance of determining a figure-of-merit for a beamline vis-a-vis the SR lattice and, more importantly, that the SR lattice can be customized at a beamline-specific level to optimize that figure-of-merit.

Diffraction

POSTER 72

Studies on Microstructures of Polyhedron Lead Dots by X-ray Microdiffraction

Y. Xiao,¹ A. Tkachuk,¹ Y. Chu,¹ Z. Cai,¹ Z. Xiao,² C.Y. Han,² and W.-K. Kwok²

¹Advanced Photon Source and ²Material Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

The lattice architectural structures of lead polyhedron particles depend on their geometries and morphologies. The crystal structures of lead decahedron and icosahedron particles (several microns) synthesized with electro-deposition were studied with x-ray microdiffraction at the 2ID-D station of the Advanced Photon Source. X-ray high-resolution (down to 200 nm) diffraction mappings at several reciprocal points against the facet and geometrical shape of the particles were performed to establish the relation of the lattice orientations to the facets of the polyhedron crystals. The x-ray microdiffraction results show that the crystallographic structure of the polyhedron particles is face-centered cubic and contains multi-twin structures. The tetrahedron unit block that composes a polyhedron particle is not necessarily a perfect single crystal, it often contains a mosaic structure whose orientation depends on the relative orientation to the crystal boundary.

Biology

POSTER 73

Detection and Mapping of Trace Elements in Biological Materials using Correlative X-Ray and Electron-Probe X-Ray Microanalysis

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Assessment of drug and cell interactions during the drug development process requires knowledge of both the effect of the drug on metabolism and physiology and the location of the drug within the cell. Drugs containing nonbiological moieties can be tracked intracellularly by two complementary techniques, x-ray probe x-ray microanalysis (XPXMA), which has extremely high sensitivity of detection (≤ 10 nmoles/mg), and electron-probe x-ray microanalysis (EPXMA), which has extremely high spatial resolution (~ 10 nm). We have tested the efficacy of examining the same specimens by these methods in three cell culture systems: MCF-7 human breast cancer cells, PC12 rat pheochromocytoma cells, and parasitic protozoan hemoflagellates (*Leishmania donovani*). Our goals in these cases were to determine the intracellular location of (1) biologically active nanocomposites targeted to the mitochondria or nucleus of cells and (2) an antimony-containing drug used in treatment of *Leishmania* parasites and for which the mechanism of action is unknown. Our results prove the utility of both techniques in tandem. The XPXMA revealed the presence of titanium inside cells, while EPXMA

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determined that the Ti-nanocomposite reached the targeted organelles. The XPXMA detected the presence of Sb at varying levels from cell to cell, while EPXMA revealed a barely detectable presence of Sb in the cytoplasm of some parasites. These tools used together will allow testing of hypotheses relevant to the mechanisms of action of the drugs. *Work supported by the U.S. Department of Energy under Contract no. W-31-109-ENG-38.*

Condensed Matter Physics

POSTER 74

Nanoscale Inhomogeneities in an Optimally Doped YBCO Superconductor

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We present unambiguous and robust evidence of a coherent coexistence of short-range, imperfectly ordered modulated regions with the average orthorhombic lattice in an optimally doped yttrium-barium-copper-oxide superconductor. These regions are quasi-2-dimensional and are < nanometers in extent within the Cu-O planes. In these regions, the lattice is strongly modulated with a periodicity of 4 unit cells, $q=(1/4, 0, 0)$; a periodicity that has manifested in earlier spectroscopic and neutron scattering measurements on related copper oxides as well. Now, for the first time, using high-energy diffraction techniques, we have determined the exact nature of these modulated regions: in these regions Cu, Ba, and O, atoms, respectively, are strongly displaced, in a mutually correlated fashion, relative to their average sites. This pattern is consistent with the presence of nanodomains of the oxygen-ordered Ortho-IV phase with the same periodicity. Intriguingly, these nanodomains are elastically “softer” than the average lattice, and they induce long-range strain throughout the host. However, the relevance to superconductivity of our observations remains unclear.

Biology

POSTER 75

Use of TiO₂-Oligonucleotide Nanocomposites for Intracellular DNA Targeting

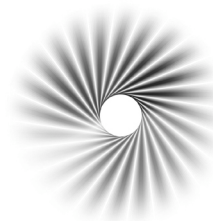
T. Paunesku,¹ S. Vogt,² N. Stojicevic,¹ J. Maser,² B. Lai,² T. Rajh,³ M. Thurnauer,³ and G.Woloschak¹

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Previous work from our groups has lead to the development of hybrid nanocomposites made up of DNA oligonucleotides bound via dopamine to TiO₂ nanoparticles (4.5 nm in size, surface coated with glycidyl isopropyl ether). Even as a part of the nanocomposites, DNA oligonucleotides retain base-pairing specificity, while the TiO₂ nanoparticles exhibit characteristic photoreactivity; henceforth excitation of TiO₂ by exposure to electromagnetic radiation of energy above 3.2 eV results in charge separation that ultimately results in scission of the DNA. This endonuclease activity is: i) excitable; ii) highly sequence specific, even to the degree that it can be directed towards a single target in a whole genome. DNA is



located in cells in two compartments—nucleus and mitochondria. In order to prove the ability of nanocomposites to migrate into both of these cellular compartments and specifically target individual DNA sequences, we did a series of test experiments with cultured cells. We mapped the intracellular distribution of nanocomposites using the hard x-ray fluorescence microprobe at the 2-ID-E beamline. Different DNA sequences attached to nanocomposites clearly resulted in different intracellular localizations, and experiments with variable lengths of cellular exposure to nanocomposites resulted in different amounts of intracellular titanium signal.

High Pressure

POSTER 76

Phase-Locked and High-Pressure X-ray Magnetic Circular Dichroism Measurements on Beamline 4-ID-D

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Two recent advances in the collection of x-ray magnetic circular dichroism (XMCD) spectra on beamline 4-ID-D have opened up new possibilities for magnetic research at the APS. The XMCD spectra at hard x-ray energies (>3 keV) are typically quite small ($<1\%$). Measuring such small difference spectra poses significant experimental challenges, since systematic noise sources in the data collection process can often be much larger than signals themselves. Using a phase-locked detection scheme, however, we have recently measured XMCD spectra with difference signals down to $1e^{-5}$. The ability to measure such small differences opens up the possibility of using helicity-modulated EXAFS spectra to obtain the local magnetic environment around an atom. Second, instrumentation for taking XMCD spectra under high pressure (up to 100 Gpa) has recently been developed. Currently there are few techniques capable of probing the magnetic properties of a material under such high pressures. As such, high-pressure XMCD measurements can offer unique information not accessible via other techniques. Details on the design of the optics and pressure cell will be presented.

Materials Science

POSTER 77

Mesoporous Silica: Pore-Size Tuning and Active Interface for Biosensing

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Mesoporous material has application in membranes, catalysis, low-dielectric-constant material, photonics and sensors. Mesoporous silica can also be used as support for ion-channel-based biosensors, creating an ionic reservoir mimicking cytosol. Ion transport through these channels is very precise and has excellent gating mechanisms. We have investigated bilayer formation on a mesoporous silica support via GISAXS, AFM and patch-clamp techniques. Synthetic ion channels with wide-ranging pore sizes have application in DNA sequencing, proteomics and biosensing. To realize these and future applications of mesoporous silica, we are creating a library of silica-film fabrication procedures with

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pore sizes ranging from 1.6 nm-50 nm. Towards this goal, we have used cationic, nonionic and block-copolymer surfactants with cosolvents, expanders, etc., as means to create desired pore sizes. We will present our results on pore-size tuning and its one application as a lipid bilayer support studied by GISAXS.

Materials Science

POSTER 78

GISAXS Studies of Silica Films Templated with Biocompatible Lipid Mesophases

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Surfactant-templating of sol-gel materials has proven to be a versatile route for engineering porosity in thin silica films. Typically, ionic or non-ionic amphiphilic surfactants are added to silica sols at concentrations less than the critical micelle concentration; during dip- or spin-coating solvent, evaporation increases the surfactant concentration to a point where ordered mesophases (lamellar, cubic, 2D hexagonal, etc.) are formed. After the silica condenses around the surfactant mesophase, removal of the surfactant leaves a porous silica network. An application that has been suggested for these materials is as an entrapment medium for biological components, such as viable cells or functional proteins. The silica matrix would serve to stabilize the biological components, while the open porosity would allow facile mass transport through the film, enabling chemical communication with the external environment.

Unfortunately, most surfactants are not biocompatible, rupturing cell membranes or denaturing proteins. For this reason, we have begun investigating phospholipids, naturally occurring amphiphilic components of cell membranes, as mesophase templates. To better understand the properties of structures that are formed when using these pore network templates, we present here the characterization of lipid/silica mesostructured films using grazing-incidence x-ray scattering (GISAXS) as a function of both lipid structure and lipid/silica ratio.

Materials Science

POSTER 79

Pore-Size Calculation of Mesoporous Silica Thin Films by Small-Angle X-ray Scattering and X-ray Reflectivity Experiments

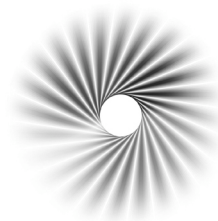
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Silica thin films of various pore sizes are of high interest for potential applications, e.g., in sensors, as membranes, or as low-dielectric-constant films. The estimation of the pore sizes of the silica thin films is not achievable with classic sorption methods due to the small amounts of absorbing material (film) on a relatively heavy support. One possibility to overcome this problem was the invention of the surface acoustic wave (SAW) technique. But this technique is not accessible to the general public. The objective



of this work is to find an independent method for the pore size calculation from x-ray scattering. For this purpose, a highly ordered silica film templated with Brij-56 was investigated before and after calcination. The GISAX patterns of the investigated films show highly ordered films with a cubic fcc structure, which is maintained during the calcination with a distortion perpendicular to the film surface. To determine the pore diameters, both samples were analyzed by x-ray reflectivity. Fitting these data with a suitable model resulted in pore diameters of 5.9 nm for the as-synthesized material and 3.9 nm for the calcined silica thin film. In future work these calculations will be verified by classical sorption methods.

Biology

POSTER 80

Location and Identification of Nanobiomineralization in Brain Tissue Associated with Neurodegenerative Disorders

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Oxides of iron and several other metals occur naturally in several biological systems in the form of nanobiomineral crystallites (~50 nm) that are very widely dispersed. Previous studies by Dobson and others have confirmed the presence of various iron oxides in the brains of pigeons and humans using extractive analysis and magnetic analysis. It has been found that there is a greatly enhanced production of iron minerals associated with neurodegenerative disorders such as Alzheimer's, Huntington's, and Parkinson's diseases (AD, HD and PD), although the mechanism for formation and precise location of the minerals in the tissue (i.e., intra- or extracellular) is not understood. Location and subsequent analysis of the nanobiominerals by conventional methods such as TEM, SEM or magnetic scanning probe microscopy are made nearly impossible by the wide dispersion of the small particles, and the chemical state of iron compounds found in tissue samples is altered by conventional tissue fixing and staining techniques.

We have developed microfocusing and XRF scanning capabilities at the Materials Research CAT (MR-CAT) that now allow us to locate the nanobiominerals in thin (<50 μm) tissue sections. Single nanoparticles can be located in an area of 200 μm x 200 μm , and then localized to within a $\sim 3 \mu\text{m}$ x $\sim 4 \mu\text{m}$ area; XAS analysis is then used to determine the chemical nature of the particle. Using this technique, we have demonstrated the ability to distinguish between ferritin iron storage proteins, iron oxide minerals such as magnetite, and metallic particles. We have also extended this technique to map other minerals of interest, such as Zn, Mn, and Cu. We have also developed fixation techniques that have been shown to maintain the chemical state of the iron compounds of interest. The results from AD, HD, and normal brain tissue show that the technique is a powerful tool for the study of neurodegenerative disorders. Results will be presented showing enhanced accumulation of iron oxides in Alzheimer's plaques, as well as spatially correlated minerals of other metals. TEM of sections of tissue in the vicinity of iron-containing particles is underway.

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POSTER 81**Temperature Evolution of Magnetic Chiral Domains in Dy**J. Lang,¹ D. Haskel,¹ G. Srajer,¹ D.R. Lee,¹ and D. McWhan²¹Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA²NSLS, Brookhaven National Laboratory, Upton, NY 11973, USA

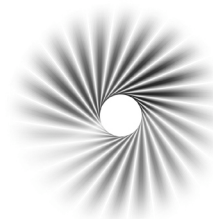
We have measured the temperature dependence of the magnetic chiral domains in Dy metal. Dysprosium has ferromagnetic ordering for temperatures up to 83K and a spiral magnetic structure between 83K and 176K. The spiral structure can be either right or left handed leading to the formation of chiral domains. Dramatic differences are seen in the chiral domain structure when cooling from the paramagnetic phase as opposed to warming from the ferromagnetic phase. Just above the ferromagnetic to spiral phase transition, the cooling domain pattern was characterized by large sharp well-defined domain structures, while the warming domain pattern consisted of smaller more ill-defined domains. Domain wall motion in the spiral phase is seen upon warming sample, with a marked increase in this motion for temperatures above 140K.

*High Pressure***POSTER 82****Pressure-Induced Structural Changes in Amorphous Silicates**

V.B. Prakapenka, G. Shen, M.L. Rivers, S.R. Sutton

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Despite great interest in amorphous silicates, there are only a few experimental reports concerning high-pressure behavior of amorphous silica at pressures limited to 42 GPa. The main reasons for such a “low” high-pressure boundary in these experiments are the weak scattering of noncrystalline silicates in the small pressure chamber of the diamond anvil cell (DAC) compared to the large scattering from the DAC and the small 2-theta opening, which made these studies challenging. In this work, we have expanded the maximum pressure to 70 GPa using a specially modified DAC with x-ray-transparent cubic BN seats that allow us to study structural changes of noncrystalline materials at ultrahigh pressures with a maximum momentum transfer above 100 nm⁻¹. The diffraction patterns were collected in a full solid angle using an online imaging plate detector and high-energy (37.44 keV) brilliant x-rays at the GSECARS beamline (APS). The high-pressure behavior (compress/decompress) of amorphous SiO₂ and MgSiO₃ has been studied up to 70 GPa with step of ~ 5 GPa at room temperature. The observed structural changes in the medium-range order of the amorphous network and kinetics of the pressure-induced amorphous-amorphous transformation in SiO₂ and MgSiO₃ provide a basis for understanding the dynamical properties of high-density noncrystalline materials at lower mantle conditions deep within the Earth.



POSTER 83**Theory of Model Nanomagnetic Systems for Time-Resolved XMCD Experiments**

K. Guslienko

Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

We will present a theory of low-frequency magnetization dynamics in model nanomagnetic systems. Then results of calculations can be used to design and optimize the time-resolved x-ray magnetic circular dichroism (XMCD) experiments at the APS. The collective spin excitations are described in terms of eigenmodes and corresponding discrete eigenfrequencies. The spin-excitation spectra of the magnetic particles are calculated on the basis of the general theory of dipolar-exchange spin waves in the framework of the Green's functions formalism. The following model systems will be considered:

1. Soft magnetic particles in a vortex state. The low-frequency response of such a particle on an external field can be described as a movement of the vortex center over the particle. The characteristic frequency is in 0.1-1 GHz range, and can be adjusted in experiments by varying the particle sizes.
2. Tri-layer films F/N/F with antiferromagnetic interlayer coupling. The developed theory predicts that the frequency of the optical mode of spin-excitation spectra has two minima in sub-GHz range when an in-plane magnetic field is applied.
3. Magnetic multilayer films (nanodots) with perpendicular magnetic anisotropy. The remanent state of such a model system is a stripe domain structure, which responds to an external field by moving the domain walls or by flipping the direction of the domains. The frequency of domain wall resonance and frequencies of domain resonances were found to be in 0.5-5 GHz range for [Co/Pt] multilayers.

Nanoscience

POSTER 84**Probing Spin Dynamics in Magnetic Nanosystems: The Development of a "Time-Dependent" XMCD Experiment at BESSRC**K. Attenkofer,¹ M. Wiedenhoft,¹ V. Novosad,² S.D. Bader,² and K. Yu. Guslienko,³¹*BESSRC-Group, Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA*²*Material Science Division, - Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA*³*Material Science Division, Argonne National Laboratory, Argonne, IL, 60439, USA*

We report on the development of a time-dependent x-ray magnetic circular dichroism (XMCD) experiment at BESSRC-CAT at the APS. The experiment is designed to study the element-specific spin dynamics of magnetic nanoparticles and dot arrays following a pulsed magnetic excitation. The samples of magnetic nanoparticles integrated in gold microcoils were microfabricated using multistep electron-beam lithography and thin-film sputtering at the Material Science Division and the Center for Nanoscale Materials of Argonne National Laboratory. A particular system of interest is [Co/Pt] patterned multilayer dots with perpendicular anisotropy and stripe remanent domain structure. An out-of-plane magnetic field pulse excites the collective spin-motion (domain wall resonance) that can be probed with circular polarized light in the 6-13 keV energy range. We will also present some theoretical predictions of spin excitations in magnetic nanosystems with restricted geometries.

POSTER 85

Refractive Sawtooth Lenses for High-Energy Focusing

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We have recently investigated Si-based refractive lenses at beamline 1-ID for the purpose of vertically focusing high-energy x-rays (E=80 keV) at low source demagnification (~1:0.7). The combined properties of relatively low divergence (due to low demagnification) and high flux should enhance a number of sector 1 programs including time-resolved diffraction and small-angle scattering. Such lenses may also be used for x-ray collimators, in conjunction with high-energy-resolution optics, as has been demonstrated with aluminum cylindrical lenses [1]. The lenses are based on a sawtooth geometry [2] and have a number of desirable properties. First, their profile approximates a single parabolic lens, so they have minimal cylindrical aberration. Second, by adjusting the taper between the top and bottom sets of the teeth, the focal length/beam size can be varied. Third, x-rays passing directly along the optical axis suffer zero absorption. Finally, since they are composed of single-crystal Si, they produce little small-angle scattering and thus a cleaner beam than lenses composed of polycrystalline material. Initial results on lens performance will be presented, together with their application to small-angle scattering.

[1] S.D. Shastri, J. Synchrotron Rad. **11**, 1 (2004).

[2] B. Cederstrom, M. Lundqvist and C. Ribbing, App. Phys. Lett. **81**, 1399 (2002).

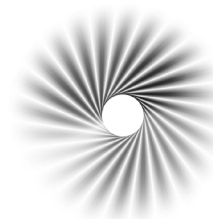
POSTER 86

X-ray Absorption Investigation of Activation of Supported Au Catalysts

H.H. Kung, C. Costello, J. Yang, M. Kung, and Y. Wang

Dept. Chemical and Biological Engineering, Northwestern University, Evanston, IL 60208, USA

Gold catalysts supported on alumina and titania are very active low-temperature CO oxidation catalysts. For samples that are prepared by deposition-precipitation of aqueous HAuCl₄, the process needed to activate these catalysts depends on the support. For alumina-supported samples, heating in a reducing gas at 100°C followed by treatment with moisture at the same temperature is needed. For titania-supported samples, exposure to CO or CO/O₂ mixture at subambient temperature is sufficient. The changes in the samples during activation were characterized by *in situ* XANES and temperature-programmed reduction and by their performance as CO oxidation catalysts. The Au was found to be present as Au(III) in the as-prepared catalysts before activation. Reduction of Au(III) occurred during activation, and the rate and extent of reduction were found to depend strongly on the reducing conditions. The data indicate that zero-valent Au is necessary for catalytic activity, but there is no correlation between the activity and the extent of reduction. There is indication that the catalytic activity is related to agglomeration of the gold metal into small particles, but additional data are needed to confirm this.



POSTER 87

Users' Research Activities at the Sector 5 Bending Magnet Beamline

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The 5-BM-D beamline can deliver to the experimental end station white dipole radiation or monochromatic beam tunable from 4 – 65 keV for normal operation using a water-cooled Si(111) monochromator. It also has a collimating mirror (for harmonic rejection) and a focusing mirror to provide a vertically focused beam in the energy range from 4 – 18 keV. One of the future developments will focus on introduction of a sagittal bender that will focus the beam in the horizontal direction to a beam size of a few hundred microns at the sample position. This will vastly increase the photon flux density. It is indeed a versatile, general-purpose beamline at which research projects range from physics, materials science, catalytic chemistry, electrochemistry, polymer science, environmental science, to biological science. In this poster we would like to present a few examples of the many research projects that are conducted at this beamline to showcase the users' research activities.

Surface Studies

POSTER 88

Studies of the Interface between Two Polar Liquids: Neat and with Electrolytes

G. Luo,¹ S. Malkova,¹ S.V. Pingali,¹ D.G. Schultz,¹ P. Vanysek,² B. Lin,³ M. Meron,³ T.J. Graber,³ J. Gebhardt,³ and M.L. Schlossman¹

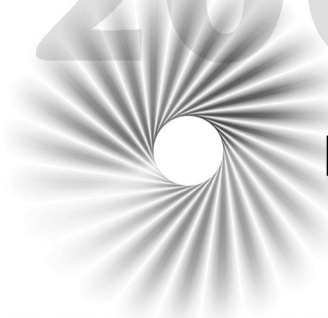
¹Dept. Physics, University of Illinois at Chicago, Chicago, IL 60607, USA

²Dept. Chemistry, Northern Illinois University, DeKalb, IL 60115, USA

³Center for Advanced Radiation Sources, The University of Chicago, Chicago, IL 60637, USA

X-ray reflectivity is used to study the liquid/liquid interface of polar liquids at the sector 15-ID (ChemMatCARS) liquid surface scattering instrument. For the pure nitrobenzene/water interface, we measured the interfacial width at 25°C to be smaller than the value predicted from capillary wave theory. With increasing temperature the roughness approaches the capillary wave value. For 0.01 M tetrabutylammonium tetraphenylborate (TBATPB) in nitrobenzene/X M tetrabutylammonium bromide (TBABr) in water interface (X = 0.0001, 0.01 and 0.05), the interfacial width varies from values below the capillary wave value to values above as the concentration of TBABr increases. Externally biasing the electric potential at the 0.01M TBATPB (nitrobenzene) / 0.1M BaCl₂ (water) interface also allows us to vary the interfacial width. Interpretations of these results in terms of an interfacial bending rigidity and the interfacial double layer will be presented.

2004 USERS



2004 USERS MEETING

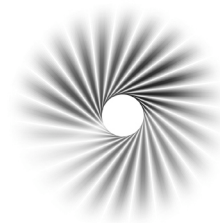
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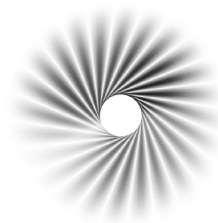
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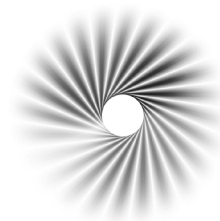
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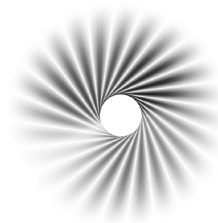
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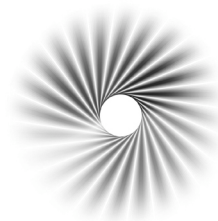


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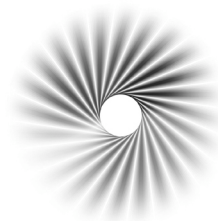
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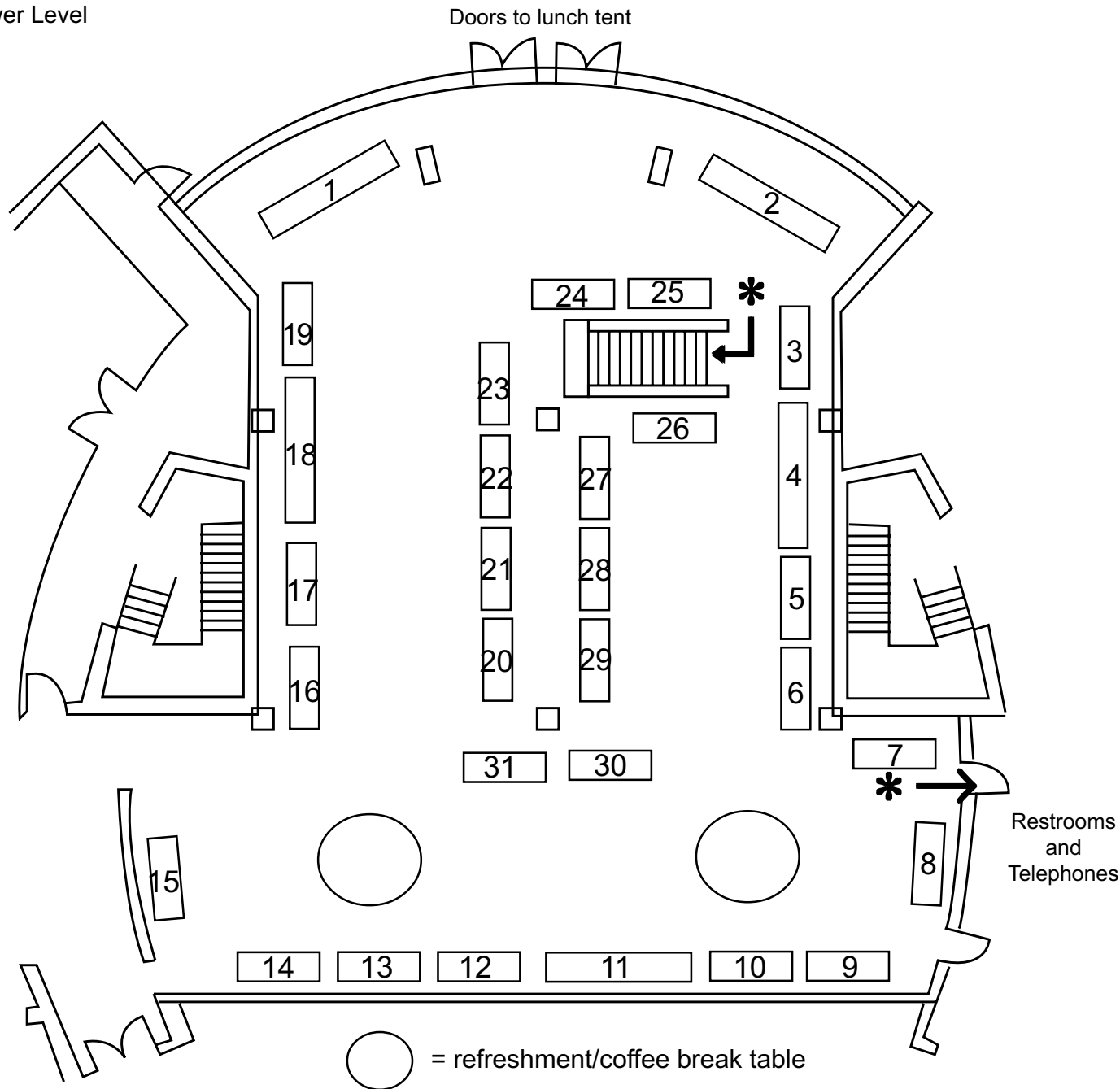
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Vendor Locations—APS Users Meeting 2004

APS Conference Center
Bldg. 402
Lower Level



○ = refreshment/coffee break table

1. Oxford Danfysik/GMW
2. Advent Associates, Ltd.
3. Blake Industries, Inc.
4. Automation Equipment Co.
5. Brush Wellman Inc.
Electrofusion Products
6. TR Electronic
7. Goodfellow Corporation
8. Veeco Instruments
9. Varian Vacuum Technologies
10. Meggitt Safety Systems, Inc.
11. Minarik Corp./Nanomotion Inc.
12. Rigaku
13. Instrument Design Technology

14. RSI
15. Fluidigm Corporation
16. Midwest Vacuum, Inc.
17. ACCEL Instruments GmbH
18. Vacuum One/Norcal
19. Mar USA, Inc.
20. X-ray Instrumentation Assoc.
21. New Focus, Inc.
22. Televac
23. Newport Corporation
24. Advanced Control Systems Inc.
25. Pfeiffer Vacuum
26. TecRep Corporation
27. Johnsen Ultravac Inc.

28. Canberra Industries, Inc.
29. A&N Corporation
30. Omicron NanoTechnology USA
31. Kurt J. Lesker Company

***UPSTAIRS/TRIUM EXHIBITS PLUS
REFRESHMENT TABLES**

32. Bruker AXS Inc.
33. MDC Vacuum Products Corp.
34. Kohzu America, Inc.
35. Alan Burrill Technical Sales
36. Xradia, Inc.
37. Oregon Micro
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2004 USERS MEETING FOR THE ADVANCED PHOTON SOURCE

	Monday — May 3			Tuesday — May 4			Wednesday — May 5			Thursday — May 6		
	What	When	Where	What	When	Where	What	When	Where	What	When	Where
Morning	CNM Meeting	8:30 – 12:30	402 Lect. Hall	WK 1	8:50 – 11:50	402 E1100/ E1200	Users Meeting — Opening Session	9:00 – 12:10	402 Lect. Hall	Users Meeting — User Science Session 2	8:30 – 12:00	402 Lect. Hall
				WK 2	8:45 – 12:00	460 Conf. A						
				WK 3	8:30 – 12:00	401 A1100						
				WK 4	8:50 – 12:40	362 Auditor.						
				WK 5	9:00 – 12:00	402 Lect. Hall						
Noon	Lunch	12:00 – 1:30	Tent	Lunch	12:00 – 2:00	Tent	Lunch	12:00 – 1:30	Tent	Lunch	12:00 – 1:30	Tent
Afternoon	CNM Meeting	1:30 – 4:30	402 Lect. Hall	WK 1	1:30 – 4:30	402 E1100/ E1200	Users Meeting — User Science Session 1	1:30 – 4:30	402 Lect. Hall	WK 6	1:30 – 6:30	402 Lect. Hall
				WK 2	1:30 – 4:45	460 Conf. A						
				WK 3	1:00 – 4:00	401 A1100						
				WK 4	2:00 – 5:30	362 Auditor.						
				WK 5	1:30 – 5:00	402 Lect. Hall						
				WK 7	2:00 – 5:00	402 E1100/ E1200						
				WK 8	1:30 – 5:00	401 A1100						
Evening	Joint APS/CNM Reception	4:00 – 6:00	402 Gallery	Poster Session	4:00 – 6:00	Sector 32	Poster Session	4:00 – 6:00	Sector 32			
	CNM Banquet	6:30 – 9:30	Guest House	Partner User Council	6:30 – 8:30	617	Banquet	6:45 – ???	Guest House			