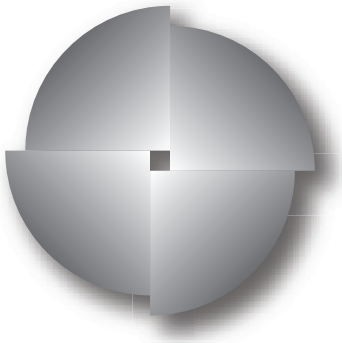
Argonne NATIONAL LABORATORY



2006 USERS MEETINGS

for DOE/BES User Facilities at Argonne National Laboratory

PROGRAM AND ABSTRACTS

May 1-5, 2006

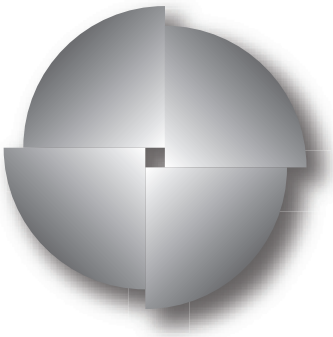
Advanced Photon Source
Center for Nanoscale Materials
Electron Microscopy Center
Intense Pulsed Neutron Source

Argonne National Laboratory
Argonne, Illinois USA

THE UNIVERSITY OF CHICAGO

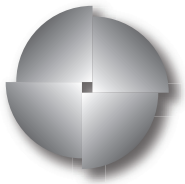
Office of Science
U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by The University of Chicago



2006 USERS MEETINGS

for DOE/BES User Facilities at Argonne National Laboratory



Advanced Photon Source

Gene Ice, Oak Ridge National Laboratory (chair)
Keith Brister, Northwestern University
Millie Firestone, Argonne National Laboratory (poster prize chair)
Thomas Gog, Argonne National Laboratory
Ward Smith, Argonne National Laboratory
APS User Organization Nominating Committee
Stephan Ginell, Argonne National Laboratory (Chair)
Thomas Gog, Argonne National Laboratory
Tim Graber, University of Chicago
David Reis, University of Michigan

Organizers

Susan Strasser, Meg Vigliocco-Hagen

Center for Nanoscale Materials

John A. Rogers, University of Illinois at Urbana-Champaign (Chair)
Paul G. Evans, University of Wisconsin-Madison
Vitali Metlushko, University of Illinois at Chicago
Nicolaie Moldovan, Northwestern University
Stephen Streiffer, Argonne National Laboratory
Gayle E. Woloschak, Northwestern University

Organizers

Carrie Clark, Joanne Day, Sharon Gunter, Rachel Miralez

Electron Microscopy Center

Dean Miller, Electron Microscopy Center, Argonne National Laboratory (Chair)
Russell Cook, Electron Microscopy Center, Argonne National Laboratory
Yasuo Ito, Northern Illinois University
Bryan Rabatic, Center for Nanoscale Materials, Argonne National Laboratory

Intense Pulsed Neutron Source

Thomas Koetzle, Argonne National Laboratory

Organizer

Maria Heinig

Overall Meeting Support and Logistics

Meeting Coordinator	Meg Vigliocco-Hagen
Registration and Web Site	Linda Carlson
Registration Support	Sharon Fisher
Workshop and Poster Support	Rachel Reed
Program Book and Web Site	Jane Andrew
Program Book Editorial Support	Susan Picologlou
Vendor Exhibits	Connie Vanni, Lori Moore
General Meeting Support	Lori Moore
Conference Services	Joan Brunsvold, Marianne Adair, Judy Benigno
Building Arrangements	Bob Whitman, Ed Russell, Becky Tasker
Shipping	Steve Downey
Social Events	Carmie White

Sponsors

Advanced Design Consulting USA, Inc. — General meeting support

MDC Vacuum Products/Insulator Seal — Tuesday poster session

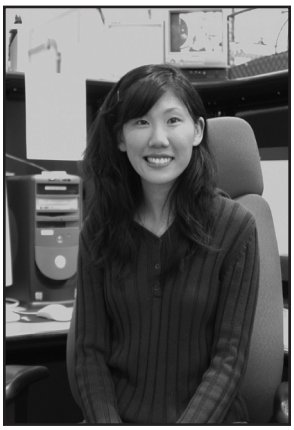
Merck Research Laboratories — General meeting support

Oxford Danfysik — $500 Student Poster Prize

Veeco Instruments — $500 Student Poster Prize

About Argonne National Laboratory

Argonne is a U.S. Department of Energy laboratory managed by The University of Chicago under contract W-31-109-Eng-38. The Laboratory's main facility is outside Chicago, at 9700 South Cass Avenue, Argonne, Illinois 60439. For information about Argonne, see www.anl.gov.



2006 Rosalind Franklin Young Investigator Award

Wendy L. Mao
Los Alamos National Laboratory

The Advanced Photon Source (APS) Users Organization is pleased to announce that Wendy L. Mao is the recipient of the 2006 Rosalind Franklin Young Investigator Award.

The $1000 award recognizes important technical or scientific accomplishment by a young investigator that depended on, or is beneficial to, the APS. Dr. Mao will receive this award on May 3 at the 2006 APS Users Meeting, at which she will also present her work.

Mao received her bachelor's degree from MIT in 1998 and her Ph.D. from the University of Chicago in 2005. She is currently a J. Robert Oppenheimer Postdoctoral Fellow at Los Alamos National Laboratory.

Mao has made contributions to an exceptionally broad range of topics, from the structure of graphite under pressure, to the properties of iron-rich materials at the boundary between the Earth's core and mantle, to the synthesis and characterization of a new family of hydrogen storage materials based on molecular compounds.

In her work on graphite, Mao looked at a half-century-old problem of the behavior of graphite when it is compressed at room temperature. It turns, not into diamond, but into an unusual and unexpected phase. The structure and bonding of this phase remained stubbornly enigmatic because existing techniques could not probe electronic structure at high pressure. At the APS, Mao applied x-ray Raman scattering (XRS) spectroscopy to map the detailed changes in the carbon K-edge that give a signature of the bonding. For graphite at high pressure, XRS revealed an unusual bonding arrangement that explained the material's properties. An additional hydrostatic x-ray diffraction study identified its structure. The high photon flux of the APS made the XRS technique feasible in this case. In fact, with this application of XRS, Mao has opened a new field—investigation of the electronic structure of light elements under pressure.

As Mao explains, "A big strength of the APS is that there are so many techniques here that can all be applied to the same sample. Our work on graphite under pressure is a good example of an old problem waiting for the right new technique to come along."

Wendy later exploited the strange properties of this graphite material—its hardness is both pressure-dependent and reversible—for experimental hardware that helped her achieve new results at the APS in a different field: studies of deep-Earth minerals. In that work, she discovered an iron-rich silicate that occurs at deep-mantle conditions. The material properties of this new phase may explain the strange seismic features of the D" zone—the lower 300 kilometers of the mantle, just outside the Earth's molten core.

Finally, in her current work at Los Alamos, Mao studies the structure of hydrogen clathrates, materials proposed for storing hydrogen for energy applications. Starting from a structure that was stable only at relatively high pressure, she found that with a slight decrease in pressure, the structure transformed into one that then remained stable at ambient pressure and liquid nitrogen temperature. Synchrotron studies helped identify

the phase and monitor its stability. This work is also relevant to planetary studies, as such processes might also occur on the gaseous planets.

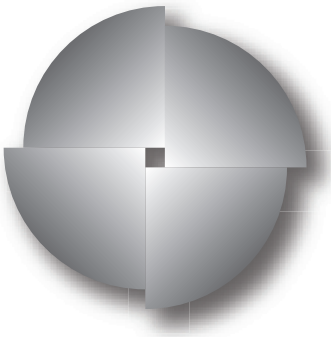
All of Mao's science seems to spring from her curiosity about how "ordinary," abundant elements can change their properties so fundamentally at extraordinary conditions. She brings this perspective to all her scientific questions. "Pressure is another dimension for changing material properties. In our daily life we forget that most of the material on Earth is at high pressure. It's not like what we see on the surface!"

Selected Publications

Wendy L. Mao et al., "Bonding Changes in Compressed Superhard Graphite," *Science* **302**, 425–427 (2003).

Wendy L. Mao et al., "Iron-rich silicates in the Earth's D" layer," *Proc. Natl. Acad. Sci.* **102**(28), 9751–9753 (2005).

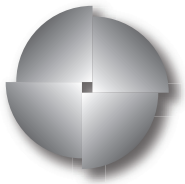
Wendy L. Mao et al., "Hydrogen Clusters in Clathrate Hydrate," *Science* **297**, 2247–2249 (2002).



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Facility Information



Advanced Photon Source

Web site: http://aps.anl.gov
User contact: apsuser@aps.anl.gov, 630-252-9090

Mission Statement

The Advanced Photon Source (APS) at Argonne National Laboratory is the Western Hemisphere's only third-generation high-energy x-ray source. Funding for the APS is provided by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences. The mission of the APS is to deliver world-class science and technology by operating an outstanding synchrotron radiation research facility accessible to a broad spectrum of researchers.

Goals

- Operate a highly reliable third-generation synchrotron x-ray radiation source,
- Foster a productive environment for conducting research,
- Enhance the capabilities available to users of the APS facility,
- Assure the safety of the facility users and staff and the environment,
- Maintain an organization that provides a rewarding environment that fosters professional growth, and
- Optimize the scientific and technological contribution to the Department of Energy and society from research carried out at the APS.

Source Information and Primary Instrumentation

The APS light source uses a 7-GeV accelerator complex (electron gun, 325-MeV linear accelerator, 325 MeV-7 GeV booster synchrotron, 7 GeV storage ring) to produce extremely bright, tunable x-ray beams for researchers to carry out frontier experimentation in areas of science including biology, chemistry, engineering, environmental science, geosciences, materials science, medicine, and physics.

The APS storage ring lattice is optimized for the use of insertion devices (rows of alternating-pole permanent magnets) that can produce x-rays whose energies are tunable from several kiloelectronvolts and higher. These insertion devices maximize flux and brightness, those x-ray beam qualities that are needed for frontier experimentation. Several types of insertion devices are used: undulators with periods of 2.7 cm, 3.00 cm, 3.30 cm, 3.50 cm, and 5.50 cm; a 12.8-cm-period circularly polarized undulator; and a 16.0-cm-period elliptical multipole wiggler.

The APS also provides bending magnet radiation in an energy range of 1 keV to 100 keV. The APS storage ring routinely operates in a top-up mode (an essentially constant-current mode) whereby the ring is refilled with electrons every two minutes. Several special operating modes are also offered to meet the needs of particular user programs.

Staffing and Users

The APS comprises three divisions—X-ray Science Division, APS Engineering Support Division, and Accelerator Systems Division—which are overseen by the Office of the Director of the Advanced Photon Source. All reside under the Argonne Associate Laboratory Director for Scientific User Facilities. As of March 2006, the APS staff numbered 475. In fiscal year 2005, more than 3,000 users visited the APS to carry out more than 2,600 experiments and published more than 1,000 papers.

Center for Nanoscale Materials

Web site: http://nano.anl.gov
User contact: 630-252-8875

Overview

The Center for Nanoscale Materials at Argonne National Laboratory is a joint partnership between the U.S. Department of Energy (DOE) and the State of Illinois, as part of DOE'S Nanoscale Science Research Center program. The CNM will serve as a user-based center, providing expertise, tools and infrastructure for nanoscience and nanotechnology research. The CNM's mission includes supporting basic research and the development of advanced instrumentation that will help generate new scientific insights and create new materials with novel properties. The existence of the CNM, with its centralized facilities, controlled environments, technical support, and scientific staff, will enable researchers to excel and significantly extend their reach.

CNM researchers will work at the leading edge of science and technology to develop capabilities and knowledge that complement those of industry. The challenges the CNM faces involve fabricating and exploring novel nanoscale materials and, ultimately, employing unique synthesis and characterization methods to control and tailor nanoscale phenomena. The unique capabilities of Argonne's Advanced Photon Source (APS) play a key role. APS's hard X-rays, harnessed in a nanoprobe beamline, will provide unprecedented capabilities to characterize extremely small structures.

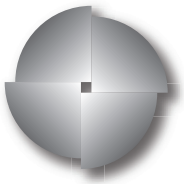
Argonne's long-standing culture of outreach to, and inclusion of, the academic and industrial communities will help support regional and national goals and strategic interests. The CNM welcomes outside users, both as independent investigators and as collaborators, from a wide range of scientific fields. This accessibility ensures a cross-disciplinary approach to nano-related research that will help ideas and activities to cross-pollinate, mature, and evolve over time into the pathways of scientific investigation and discovery that will help shape the future of our society.

User Capabilities

This list summarizes the capabilities, tools, and facilities that the CNM will be providing to users through a peer-reviewed proposal submission process. The CNM user program is beginning its ramp-up to full operations, and all capabilities given below will be available by the summer of 2007.

Although capabilities are grouped by theme, users should be aware that many capabilities cut across theme lines and are intended to be used across the CNM scientific portfolio. Novel applications of capabilities in ways that overlap more than one area are encouraged.

Before submitting a proposal for access, prospective users should familiarize themselves with all capabilities that are offered and communicate with one of the contact persons listed below to confirm that a capability has been brought on-line and is available for access and to identify CNM staff who can best enable their research. (Contacts are listed in alphabetical order within each area; contact details are available on the CNM or Argonne web sites.)



BioNanoComposites

Contacts: John Carlisle, Seth Darling, Millicent Firestone, Dieter Gruen, Xiao-Min Lin, Tijana Rajh, David Tiede

Synthesis of nanoparticles and bio-inorganic composites, including diamond films

- Synthesis of surface-modified metal and metal-oxide nanoparticles and quantum dots
- Peptide and molecular synthesis
- Post-self-assembly processing of soft materials
- Chemical vapor deposition of ultrananocrystalline and microcrystalline diamond

Functionalization of surfaces

- Photochemical functionalization
- Electrodeposition, electrochemical functionalization

Characterization

- Expertise in synchrotron x-ray spectroscopy and analysis, scattering, and nanoparticle diffraction
- Thermal and rheological analysis
- Scanning probe microscopy
- Electrochemical analysis
- Laser scanning confocal microscopy
- Optical spectroscopy

Electronic & Magnetic Materials & Devices

Contacts: Orlando Auciello, Samuel Bader, Kristen Buchanan, Axel Hoffmann, Stephen Streiffer

Synthesis

- Molecular beam epitaxy deposition systems for synthesis of complex oxides
- Electron-beam evaporation and sputtering systems for deposition of metal and oxide layers on up to 150-mm-diameter substrates

Characterization

- Powder and high-resolution x-ray diffractometery: lattice parameter, phase and orientation determination via high-angle diffraction, x-ray reflectivity, reciprocal space mapping.
- Scanning probe microscopy
- Electrical characterization: Impedance analysis, I-V, polarization
- 4-probe UHV STM/SEM
- SQUID magnetometry
- Magnetotransport measurements
- Raman spectrometry

Nanopatterning

Contacts: Ralu Divan, Derrick Mancini, Leonidas Ocola

Lithography

- Raith 150 30kV electron beam lithography system
- JEOL 9300F 100kV electron beam lithography system
- Focused ion beam patterning
- Karl Suss MA6 aligner optical lithography system for up to 100-mm-diameter substrates
- Oriel exposure system for up to 100-mm-diameter substrates; mask sizes: 5" and 4" square

Dry Etching

- Reactive ion etching using both fluorine and chlorine chemistries

Wet Processing

- Resist processing: spin, coating and bake
- Selective etching of metals and dielectrics
- Silicon anisotropic etching, silicon and silicon nitride membrane fabrication
- Au, Pt, Cu, and Ni electroforming

Nanophotonics

Contacts: Stephen Gray, Gary Wiederrecht

Near-field and far-field optical characterization

- Aperture and apertureless near-field scanning optical microscopy (NSOM) with CW and ultrafast laser excitation, for fluorescence, resonant scattering, Raman, nonlinear imaging over the ultraviolet, visible, and near-infrared spectral regions
- Confocal imaging and pump-probe spectroscopy
- Ultrafast transient absorption apparatus for far-field characterization of nanoparticles and nanostructures

Theory and Simulation (Virtual Fab Lab)

Contacts: Stephen Gray, Peter Zapol

Custom computational packages

- Web-based magneto-optic simulation package
- Time-domain nanophotonics simulation package
- Density-functional-based tight-binding electronic structure package (clusters, periodic structures, *ab initio* MD)
- MPI-based parallel versions of the nanophotonics and tight-binding codes

Argonne National Laboratory computational resources

- Access to 350 Xeon processor Linux cluster, "Jazz," at the Laboratory Computing Resource Center (LCRC)

X-ray Imaging/Nanoprobe

Contacts: Martin Holt, Jörg Maser, Brian Stephenson, Robert Winarski

X-ray microscopy and microspectroscopy

- Expertise in synchrotron microbeam and nanobeam techniques
- Hard x-ray nanoprobe, with 30-nm resolution at 10 keV for imaging, fluorescence, and scattering.

Electron Microscopy Center

Web site: http://emc.msd.anl.gov/
User contact: 630-252-4108

Facility Summary

The Electron Microscopy Center (EMC) is a DOE-supported research center that provides scientific researchers with forefront resources for electron beam characterization of materials. The EMC operates as part of Argonne National Laboratory within the Materials Science Division and maintains a suite of instrumentation that includes some of the world's unique electron microscopes. There is a very strong synergy between science programs that use the EMC and facility operation and development that provides an excellent model for operation of user facilities.

The mission of the Electron Microscopy Center is to enable world-class research through electron beam characterization for the local, national, and international research community. We achieve this goal by providing students, faculty, and scientists with resources and expertise to analyze structural and compositional information over critical length scales complementary to those probed by neutrons and photons. The EMC advances this mission by:

- Developing and expanding the frontiers of microanalysis through the development of new instrumentation, techniques and scientific expertise.
- Maintaining unique resources and facilities for scientific research.
- Conducting and enabling materials research using advanced characterization methods.

A nationally recognized Steering Committee counsels the EMC by providing external review and input for research proposals submitted to the Center, offering advice on future directions and fostering interactions with the scientific community. This guidance and interaction ensures that research carried out in the EMC is of the highest scientific caliber and at the forefront of the field.

Pioneering new capabilities and techniques that advance the field of electron beam characterization is a key to maintaining world-class facilities, and EMC staff vigorously pursue these new directions. A major component of these activities is the development of aberration-correcting optics, which is being carried out in the EMC through the TEAM (Transmission Electron Aberration-corrected Microscope) project. Beyond aberration-correcting optics, the EMC is developing new capabilities for dynamical studies in the electron microscope, especially imaging and quantification of internal magnetic and electric fields. Creating leading research programs and capabilities in these areas while maintaining strong efforts in existing core activities and a strong user program are essential elements of the strategic plan for the EMC.

Instrument Overview

The EMC currently operates and administers seven instruments together with support facilities that include specimen preparation, image analysis, and computational facilities. The instrumentation in the Center is organized according to specialized functions. Major areas in the EMC include the IVEM-Tandem facility, the Analytical Electron Microscopy facility, and Support facilities. An additional instrument, the Advanced Analytical Electron Microscope (AAEM), is used for developmental work. A synopsis of each of these areas is provided below.

IVEM-Tandem

This unique facility consists of an intermediate voltage TEM (IVEM), a Hitachi H-9000 NAR, that is interfaced to two ion accelerators. This combination allows in situ observation of ion beam modification and effects of irradiation. In addition, a number of holders allow a variety of in situ experiments to be performed, ranging from low temperature studies using a liquid He stage to studies at high temperatures with hot stages. The ion beam accelerators also are used independently for ion beam irradiation/implantation and analysis including research for other programs. The IVEM-Tandem serves an international user community.

Analytical Electron Microscopy (AEM)

The Analytical Electron Microscopy facility consists of electron microscopes that are optimized for high resolution and/or analytical work as well as core instrumentation that is essential for forefront materials research programs. Major capabilities include a field-emission TEM/STEM and dual scanning-electron and -ion beam (cross-beam FIB). Core capabilities include high-resolution and analytical TEMs and scanning electron microscopes that are essential to the day-to-day operations of the Center. These resources provide microstructural capabilities for a wide range of users. This part of the Center primarily serves a regional and local user community.

Support Facilities

The EMC maintains an array of specimen preparation capabilities that are available to all users. While individuals are generally expected to carry out their own specimen preparation, expertise and guidance is provided by EMC staff. In addition, the staff in the EMC have developed a number of new methods and approaches to sample preparation that are communicated to the user and scientific community.

The EMC Imaging Laboratory provides resources for image processing and analysis. The center includes computers and workstations with commercial software for image simulation, modeling, manipulation and analysis together with input and output devices for image handling.

Advanced Analytical Electron Microscope (AAEM)

The Advanced Analytical Electron Microscope is a unique instrument capable of numerous operating modes, Telepresence, CTEM, STEM, SEM, CBED, EELS, XEDS, CCD TV-rate imaging, SIMS, as well as a variety of non-conventional operating modes (LSTEM, PRD, SCEM). This instrument serves as a test bed for pioneering new concepts as well as the evolution of innovative technologies and methods for materials characterization.

User Access

All research access to the EMC is through proposal submission. All proposals are reviewed with the goal of ensuring that the best scientific outcomes are realized. All proposals undergo a technical review by EMC staff. This review evaluates technical feasibility of the proposed work. Major proposals to the EMC are reviewed by the Steering Committee. The reviewers are asked to evaluate the scientific and technical merit of the proposal, whether the proposed approach is appropriate, the probability of success (based on the resources and competency of the investigators), and the appropriateness of EMC resources requested. The reviewers provide an overall evaluation of the proposal and a rating for priority.

The type of access granted to a user is based on the nature of the proposed work, their qualifications, and the instrumentation requested. Research projects may be allocated a specific amount of instrument time (allocated access) or may be granted continuous access. Under certain circumstances, rapid access may be awarded. All access is regulated by a *scheduling policy* that works well to provide fair access to instruments yet also meets the occasional need for enhanced access.

Under *allocated access*, a user is allocated a specific amount of instrument time to complete the proposed project. Allocated access is used primarily to manage instrument time for those instruments that have very high demand and for projects that require exceptional staff assistance.

Continuous access allows a user to use specified EMC instrumentation without preset limits on the amount of instrument time. Continuous access is intended for those users for whom Argonne is their primary research home or who otherwise demonstrate a consistent need for access to EMC instruments. Continuous access proposals terminate at the end of each fiscal year and must be renewed annually. This type of access is a key feature of facilities like the EMC based on the nature of research conducted.

Recognizing the occasional urgent need, a proposal may be granted *rapid access* at the discretion of the EMC Director. Rapid access is intended to facilitate discovery and dissemination of significant, new scientific results and may be granted if an exceptional case is presented. Users are expected to complete a proposal and user agreement as soon as possible following experiments carried out under this access path.

Scheduling

The majority of instrument time is scheduled directly by users, moderated by a self-regulating reservation policy. Under this policy, a user may have one "prime-time" session (per instrument) reserved at any given time. Research projects of users that require staff assistance, including training sessions, are scheduled by the Instrument Scientist at a mutually agreed upon time.

More Information

Prospective users can find more information about the facility, its instrumentation and capabilities, and the proposal submission process at the EMC's web site.

Nominal Performance Specifications and Characteristics of EMC Instruments

Instrument	Operating Modes	Specimen Holders	Resolution
IVEM-Tandem Hitachi H-9000 TEM coupled to ion accelerators 100 – 300 kV LaB_6	CTEM, CBED, SAED, TV-rate video, digital image capture, light element EDXS, *in situ* ion irradiation with dosimetry (any allowed ion up to 600 kV and 2 MV He^{+1} through 0.4 MV Kr^{+1}).	*Double Tilt (±45° α, ±30° β):* Be cup for XEDS, liquid He cooled, heating (1200 K) *Single Tilt (±45°):* tensile/heating (600 K), electrical bias/heating (800 K), gas reaction cell.	~0.25 nm point ~0.14 nm lattice
Tecnai F20ST 80 – 200 kV Schottky FEG FEI	CTEM, STEM (BF/ADF, HAADF), CBED, SAED, CCD camera (16 Mpixel), light element EDXS, PEELS, spectrum imaging, energy-filtered imaging, Lorentz magnetic imaging, electron holographic imaging.	*Double Tilt (±40° α, ±30° β):* Be cup for EDXS; liquid N_2 cooling (93 K) with Be cup; heating (1270 K). *Single Tilt (±40° α):* magnetic field holder. *Tilt/rotate (±40° α, 360°):* liquid He cooling.	~0.24 nm point ~0.1 nm lattice Cs obj. ≈ 1.2 mm Cc obj. ≈ 1.2 mm probe ≈ 0.2–1 nm
JEM-4000EXII 100 – 400 kV LaB_6 JEOL	HREM, CTEM, SAED, CBED, TV-rate video, CCD camera (1 Mpixel), electron dosimetry, fluctuation.	*3 Top-Entry Double Tilt (±20°)* *2 Top-Entry Zero Tilt*	~0.165 nm point ~0.1 nm lattice
CM30T ≤50 – 300 kV LaB_6 Philips (FEI)	CTEM, CBED, SAED, fast CCD camera (1 Mpixel, 15 fps), hollow-cone DF, light element EDXS, PEELS, electron dosimetry.	*Double Tilt (±60° α, ±30° β):* Be cup for EDXS; cooling (93 K) with Be cup; heating (1270 K).	~0.25 nm point ~0.14 nm lattice probe size ≥ 9 nm
S-4700-II 0.5 – 30 kV Cold FEG Hitachi	SEI, BEI, light element EDXS (mapping & spectrum imaging).	5-axis motorized stage. Maximum sample size: 27 mm (H) x 150 mm (dia.).	SEI resolution 1.5 nm at 15 kV 2.5 nm at 1.0 kV
1540XB FIB 0.2 – 30 kV Schottky FEG Zeiss	FIB-SEM with 5 gas injectors & 2 *in situ* manipulators, SEI, BEI, STEM, EBSD/OIM, light element EDXS (mapping & spectrum imaging).	6-axis motorized eucentric stage. Maximum sample size: 100 mm diameter.	SEI resolutions: 1.1 nm at 20 kV 2.5 nm at 1.0 kV
Quanta 400F ESEM 0.2 – 30 kV Schottky FEG FEI	SEI, BEI, specimen chamber pressures ≤ 3000 Pa (air & water vapor standard).	5-axis motorized stage. Maximum sample size: 100 x 100 mm. Heating stages (≤ 1770 K). Peltier-cooled stage (248 K to 328 K).	SEI resolution at 30 kV ≤ 3 nm at 1330 Pa ≤ 10 nm at 2660 Pa

Intense Pulsed Neutron Source

Web site: http://www.pns.anl.gov/
User contact: 630-252-6485

Mission Statement

The Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory is a National User Facility for performing neutron scattering experiments to determine the properties of materials by studying atomic arrangements and motions in liquids and solids. IPNS provides qualified users with reliable optimized neutron scattering instruments; provides users the assistance of experienced scientific and technical staff; and ensures the safe and timely completion of users' experiments.

Source Information

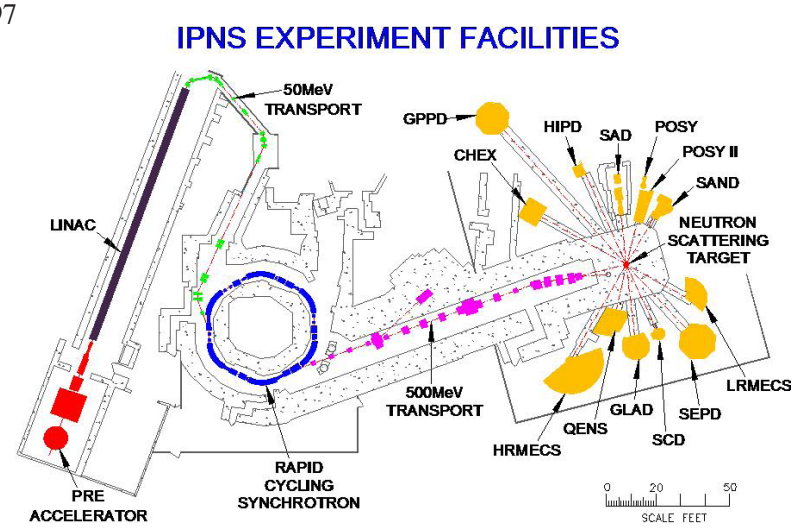
IPNS is a 30 Hz short-pulsed spallation neutron source using protons from a linac/rapid cycling synchrotron to produce neutrons in a uranium target. The target is depleted uranium, which produces a factor of two more neutrons than non-fissionable targets such as tungsten or tantalum. Two cryogenic moderators (decoupled solid CH_4 and decoupled poisoned liquid CH_4) produce short pulses of slow neutrons while one (coupled, solid CH_4) produces longer pulses of slow neutrons. Although the proton beam power is only 7 kW, the combination of a uranium target (factor of 2) and solid CH_4 moderators (x3.6) yield an equivalent power for cold neutrons of 50 kW when compared to a non-fissionable target with liquid H_2 moderators. Twelve beam lines currently serve 14 instruments, one of which is a test station for instrument development. All instruments have independent computer systems with standardized control programs. Analysis software is typically personal computer–based, and computers are available for users to use while they are at IPNS.

Staffing and Users

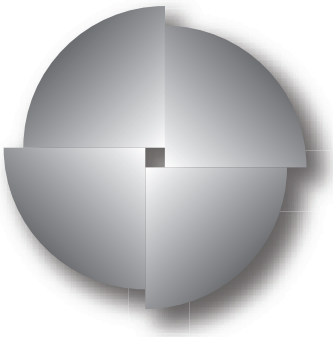
Currently the IPNS Division includes 87 technical, scientific, and administrative personnel. The IPNS workforce is relatively stable with a low rate of turnover. In FY05, 397 (244 "traditional" and 153 "remote") users performed 430 experiments at IPNS, resulting in 125 publications. These users include scientists from universities (67%), government (18%), and foreign institutions (15%).

Instrumentation

IPNS operates 13 instruments at 12 beamports (see figure). Instruments available to users include three inelastic scattering (HRMECS, LRMECS, QENS), five wide angle diffractometers (GPPD, HIPD, SEPD, SCD and GLAD) for powder and single crystal samples, two small angle scattering instruments (SASI and SAND) and two reflectometers (POSY I and II). Two beamports are currently used for instrument and/or device test work.



Inelastic Scattering	Large Scale Structures	Diffraction
Direct geometry: HRMECS, LRMECS Inverse geometry: QENS, CHEX	Small-angle scattering: SAND, SASI (formerly SAD)	Powder diffraction: SEPD, GPPD, HIPD Single crystal: SCD Liquids and amorphous materials: GLAD Reflectometry: POSY I, POSY II



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Comprehensive Program

Monday, May 1
CNM Plenary Session

Exhibit Hours: 8:00 am – 5:00 pm
Bldg. 402, Gallery and Bldg. 401, Atrium

Agenda subject to change

Introductory Session—Chair: Eric Isaacs, Center for Nanoscale Materials

8:30 – 8:40 Welcome from Argonne
Robert Rosner, Director, Argonne National Laboratory

8:40 – 9:10 View from the Hill
Speaker TBD

9:10 – 9:35 DOE Perspective
Tof Carim, Office of Science, U.S. Department of Energy

9:35 – 10:05 CNM Scientific and User Programs
Eric Isaacs, Director, Center for Nanoscale Materials

10:05 – 10:30 Break
Atrium and Gallery

10:30 – 11:00 CNM Facility Update
Derrick Mancini, Project Manager, Center for Nanoscale Materials

11:00 – 11:30 User Programs and Tools
Stephen Streiffer, Center for Nanoscale Materials

Nanoscience I—Chair, Gayle Woloschak, Northwestern University

11:30 – 12:00 Biomolecular Directed Assembly
Chad Mirkin, Northwestern University

12:00 – 1:30 Lunch with Theme Leaders
*Bldg. 402, Lower Level, Tent**

1:30 – 2:00 Biomimetics (tentative title)
Joanna Aizenberg, Bell Labs, Lucent Technologies (tentative)

2:00 – 2:30 Harnessing Self-Assembly for Hierarchical Fabrication of Hybrid Nanostructures
Seth Darling, Center for Nanoscale Materials

2:30 – 3:00 Break
Atrium and Gallery

*Look for tables with placards identifying themes.

Nanoscience II—Chair, Paul Evans, University of Wisconsin-Madison

3:00 – 3:30 Defects as Nanodevices
Gabriel Aeppli, University College London

3:30 – 4:00 Imaging Non-Collinear Spin Structures on the Atomic Scale
Matthias Bode, Institute of Applied Physics, University of Hamburg

4:00 – 4:30 Monodispersed Inorganic Colloidal Nanoparticles: Functional Building Blocks of Future Matierals
Phillipe Guyot-Sionnest, The University of Chicago

4:30 Adjourn

Poster Session & Banquet

5:00 – 7:00 CNM Poster Session and Opening Reception
*Bldg. 440 (CNM Building)**

7:30 – ?? CNM Banquet
Argonne Guest House

*The atrium of Building 440 may be entered from the parking lot off Kearney Road or from the APS experiment hall at sector 26 (follow posted signs to the atrium). **Be aware that portions of Building 440 may have work-in-progress areas. Do not enter any areas posted as such; obey all signs.**

Tuesday, May 2
Nanoscience Workshops

Exhibit Hours: 8:00 am – 5:00 pm
Bldg. 402, Gallery and Bldg. 401, Atrium

9:00 – 12:15 ***Workshop 1–Part 1***
Toward 1-nanometer X-ray Beams (p. 31)
Bldg. 402, Lecture Hall

8:30 – 12:30 ***Workshop 2***
Nanomaterials for Energy (p. 37)
Bldg. 402, Room E1100/E1200

8:30 – 12:00 ***Workshop 3***
Nanophotonics (p. 39)
Bldg. 401, Room A5000

8:30 – 12:10 ***Workshop 4–Part 1***
Microscopy and Imaging in Materials Science (p. 43)
Bldg. 401, Room A1100

12:00 – 1:30 Lunch
Bldg. 402, Lower Level, Tent

1:30 – 4:30 ***Workshop 1–Part 2***
Toward 1-nanometer X-ray Beams (p. 31)
Bldg. 402, Lecture Hall

1:30 – 4:20 ***Workshop 4–Part 2***
Microscopy and Imaging in Materials Science (p. 43)
Bldg. 401, Room A1100

1:30 – 5:30 ***Workshop 5***
Nanopatterning (p. 51)
Bldg. 402, Room E1100/E1200

1:30 – 5:30 ***Workshop 6***
Quantum Nanomagnetism (p. 53)
Bldg. 401, Room A5000

4:30 – 6:30 Poster Session & Reception:
Electron Microscopy Center and Intense Pulsed Neutron Source
*Bldg. 440 (CNM Building)**

6:30 – 8:30 APS Partner User Council
Location TBD

*The atrium of Building 440 may be entered from the parking lot off Kearney Road or from the APS experiment hall at sector 26 (follow posted signs to the atrium). **Be aware that portions of Building 440 may have work-in-progress areas. Do not enter any areas posted as such; obey all signs.**

Wednesday, May 3
APS Plenary Session

Exhibit Hours: 8:00 am – 5:00 pm
Bldg. 402, Gallery and Bldg. 401, Atrium

9:00 – 12:00 ***Opening Session and Science Session I***
Bldg. 402, Lecture Hall

9:00 – 9:05 Welcome
Carol Thompson, Chair, APS Users Organization

9:05 – 9:10 Welcome
Robert Rosner, Director, Argonne National Laboratory

9:10 – 9:45 View from the Hill
Michael Holland, U.S. House of Representatives Committee on Science (tentative)

9:45 – 10:30 Advanced Photon Source Update
J. Murray Gibson, Director, Advanced Photon Source

11:00 – 11:30 Addressing Energy Grand Challenges through Advanced Materials
Michelle Buchanan, Oak Ridge National Laboratory

11:30 – 12:00 Complexity and Length Scales in Biology
Dean A. A. Myles, Oak Ridge National Laboratory

12:00 – 1:30 Lunch, Tent
Bldg. 402, Lower Level, Tent

1:30 – 4:30 ***Science Session II***
Bldg. 402, Lecture Hall

1:30 – 1:35 Introduction of the Franklin Award Winner

1:35 – 2:00 Studying Novel High Pressure Phenomena Using Integrated Synchrotron Techniques
Wendy L, Mao, Franklin Award winner, Los Alamos National Laboratory

2:00 – 2:30 Using X-ray Speckle to Test Dynamical Scaling
Mark Sutton, McGill University

2:30 –3:00 Synchrotron-Based Measurements of Magnetically Doped Transition Metal Oxides
Scott Chambers, Pacific Northwest National Laboratory

3:00 – 3:20 Break
Atrium and Gallery

3:20 – 3:50 From X-rays to Biogeochemistry to Beethoven
Ken Kemner, Argonne National Laboratory

3:50 – 4:20 Biological Methane Oxidation
Amy C. Rosenzweig, Northwestern University

4:20 – 4:30 Announcement of new APSUO Steering Committee members

4:30 Adjourn

Poster Session & Banquet

4:30 – 6:30 Poster Session & Reception: Advanced Photon Source
*Bldg. 440 (CNM Building)**

6:45 – ?? User Banquet
Argonne Guest House

*The atrium of Building 440 may be entered from the parking lot off Kearney Road or from the APS experiment hall at sector 26 (follow posted signs to the atrium). **Be aware that portions of Building 440 may have work-in-progress areas. Do not enter any areas posted as such; obey all signs.**

Thursday, May 4
Workshops

Exhibit Hours: 8:00 am – 12:00 pm
Bldg. 402, Gallery and Bldg. 401, Atrium

8:30 – 12:00 ***Workshop 7–Part 1***
Texture and Strain Mapping with X-rays, Neutrons, and Electrons (p. 57)
Bldg. 401, Room A1100

8:30 – 12:20 ***Workshop 8–Part 1***
Inelastic X-ray Scattering: Present and Future at the APS (p. 63)
Bldg. 402, Room E1100/E1200

8:30 – 12:10 ***Workshop 9–Part 1***
X-ray Spectromicroscopy: A Tool for Environmental Science? (p. 71)
Bldg. 402, Lecture Hall

8:30 – 12:00 ***Workshop 10***
Diffuse Scattering: Emerging Opportunities with Advanced X-ray and Neutron Sources (p. 81)
Bldg. 401, Room A5000

12:00 – 1:30 Lunch
Bldg. 402, Lower Level, Tent

12:15 – 1:30 APSUO Steering Committee Meeting
Bldg. 401, Fifth Floor Gallery

1:30 – 5:00 ***Workshop 7–Part 2***
Texture and Strain Mapping with X-rays, Neutrons, and Electrons (p. 57)
Bldg. 401, Room A1100

1:15 – 5:00 ***Workshop 8–Part 2***
Inelastic X-ray Scattering: Present and Future at the APS (p. 63)
Bldg. 402, Room E1100/E1200

1:30 – 6:00 ***Workshop 9–Part 2***
X-ray Spectromicroscopy: A Tool for Environmental Science? (p. 71)
Bldg. 402, Lecture Hall

1:30 – 5:30 ***Workshop 11***
Beam Line Controls at the APS (p. 85)
Bldg. 401, Room A5000

5:00 – 6:30 Tenth Anniversary of APS Operations Celebration—Symposium
Argonne Guest House, Conference Room A

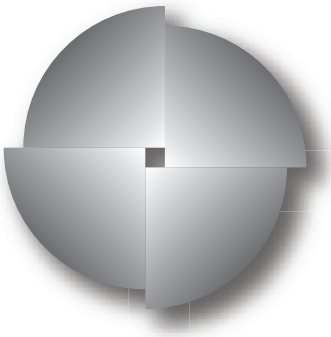
7:00 – 9:30 Tenth Anniversary of APS Operations Celebration—Dinner
Argonne Guest House

Friday, May 5
Workshop

9:00 – 11:45 ***Workshop 12–Part 1***
Microdiffraction in Structural Biology (p. 87)
Bldg. 402, Lecture Hall

11:45 – 1:30 Lunch
Bldg. 402, Lower Level, Tent

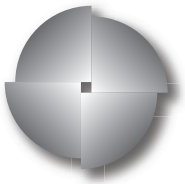
1:30 – 4:15 ***Workshop 12–Part 2***
Microdiffraction in Structural Biology (p. 87)
Bldg. 402, Lecture Hall



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Plenary Talk Abstracts



Center for Nanoscale Materials Plenary Session

Imaging Non-Collinear Spin Structures on the Atomic Scale

M. Bode
Institute of Applied Physics, University of Hamburg, 20355 Hamburg, Germany

The experimental progress in spin-polarized scanning tunneling microscopy (SP-STM)—a magnetically sensitive imaging technique with ultra-high resolution—allows the investigation of surfaces, thin films, and epitaxial nanostructures with unforeseen precision [1]. Together with its high surface sensitivity, the atomic-resolution capability of SP-STM makes it particularly suited for the investigation of antiferromagnetic and superparamagnetic surfaces which could only be studied in some rare cases with moderate spatial resolution in the past because of the lack of macroscopic magnetization. By choosing appropriate substrates and growth conditions Fe nanostructures with a wide range of magnetic properties, including of ferro-, antiferro-, and superparamagnets, can be prepared. For example, we have recently shown by spin-polarized scanning tunneling microscopy that the Fe monolayer on W(001) isc(2×2) antiferromagnetic, i.e., it exhibits a checkerboard pattern of antiparallel magnetic moments. As can be concluded from measurements in an external magnetic field, the easy magnetization axis is out-of-plane [2]. On this antiferromagnetic Fe layer we have occasionally observed phase domain walls which are typically very short and clamped between defects like adsorbates or islands. Our results demonstrate that atomic resolution SP-STM can also be applied to non-periodic and non-collinear spin structures. The domain wall width amounts to 6-8 atomic rows only and the walls are centered between two atomic rows. The results are compared to Monte-Carlo simulations. While walls oriented along (100) directions are found to be fully compensated, the detailed analysis of (110) walls reveals an uncompensated perpendicular magnetic moment. This may be of technological importance as uncompensated moments are responsible for the exchange-bias effect which is widely used in state-of-the-art magnetic storage devices.

[1] M. Bode, *Rep. Prog. Phys.* 66, 523 (2003).

[2] A. Kubetzka et al., *Phys. Rev. Lett.* 94, 087204 (2005).

Advanced Photon Source Plenary Session

Addressing Energy Grand Challenges through Advanced Materials

Michelle V. Buchanan
Associate Laboratory Director, Physical Sciences, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

The world's demand for energy is projected to double by 2050, requiring tremendous growth in energy generation capacity. This demand will require a more diverse portfolio of energy sources than our current reliance on fossil-based fuels, especially when one takes into account the limited supply of readily available sources and environmental concerns. To meet future energy requirements, basic research in materials science is needed to provide revolutionary new capabilities in the overall energy economy, including production, storage, and use. All the elementary steps of energy conversion—charge transfer, molecular rearrangement, chemical reactions—take place on the nanoscale. Thus the development of new nanoscale materials, as well as the analytical tools for characterizing and computational tools for modeling and predicting these materials, has the potential to create an entirely new paradigm for developing new and revolutionary energy technologies.

Complexity and Length Scales in Biology

D.A.A. Myles
Center for Structural Molecular Biology, ORNL, Oak Ridge, TN 37831, USA

X-ray scattering and diffraction provide information on biological processes on time and length scales that span from the atomic to the cellular levels of detail. These insights are often profound, extending from the location of the individual atoms in protein structures that precisely define chemical specificity and selectively, to the resolution of the structures of the large and complex machines of the cell. Fast x-ray diffraction and spectroscopy extend this view to the dynamic world, capturing precise snapshots of molecular processes in motion. With more than 30,000 proteins structures now known, and new structures being added almost daily, major new challenges involve understanding how biomolecular form, function, and interactions are coordinated in solution, at membrane surfaces, and in the crowded environment of the cell. Integrated approaches that combine multiple levels of structural, biochemical and computational information are beginning to meet these challenges.

Using X-ray Speckle to Test Dynamical Scaling

Mark Sutton
McGill University, Montreal, Quebec, H3A 2T8, Canada

Many of the properties of a material depend more on its microstructure than its atomic structure. Intensity fluctuation spectroscopy (IFS) is an ideal way to study fluctuations of this structure. For the last three decades or so, it has been extensively used with light scattering to study a large variety of transparent systems. Extending IFS to x-rays allows us to study opaque materials and to probe much shorter length scales, as required for example by binary alloys. Furthermore, although IFS is extremely successful for equilibrium systems, this method is not often used to study fluctuations in nonequilibrium systems. In this talk, I will discuss the success we have obtained by using IFS to study nonequilibrium fluctuations in binary alloys. In particular, I will discuss how the measurement of two-time correlation functions after a quench through a first-order phase transition presents a new test of the general concept of dynamical scaling.

Synchrotron-Based Measurements of Magnetically Doped Transition Metal Oxides

Scott A. Chambers
Fundamental Science Directorate, Pacific Northwest National Laboratory, Richland, WA 99352, USA

Synchrotron-based spectroscopic measurements have been invaluable in determining the detailed properties of transition-metal-doped oxides, which are of potential interest in semiconductor spintronics. The atom-specific nature of XANES and EXAFS allow the photoemission investigator to probe the local charge and structure of the magnetic dopant with excellent sensitivity at third-generation light sources. XES, in combination with XANES and UV, reveals useful information on dopant hybridization with the host lattice. XMCD probes the dopant empty states to sense exchange splitting. In combination with more conventional experiments, such as TEM, XRD, and ion channeling, synchrotron-based spectroscopies reveal structure-magnetic function relationships, which in turn allow the mechanism(s) of magnetism to be elucidated. In this talk I will illustrate what we have learned about diluted magnetic oxide semiconductors at third-generation storage rings, drawing from our experience on Co- and Cr-doped TiO_2 anatase. I will also discuss lessons learned and pitfalls to be avoided in the high-T_c DMS field to date and look forward to what might be the most fruitful directions for future research.

From X-rays to Biogeochemistry to Beethoven

Ken Kemner
Argonne National Laboratory, Argonne, IL 60439, USA

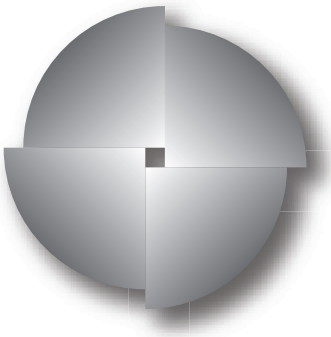
Understanding the fate and transport of heavy metal and radionuclide contaminants in soils, sediments, and groundwater; through the food chain; and in human physiological responses to contaminant exposure is fundamentally important in the development and evaluation of risk assessment and effective remediation and sequestration strategies. In addition to the physical and chemical processes at the mineral surface, bacteria and the extracellular material associated with them are thought to play a key role in determining a contaminant's speciation and thus its mobility in the environment. Increasing our understanding of the role of bacteria in determining the fate of heavy metals and radionuclides in the environment requires the integration of many different scientific disciplines, including physics, geology, chemistry, and (micro)biology. Similarly, an increased understanding of the movement of metals and radionuclides through the human body upon exposure to these contaminants is needed to better understand their toxicity. This presentation will provide a general overview of the field of environmental science and geomicrobiology, present forensic studies of hair and bone relics used to identify causes of Ludwig van Beethoven's documented maladies and death, illustrate the importance of integrating multiple scientific disciplines to address key questions in these fields, and demonstrate how novel uses of synchrotron-based x-ray absorption spectroscopy and x-ray microscopy, coupled with electron microscopy, have provided key insights to some of these questions.

This work was supported by the U.S. DOE Office of Science (OS), Office of Biological and Environmental Research, Environmental Remediation Science Program, and Argonne National Laboratory LDRD funds. The Advanced Photon Source is supported by the U.S. DOE OS, Office of Basic Energy Sciences. Work at MR-CAT is also supported by the member institutions. Additional support was provided by the Presidential Early Career Scientist and Engineer Award.

Biological Methane Oxidation

Amy C. Rosenzweig
Departments of Biochemistry, Molecular Biology, and Cell Biology and of Chemistry, Northwestern University, Evanston, IL 60208, USA

Methanotrophic bacteria are a unique family of gram negative eubacteria that utilize methane as their sole source of carbon and energy. The first step in their metabolic pathway is the oxidation of methane to methanol by methane monooxygenase (MMO) enzyme systems. There are two types of MMO systems, a soluble cytoplasmic complex (sMMO) and a membrane-bound particulate system (pMMO). All methanotrophs produce pMMO, and several strains also produce sMMO under copper-limiting conditions. The crystal structure of the sMMO hydroxylase, which contains a carboxylate-bridged diiron center, has been known for more than a decade. By contrast, most questions surrounding the biochemistry, structure, and mechanism of the predominant methane oxidation enzyme, pMMO, have remained unanswered despite considerable research efforts in the last 20 years. In particular, the pMMO metal ion composition and stoichiometry have been controversial. We have determined the crystal structure of pMMO from the methanotroph *Methylococcus capsulatus* (Bath) to 2.8 Å resolution. The enzyme is a 300 kDa trimer, comprising three copies each of the pmoB, pmoA, and pmoC subunits. Two metal centers, modeled as mononuclear copper and dinuclear copper, are located in the soluble regions of each pmoB subunit, which resembles cytochrome *c* oxidase subunit II. A third metal center, occupied by zinc in the crystal, is located within the membrane. The crystallographic model for the metal centers is supported by spectroscopic data obtained for purified pMMO.



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Workshops Agendas and Agendas

Workshop 1

Toward 1-nanometer X-ray Beams

Organizers: Paul Evans, University of Wisconsin-Madison
Jörg Maser, Argonne National Laboratory

Tuesday, May 2
Bldg. 402, Lecture Hall

9:00 am – 12:15 pm
1:30 pm – ~4:30 pm

This workshop features discussions of the ultimate spatial resolutions that can be reached by x-ray focusing, as well as presentations of the state of the art and potential intrinsic limits of optical fabrication technologies. An informal session following the invited talks will allow attendees to provide thoughts and feedback and present directions for the rapidly evolving field of focused x-ray beams. (A more detailed description of the workshop theme is given after the agenda.)

9:00 – 9:10	Welcome *Organizers*
9:10 – 9:30	The Scientific Case for Nanometer X-ray Beams *Eric Isaacs, Center for Nanoscale Materials, Argonne National Laboratory*
9:30 – 10:00	Nanometer X-ray Focusing Using Diffractive Optics: Where is the Limit? *Jörg Maser, Center for Nanoscale Materials, Argonne National Laboratory*
10:00 – 10:30	Fundamental Limitations of Focusing Hard X-rays with Refractive Optics *Christian Schroer, Institute of Structural Physics, Technische Universität Dresden, Germany*
10:30 – 10:45	Break, Atrium and Gallery
10:45 – 11:15	Challenges and Opportunities for Achromatic Hard X-ray Focusing *Gene Ice, Oak Ridge National Laboratory*
11:15 – 11:45	Sub-10 nm Hard X-ray Focusing by KB Mirrors *Kazuto Yamauchi, Osaka University*
11:45 – 12:15	Nanofocusing with Reflective X-ray Optics *Christian Morawe, European Synchrotron Radiation Facility*
12:15 – 1:30	Lunch, Bldg. 402, Lower Level, Tent
1:30 – 2:00	Fabrication of Optics for Nanofocusing of Hard X-rays *Albert Macrander, Advanced Photon Source, Argonne National Laboratory*
2:00 – 2:30	Sub-10 nm X-ray Microscopy: Status and Pathways *Wenbing Yun, Xradia, Inc.*
2:30 – 3:00	Fabrication Techniques for High-Resolution Zone Plates: Future Directions and Limitations *J. Alex Liddle, Molecular Foundry, Lawrence Berkeley National Laboratory*
3:00 – 3:30	Break, Atrium and Gallery
3:30 – open	Discussion

Detailed Workshop Statement

Since the nature of x-rays was first understood more than one hundred years ago, it has been realized that they would be an ideal tool for microscopy. The availability of x-ray optics that provide the ultimate spatial resolution would open up new frontiers in x-ray studies from nanoscale imaging of objects buried deep inside a specimen to coherent manipulation of nanoparticles. Spatial resolutions of x-ray probes could in principle approach the wavelength of hard x-rays (well below 1 nm) and provide information based on atomic and electronic structure, chemical composition, and magnetism. Such unique capabilities would impact many disciplines across the biological and physical sciences.

The resolving power for any optical element is limited simply by its numerical aperture and the wavelength of the radiation. Current hard x-ray optics have very small numerical apertures (<0.001), and their resolving power is far from the ultimate limit. To achieve large numerical apertures fundamentally requires deflecting x-rays through large angles. Since hard x-rays with wavelengths of order 1 Å interact only weakly with matter and are not affected by electric or magnetic fields it has been a challenge to produce optics that produce sufficiently large deflections.

The three basic approaches to achieving x-ray optics with high numerical apertures have been refraction, reflection, and diffraction. These approaches are complementary in their capabilities. While it is not yet clear which approach will produce the ultimate resolution, they share many of the same challenges for fabrication and at the smallest spot sizes, the already artificial distinctions between these techniques are blurred. It is even possible that the ultimate optics could employ compound optics incorporating combinations of the three approaches.

Today, the highest resolutions are close to 10 nm in the soft x-ray range using Fresnel zone plate optics and better than 50 nm in the hard x-ray range using approaches based both on reflective and on diffractive optics. With recent advances in state-of-the-art fabrication techniques, hard x-ray optics with a focal spot below 10 nm seem feasible, and a push toward exploring true nanometer focusing is already beginning. As with all other microscopy techniques, including electron and optical microscopes, x-ray microscopes require highly brilliant sources in addition to large numerical aperture optics. Bright sources of hard x-rays are already available at third-generation light sources including the Advanced Photon Source the upcoming NSLS II and are planned for electron lasers such as the DOE's Linac Coherent Light Source.

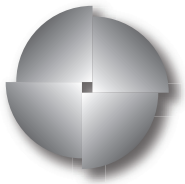
It is now possible to articulate a vision for what the ultimate x-ray focusing optics should look like. This vision will most likely involve several types of optical devices. For nanocrystalline materials structural studies may require multi-wavelength or "white" beams of x-rays and hence mirror optics. Elemental mapping, for example of trace metals in biological cells, will require single-wavelength excitation at maximum resolution and hence diffractive optics. High-resolution diffraction and coherent imaging require beams with well-defined incident wave fronts, but could in principle make use of any one of several focusing techniques.

WK1

Nanometer X-ray Focusing Using Diffractive Optics: Where is the Limit?

Jörg Maser, Stefan Vogt, Hyon Chol Kang, Brian Stephenson, Al Macrander, Chian Liu, and Ray Conley
Argonne National Laboratory, Argonne, IL 60439, USA

The ultimate spatial resolution limit of x-ray optics has been the topic of considerable debate over the last several years [1,2]. While the spatial limitation of far-field optics is given by the wavelength, below 0.1 nm for hard x-rays, the weak interaction of x-rays with matter and the resulting long interaction lengths raise the question whether there are intrinsic limitations to x-ray optics. The thrust of this presentation will be a discussion of the diffraction properties of high-resolution diffractive optics and their spatial resolution limit.



The focusing properties of high-resolution diffractive optics are governed by volume diffraction, which has aspects of thin-grating behavior in the center of the optic and crystal-like diffractive behavior in outermost areas of the optic. Satisfying the Bragg condition becomes critical to both maintaining good efficiency and minimizing wavefront aberrations, both of which lead to a reduction of spatial resolution. We will discuss an ideal structure capable of achieving a spatial resolution of 1 nm or below.

We will also present a new type of x-ray optic we have developed, the multilayer Laue lens, aimed at achieving true nanometer focusing [3]. These optics have achieved a spatial resolution of 30 nm at a photon energy of 19.5 keV, with a diffraction efficiency of 44%. We will show measurements of volume diffraction effects and more recent spatial resolution measurements and will discuss a path towards nanometer focusing.

[1] C. G. Schroer et al., *Appl. **Phys. Lett.*** **87**, 124103 (2005).

[2] C. Bergemann, H. Keymeulen, and J. F. Van der Veen, *Phys. **Rev. Lett.*** **91**, 204801 (2003).

[3] H. C. Kang et al., *Phys. Rev. Lett.* **96**, 127401 (2006).

WK1
Fundamental Limitations of Focusing Hard X-rays with Refractive Optics

Christian Schroer
Institute of Structural Physics, Technische Universtät Dresden, D-01062 Dresden, Germany

The question is addressed of what is the smallest spot size that hard x rays can be focused to using refractive optics. This question is linked to the limitation of the refractive power per unit length of the optic along the optical axis at a given aperture. A thick refractive x-ray lens is considered, whose aperture is gradually (adiabatically) adapted to the size of the beam as it converges to the focus. These adiabatically focusing lenses (AFLs) are shown to have a relatively large numerical aperture, focusing hard x rays down to a lateral size of 2 nm (FWHM). Although there is not a sharp fundamental limit for the focal size, atomic resolution seems to be out of range for these optics.

WK1
Challenges and Opportunities for Achromatic Hard X-ray Focusing

Gene E. Ice
Oak Ridge National Laboratory, Oak Ridge National Laboratory, Oak Ridge TN 37831-6118

Achromatic hard x-ray focusing is essential for a wide range of experiments including polychromatic micro/nano-diffraction, wide-bandpass fluorescence microscopy, scanning microbeam spectroscopies, and edge contrast projection imaging. Although x-rays can be focused in various ways, Kirkpatrick-Baez x-ray optics are currently the leading method for achromatic focusing of x-rays above 5 keV. Like all focusing optics, KB mirrors are ultimately limited by diffraction, but only recently has this limit been approached with ultra-accurate elliptical surfaces. A collaboration between scientists at ORNL and the APS has recently demonstrated achromatic x-ray optics capable of focusing to better than 70 nm on a type A undulator at the APS. These optics have been used to do first experiments including projection microscopy and measurements of deformation in nanograins near precipitates. Researchers at SPring-8 and the ESRF have similarly demonstrated achromatic focusing optics in the 30-40 nm regime, and scientists around the world are racing to produce 10 nm and smaller beams. To achieve beams below 30 nm, new strategies will be required to circumvent the diffraction limit imposed by the current generation of KB optics. Some strategies that appear possible within the next decade include the use of multilayer optics to improve the diffraction limit and/or the use of multiple mirrors in each plane. These strategies can provide for much smaller beams, but with experimental compromises. The technical challenges and scientific prospects for various approaches will be discussed.

Work sponsored by the Department of Energy, Office of Basic Energy Science, through the Oak Ridge National Laboratory (ORNL). ORNL is operated by UT-Battelle, LLC, for the U.S. Department of Energy under contract DE-AC05-00OR22725. Experiments performed on beamline 34-ID at the APS, both of which are also funded through the Department of Energy, Office of Basic Energy Science.

WK1

Sub-10 nm Hard X-ray Focusing by KB Mirrors

K. Yamauchi[1], H. Mimura[1], H. Yumoto[1], S. Matsuyama[1], Soichiro Handa[1], K. Yamamura[2], Y. Sano[1], Y. Mori[2] Y. Nishino[3], K. Tamasaku[3], M. Yabashi[4], and T. Ishikawa[3,4]

[1] *Department of Precision Science and Technology, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan*

[2] *Research Center for Ultra-Precision Science and Technology, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan*

[3] *SPring-8/RIKEN, 1-1-1 Kouto, Mikazuki, Hyogo 679-5148, Japan.*

[4] *SPring-8/Japan Synchrotron Radiation Research Institute (JASRI), 1-1-1 Kouto, Mikazuki, Hyogo 679-5148, Japan*

We have developed an ultraprecise figuring system with accuracy close to 0.1 nm (RMS) over a large area of 100 mm-scale and applied it for fabrication of high performance total-reflection mirror optics to realize chromatic-aberration-free hard x-ray nanofocusing. Performance of the fabricated mirrors was demonstrated at the 1-km-long beamline (BL29-XUL) of SPring-8, and 36 nm x 48 nm focusing and 30 nm line-focusing under nearly diffraction-limited conditions were achieved at an x-ray energy of 15 keV. We briefly summarize the fabrication process of the mirrors [1]-[7].

Then, I propose the next technologies to be realized for 10 nm hard x-ray focusing by KB mirrors. Multilayer film coating is said to be necessary technology to realize large NA mirrors, which enable a small focal spot and large photon flux. From the wave-optical estimations of required figure accuracy, I can say that accuracy higher than 1 nm (P-V) is needed for 10-nm focusing. Imperfection of the multilayer film thickness also becomes the origin of the phase error of the reflected beam, which means the same as figure error of the mirror surface. To realize such next generation x-ray mirrors, at-wavelength measurement of the wavefront shape of the reflected beam will become an indispensable technology. On the other hand, methods to compensate for such phase error by additional processes, such as additional deposition or adaptive phase control technology, will also become important. For the at-wavelength measurement, a phase retrieval method using the observed intensity distribution at the beam waist is already confirmed to be effective. To compensate the wavefront shape of the reflected beam after the measurement, we found that the additional deposition of only 5 or 10 bi-layers with the spacer layer having suitable thickness distribution can shift the phase of the reflected beam in the range of μrad without destructive reduction of the reflectivity.

[1] K. Yamauchi et al., *J. Synchrotron Rad*. 9 (2002), 313-316.

[2] K. Yamauchi et al., *Jpn. J. Appl. Phys*. Part1 42 (2003), 7129-7134.

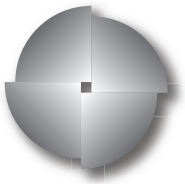
[3] Y. Mori et al., *Rev. Sci. Instrum*. 71 (2000), 4627-4632.

[4] K. Yamauchi et al., *Rev. Sci. Instrum*. 73 (2002), 4028-4033.

[5] K. Yamauchi et al., *Rev. Sci. Instrum*. 74 (2003), 2894-2898.

[6] H. Mimura et al., *Rev. Sci. Instrum*. 76 (2005) 045102.

[7] H. Mimura et al., *Jpn. J. Appl. Phys*. Part 2, 44 (2005) No.18 539



WK1

Nanofocusing with Reflective X-ray Optics

Christian Morawe
European Synchrotron Radiation Facility, BP 220, F-38043, Grenoble, France

In the context of present and future nano-focusing applications using hard x rays, the performance limits of reflecting optical devices will be discussed. Basic optical considerations of spatial resolution limits will be complemented by particular aspects of total reflecting mirrors and multilayers. The present status of ESRF fabrication techniques and recent experimental results will complete the presentation.

WK1

Fabrication of Optics for Nanofocusing of Hard X-rays

A.T. Macrander*[,1], C. Liu[1], R. Conley[1], L. Assoufid[1], A. Khounsary[1], J. Qian[1], C. Kewish[1], R. Headrick[2], and Yi-Pang Wang[2]
[1] Advanced Photon Source, Argonne National Laboratory, 9700 S. Cass Avenue, Argonne, IL 60439, USA
[2] Department of Physics, University of Vermont, Burlington, VT 05405, US

Progress in both multilayer Laue lenses (MLLs) [1,2] and Kirkpatrick-Baez (K-B) mirrors [3,4] at the Advanced Photon Source will be highlighted. Successful MLLs require multilayer depositions consisting of many layers [1]. Focusing to 30 nm for 19.5 keV has been demonstrated at APS beamlines with a WSi_2/Si MLL having 728 layers made at the APS [2]. The techniques used to achieve this result, as well as for a more recently achieved partial linear zone plate structure having a 5-nm outermost zone width and consisting of 1588 total layers, will be summarized. In addition, results corroborating the suitability of the WSi_2/Si bilayer pair for the MLL structure in an *in situ* synchrotron x-ray scattering study will be presented [5]. Achromatic focusing to 80 nm of x-rays in the range ~7 to 22 keV by an elliptically figured K-B mirror has also been demonstrated at the APS with a mirror coated at the APS [4]. The mirror was made by profile coating a substrate with Au to achieve the elliptical surface shape [3]. The elliptical mirror was made starting from a flat substrate. Essential non–x-ray–based metrology data for both the K-B effort [3,6] and the MLL effort [1] will also be highlighted.

[1] C. Liu, R. Conley, A.T. Macrander, J. Maser, H.C. Kang, M. Zurbuchen, and G.B.Stephenson, *J. Appl. Phys.* **98**, 113519 (2005).

[2] H.C. Kang, J. Maser, G.B. Stephenson, C. Liu, R. Conley, A.T. Macrander, and S. Vogt, *Phys. Rev. Lett.* **96**, 127401 (2006).

[3] C. Liu, L. Assoufid, R. Conley, and A.T. Macrander, *Opt. Eng.* **42**, 3622 (2003).

[4] W. Liu, G.E. Ice, J.Z. Tischler, A. Khounsary, C. Liu, L. Assoufid, and A.T. Macrander, *Rev. Sci. Instrum.* **76**, 113701 (2005).

[5] Y.-P. Wang, H. Zhou, L. Zhou, R. Headrick, A.T. Macrander, A.S. Ozcan, K.F. Ludwig, *J. Appl. Phys.*, submitted.

[6] G. Ice, J.-S. Chung, J.Z. Tischler, A. Lunt, and L.Assoufid, *Rev. Sci. Instrum.* **71**, 2635 (2000).

This work supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38, and under contract DEFG0203ER46032, and the National Science Foundation under contract DMR-0216704.

**E-mail: macrander@aps.anl.gov*

WK1
Sub-10 nm X-ray Microscopy: Status and Pathways

W. Yun
Xradia Inc., 4075A Sprig Drive, Concord, CA, 94520, USA

X-ray microscopy offers important and desirable visualization and characterization capabilities of great importance to broad range of scientific disciplines, including for example biology and emerging nanoscience and nanotechnology. Its short wavelength permits nanometer-resolution imaging without the limitation of wavelength. Its high penetration power allows nondestructive imaging of internal structures of an object. It also has many contrast mechanisms that can be employed beyond simple structural imaging, such as chemical state imaging or elemental specific imaging.

Over the last decade, the resolution of x-ray microscopy has improved significantly. Thanks mainly to the advancement in developing high resolution x-ray focusing optics, x-ray imaging with a spatial resolution better than 15 nm and 20 nm has been demonstrated with soft and hard x-rays, respectively. Sub-10-nm resolution x-ray imaging is expected to be realized within the next few years. My talk will present the current status of the state-of-the-art x-ray imaging in terms of spatial resolution and discuss pathways toward achieving sub-10-nm x-ray imaging.

WK1
Fabrication Techniques for High-Resolution Zone Plates: Future Directions and Limitations

J. Alexander Liddle
Center for X-ray Optics/Molecular Foundry, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

Conventional limits on the resolution of electron-beam lithography can be overcome by the use of novel resists, resist processing techniques and, most importantly, precisely overlaid exposures. Under ideal circumstances it is possible for such an approach to yield perhaps 5 nm features on a 10 nm pitch: suitable for the fabrication of a zone plate with a resolution of approximately 4 nm. However, at these dimensions the ability of such a zone plate to focus with reasonable efficiency is controlled by its thickness and hence aspect ratio of the features. I will describe our current experimental program that has lead to 15 nm outer zone width zone plates for use in the soft x-ray regime and discuss a number of strategies that might enable the fabrication of smaller zones at thicknesses appropriate for the hard x-ray energies.

Workshop 2

Nanomaterials for Energy

Organizers: David Tiede and Nenad Markovic, Argonne National Laboratory

Tuesday, May 2
Bldg. 402, Room E1100/E1200

8:30 am – 12:30 pm

8:30 – 8:40	Welcome: Molecular and Bio-inspired Materials *David Tiede*
8:40 – 9:00	Relating Photoinduced Charge Transport to Structure in Self-Assembled, Biomimetic Systems *Richard Kelley, Northwestern University*
9:00 – 9:30	Molecularly Wired Hybrid Assemblies for Solar Energy Conversion *Prashant Kamat, University of Notre Dame*
9:30 – 10:00	Designed Membrane Protein Maquettes for Light Capture and Electron Transfer *Bohdana Discher, University of Pennsylvania*
10:00 – 10:30	Designed Supramolecular Containers for Catalysis and Energy Conversion *Joseph Hupp, Northwestern University*
10:30 – 10:50	Break, Atrium and Gallery
10:50 – 11:00	Introduction: Nanoparticle Catalysis *Nenad Markovic*
11:00 – 11:30	Heterogeneous Electrocatalysis *Philip Ross, Lawrence Berkley Laboratory*
11:30 – 12:00	*In situ* X-ray Characterization of Nanoscale Catalysts *H.-D. You, Argonne National Laboratory*
12:00 – 12:30	Nanomaterials for Hydrogen Production and Conversion *D.-J. Liu, Argonne National Laboratory*

Workshop 3
Nanophotonics

Organizers: Gary Wiederrecht and Stephen Gray, Argonne National Laboratory

Tuesday, May 2
Bldg. 401, Room A5000

8:30 am – 12:00 pm

8:30 – 9:00	Nanoscale Imaging and Manufacturing with Photonic "Atoms" *Nick Fang, University of Illinois at Urbana-Champaign*
9:00 – 9:30	Plasmonic Effects in Nanoparticles and Nanoholes *George Schatz, Northwestern University*
9:30 – 10:00	Near-Field Scanning Optical Spectroscopy and Magnetoluminescence of Nitrogen-Containing Compound Semiconductors *Jim Merz, University of Notre Dame*
10:00 – 10:30	Break, Atrium and Gallery
10:30 – 11:00	Dynamics of Single NanoPlasmonic Objects *Norbert Scherer, The University of Chicago*
11:00 – 11:30	Exploration of Peptide-Gold Interfaces Using Surface-Enhanced Raman Scattering *Sandra Whaley Bishnoi, Illinois Institute of Technology*
11:30 – 12:00	Subwavelength Focusing and Guiding of Surface Plasmons *Ulrich Welp, Argonne National Laboratory*

WK3

Nanoscale Imaging and Manufacturing with Photonic "Atoms"

Nicholas X. Fang
University of Illinois at Urbana-Champaign

In this talk, I will present the design and manufacturing of sub-wavelength photonic "atoms" and "molecules" and discuss their potential applications in nanoscale imaging and nanomanufacturing. We created, for the first time, a substance with terahertz magnetic resonance by using artificially structured "molecules." Furthermore, using silver as a natural optical superlens, we demonstrated sub-diffraction-limited near-field imaging and lithography with better than 50-nm resolution. The plasmonic structures indeed promise exciting avenues to nanoscale optical imaging and highly compact optoelectronic devices.

WK3

Plasmonic Effects in Nanoparticles and Nanoholes

George C. Schatz, Kevin Shuford, and Shengli Zou
Department of Chemistry, Northwestern University

In this talk I will present the results of a variety of studies that we have done recently concerning the interaction of light with metal nanoparticles and holes in metal films. These studies have been performed using either the discrete dipole approximation (DDA) or the finite difference time domain (FDTD) method, both of which are finite element approaches for solving Maxwell's equations for light interacting with materials.

An important recent study we have done has been concerned with gold nanorods that are made by electrodeposition in an oxidized aluminum oxide membrane. These rods can be made highly monodisperse, and as a result they show multipole plasmon resonances that can be accurately modeled with DDA calculations. In addition, it is possible to make dimers and small arrays of gold disks with this technology, and this enables a detailed study of SERS intensities as a function of disk size and gap. Our calculations show why the peak Raman intensity is not associated with minimum gap size.

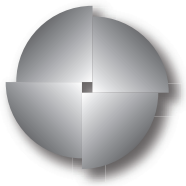
We have also studied the transmission spectra and Raman intensities associated with holes in silver films. The transmission spectra show systematic variation with hole size, as well as the presence of grooves around the holes that reflect both localized and propagating plasmon excitation. In addition, SERS intensities are enhanced by plasmon excitation, but the wavelength dependence of the SERS enhancements is quite distinct from that for transmission spectra.

WK3

Near-Field Scanning Optical Spectroscopy and Magnetoluminescence of Nitrogen-Containing Compound Semiconductors

James Merz
University of Notre Dame

Near-field magnetoluminescence and NSOM experiments on compound semiconductors will be described. Applications will include measurements on the so-called dilute nitrides (InGaAs:N), with In compositions in the range of 0-10% and only a few percent N, and multi-layer structures of InGaN/GaN. The properties of individual quantum dots are investigated in these materials, which result from N-clustering or spontaneous phase separation.



WK3

Dynamics of Single NanoPlasmonic Objects

Norbert F. Scherer
University of Chicago

This talk will illustrate a range of ultrafast optical measurements and manipulations of single metal nanoparticles (rods and strongly coupled aggregates) that define magnitudes of material nonlinearities, their relaxation dynamics and mechanism and manifestation of resultant nonlinear optical responses. It will also examine manipulation of single Au nanorods via optical tweezers and insights into particle interactions. The latter hint at new nonlinearities and field enhancements that may be possible with designed trapping potentials.

WK3

Exploration of Peptide-Gold Interfaces Using Surface-Enhanced Raman Scattering

Sandra Whaley Bishnoi
Illinois Institute of Technology

Many groups have hypothesized that short (<20 amino acid) peptides can be used for the delivery of metal nanoparticles to specific targets for either biomedical or nanofabrication applications. Though several groups have been successful in the selection of peptides that will specifically bind to metal and semiconductor surfaces from biological/combinatorial libraries (phage, bacterial, and yeast displays), there has been limited discussion of the mechanism of binding between peptides and such surfaces. Our group has recently focused on the use of molecular spectroscopy (namely, surface-enhanced Raman scattering) to study peptide-metal interfaces to help elucidate the binding mechanism of "gold-specific" peptides isolated by other groups from biological, phage, or yeast display systems. The goal of this project is to understand the molecular basis of this binding in order to improve upon the evolutionary technique used by others and design peptides for directed delivery of nanoparticles to specific biological targets.

Workshop 4

Microscopy and Imaging in Materials Science

Organizers: Qun Shen, Advanced Photon Source, Argonne National Laboratory
Brian Stephenson, Materials Science Division and Center for Nanoscale Materials, Argonne National Laboratory

Tuesday, May 2
Bldg. 401, Room A1100

8:30 am – 12:10 pm
1:30 pm – 4:20 pm

Most of our fundamental knowledge to date on solid state physics is built upon research on ideal uniform materials or statistically averaged ensembles of structures. As materials scientists focus more attention on the structure-function correlations in functional materials, spatially resolved, *in-situ*, structural studies at nanometer length scales on nonuniform, inhomogeneous materials become increasingly important. Because of this, x-ray microscopy and imaging have emerged as a growing area of synchrotron science that is ideally suited for studying structure and evolution of inhomogeneities in a vast range of materials at micrometer to nanometer scales.

These microscopy and imaging experiments range from structural studies under external gradients in functioning units such as nano-electro-mechanical and multiferroic devices, to in-situ observations of corrosion and crack propagation in alloys, to nanoscale characterizations of individual local functioning domains in macroscopically homogeneous materials such as grain boundaries in engineering materials, solar cells with efficient transport properties, and local crystallographic phases in high-Tc superconductor and giant magnetoresistive thin films. In addition, the energy tunability of synchrotron x-rays allows near-absorption-edge microspectroscopy and spatially resolved anomalous/resonant scattering studies of chemical states and chemical/orbital ordering in a variety of technologically important materials.

This full-day workshop aims to highlight the most recent advances in areas of materials science with synchrotron x-ray microscopy and imaging. Topics to be discussed will include both novel materials applications using existing, well-established techniques and emerging developments that may open up new areas of research in materials sciences.

8:30 – 8:45	Welcome *J. Murray Gibson, Argonne National Laboratory*
8:45 – 9:15	High-Resolution 3D X-ray Microscopy for Investigating Materials Microstructure *Bennett Larson, Oak Ridge National Laboratory*
9:15 – 9:45	*In situ* Synchrotron X-ray Microtomography Studies of Corrosion of Aluminum and Magnesium Alloys *Alison Davenport, University of Birmingham, UK*
9:45 – 10:15	Defect Engineering in Si Solar Cells *Tonio Buonassisi, University of California, Berkeley and Lawrence Berkeley National Laboratory*
10:15 – 10:45	Break, Atrium and Gallery

10:40 – 11:10 Imaging Nanomagnetic Systems and Dynamics
Samuel Bader, Argonne National Laboratory

11:10 – 11:40 X-ray Reflection Phase-Contrast Microscopy of Interfaces
Paul Fenter, Argonne National Laboratory

11:40 – 12:10 Controlled Synthesis of Novel Epitaxial Films of Si-Based Magnetic Semiconductors
Frank Tsui, University of North Carolina

12:10 – 13:30 Lunch, Bldg. 402, Lower Level, Tent

13:30 – 14:00 X-ray Imaging with Ultra-Small Angle X-ray Scattering as a Contrast Mechanism
Gabrielle Long, Argonne National Laboratory

14:00 – 14:30 Structure and Dynamics in Functional Materials
Paul Evans, University of Wisconsin, Madison

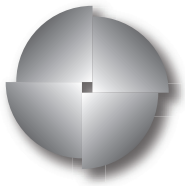
14:30 – 15:00 Fluctuation X-ray Microscopy and What It Tells Us about Medium-Range Order in Self-Assembled Materials
Michael Treacy, Arizona State University

15:00 – 15:20 Break, Atrium and Gallery

15:20 – 15:50 Nondestructive Three-Dimensional Imaging of Nanostructured Materials by Using Coherent X-rays
Jianwei Miao, University of California, Los Angeles

15:50 – 16:20 First Results on Ultrafast Coherent Diffraction Imaging Using a Free-Electron Laser
Stefano Marchesini, Lawrence Livermore National Laboratory

16:20 Adjourn



WK4

High-Resolution 3D X-Ray Microscopy for Investigating Materials Microstructure

B. C. Larson
Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Although x-ray microscopy dates to the 1940's, revolutionary advances in x-ray microscopy are now in progress. The availability of high-brilliance synchrotron x-ray sources, recent developments in high-precision x-ray focusing optics, and the development of new x-ray diffraction and contrast imaging techniques has stimulated entirely new x-ray microscopy capabilities, including submicron 3D spatial resolution. Of course, electron microscopy techniques provide structure and spectroscopy tools with better spatial resolution for samples up to a few tenths micron thickness, and electron backscattering diffraction (EBSD) microscopy provides detailed surface structure and microstructure information with submicron resolution. However, the inherently nondestructive, penetrating nature of x-ray microscopy and its *complementarity* with electron techniques are the important aspects for materials microstructure investigations addressing critical mesoscopic length scales of tens of nanometers to hundreds of microns. X-ray microscopy provides the capability of 3D submicron spatially resolved measurement [1] of local crystal structure, phase, orientation, and both elastic and plastic strain tensors in single crystals, polycrystals, composites, functionally graded materials, and deformed materials on length scales ranging from nanometer length scales to hundreds of microns and beyond.

Virtually all technological and biological materials owe their desirable physical properties to inhomogeneous electronic and atomic structures on length scales ranging from atomic sizes to nanometers, microns, and millimeters. Thus, nondestructive investigative probes with a range of penetration distances and varying spatial resolutions are essential to realize a full understanding of the microstructure, properties, and the evolution of materials. Three-dimensional measurements of thermal grain growth in polycrystals and nanoindentation-induced deformation in ductile and brittle materials will be used to illustrate the direct, quantitative link that is now possible between the microstructure and evolution in real materials and increasingly powerful computer simulations and multi-scale modeling. Ongoing developments and direct correlations between x-ray and electron microscopy of deformed metal microstructures will be discussed as well.

Research sponsored by the U.S. Department of Energy, Office of Science, Division of Materials Sciences at ORNL managed by UT-Battelle, LLC, under contract DE-AC05-00OR22725. Measurements were performed on XOR/UNI Sector-34 IDE at the Advanced Photon Source. The Advanced Photon Source is supported by the U.S. Department of Energy.

[1] B.C. Larson, W. Yang, G.E. Ice, J.D.Budai, and J.Z. Tischler, *Nature* **415**, 887 (2002).

[2] For an overview see "High-Resolution Three-dimensional X-Ray Microscopy," theme article in *Materials Research Bulletin* **29**, 152 (2004) and following papers, B.C. Larson and B. Lengler, Guest Eds.

WK4

In situ Synchrotron Microtomography Studies of Corrosion in Alloys

Alison J. Davenport
University of Birmingham, Metallurgy and Materials, Birmingham, UK

Light alloys such as aluminum and magnesium are very important structural metals in the aerospace and automotive industries. They have excellent mechanical properties but can show some susceptibility to corrosion in wet environments. The corrosion is usually localized in nature, which can lead to structural damage and even to initiation sites for environmentally assisted cracking. Localised corrosion attack is associated with chemical heterogeneity in the microstructure, particularly at grain boundaries.

Synchrotron x-ray microtomography is an excellent technique for characterizing the progress and morphology of corrosion attack and relating it to microstructural features. It offers resolution at the micron scale, and measurements can be made *in situ* in wet environments to monitor the evolution of corrosion attack in

real time. Furthermore, it is possible to characterize the morphology of fragile corrosion products and the location of gas bubbles.

Microtomography has been used to investigate intergranular corrosion of the high-strength aluminum alloy AA2024, following a new joining process called friction stir welding. The results show accelerated corrosion attack in the heat-affected zone of the weld, which propagates rapidly along grain boundaries parallel to the rolling direction of the original plate. In the "nugget" region at the center of the weld, there is significant attack, but it is localized to smaller corrosion sites, which do not spread significantly once initiated. Furthermore, it is possible to observe the dissolution of large (micron-sized) intermetallic particles in the interior of the corrosion sites. This is a significant observation, since they are electrochemically noble, and dissolution might not be expected. Furthermore, their dissolution changes the chemistry of the corrosion cavity.

Investigations have also been made of AA2024 surfaces that have been laser treated to improve their corrosion resistance. It is evident that the laser-treated layer itself is highly resistant to corrosion, but there is a region underneath the layer that shows enhanced susceptibility to attack, which propagates laterally along the surface.

Aluminum alloys of the 6XXX series are used for automotive applications. They are also susceptible to intergranular attack after heat treatment. Tomography measurements of these alloys also show variation in the morphology of attack after different heat treatments.

WE43 is a Mg-Y alloy used for aerospace applications. Yttrium is added to improve the corrosion resistance of the alloy, but its distribution must be highly homogeneous. X-ray microtomography above and below the Y edge has been used to determine the distribution of Y in an as-cased Mg-Y alloy, which can be related to its corrosion susceptibility.

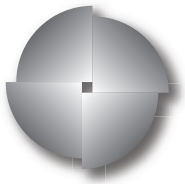
WK4
Defect Engineering in Si Solar Cells

Tonio Buonassisi
Department of Materials Science & Engineering, University of California, Berkeley, CA 94720

Photovoltaics (PV)—the conversion of sunlight into electricity—offers the promise of a clean, domestically produced, renewable energy source. However, the high cost of conventional PV relative to conventional fossil-fuel-based sources is currently an obstacle for widespread implementation. To overcome this cost barrier, the PV research community is developing novel multicrystalline silicon materials employing rapid-growth technologies and cheaper silicon sources. However, precisely because of these low-cost growth methods and materials, transition-metal impurities and defects limit the efficiencies of these next-generation devices, partially offsetting their cost advantage. The small size and inhomogeneous distributions of these efficiency-limiting transition-metal defects hinder their study via traditional analytical techniques.

To reveal the distribution and chemical state of efficiency-limiting defects in cost-effective solar cells, our group employed a suite of synchrotron-based analytical techniques, including x-ray fluorescence microscopy (μ-XRF) and x-ray absorption microspectroscopy (μ-XAS), which can detect and characterize the elemental natures and chemical states of metal-rich inclusions and precipitates as small as about 30 nanometers. Additionally, a novel technique, spectrally resolved x-ray–beam–induced current (SR-XBIC), was developed to measure the impact of metal-rich micro- and nanoprecipitates on the local minority carrier diffusion length, a critical parameter for determining solar cell efficiency.

Statistically meaningful data was obtained on the elemental, chemical, size, and spatial distributions of efficiency-limiting, transition-metal-related micro- and nanodefects in a variety of commercial and R&D solar cell materials. From this body of data, (1) we identified several possible origins of transition metal contamination in solar cell material; (2) we established strong relationships between crystal growth conditions,



solar cell processing, metal impurity distributions and states, and their impact on material performance; and, most importantly, (3) we demonstrated that the distributions and states of metal nanodefects—and thus, their impact on cell efficiency—can be controlled via designed processes, termed "defect engineering," offering the potential for producing cost-effective solar cells from impurity-rich, lower-cost silicon materials.

WK4
Imaging Nanomagnetic Systems and Dynamics

S. D. Bader
Materials Science Division and Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439

This talk will provide an overview of the grand challenges in the emerging discipline of nanomagnetism from basic and applied perspectives [1] and then will focus on the synergistic information [2] that can be provided by x-ray imaging experiments that explore the dynamic behavior of metallic systems. Examples of complementary PEEM studies at the ALS [3], SLS [4], and APS [5] will be used to illustrate the ability of x-rays to address cutting edge issues in the field. The outlook for the future will be visited by highlighting the recent report [6] on the utilization of x-rays to explore nanomagnetism and will include anticipated developments associated with the CNM x-ray nanoprobe beamline at the APS and the PEEM-3 project at the ALS.

[1] S. D. Bader, *Rev. Mod. Phys*. **78,** 1 (2006).
[2] K. S. Buchanan, et al., *Nature Physics*. **1,** 172 (2005).
[3] S.-B. Choe, et al., *Science* **304,** 420 (2004).
[4] J. Raabe, et al,. *Phys. Rev. Lett*. **94,** 217204 (2005).
[5] K. Y. Guslienko, et al., *Phys, Rev. Lett*. **96,** 067205 (2006).
[6] G. Srajer, et al., *J. Magn. Magn. Mater.* (submitted, 2006).

Work supported by DOE-BES, under contract W-31-109-ENG-38.

WK4
X-ray Reflection Phase Contrast Microscopy of Interfaces

Paul Fenter
Chemistry Division, Argonne National Laboratory, Argonne, IL 60439

The various forms of interfacial x-ray scattering (x-ray reflectivity, grazing incidence scattering, crystal truncation rods, etc.) have been extremely successful in providing an unprecedented understanding of interfacial processes (e.g., adsorption, growth, dissolution) through direct *in situ* measurements in a wide range of environments, and even as a function of time. With the development of third-generation synchrotron facilities, perhaps the primary limitation that has yet to be addressed is the inherent spatial averaging associated with these measurements. This question is especially important in studies of geochemical interfaces because large single crystals of many important minerals are unavailable, and therefore it becomes essentially impossible to probe such interfaces with traditional high-resolution x-ray scattering. We have recently built a new instrument that couples the recent advances in x-ray optics (e.g., Fresnel zone plates) to image the interface topography directly using the weak specularly reflected x-ray beam. In essence, this x-ray reflection interface microscope (XRIM) is the x-ray analogue of the low-energy electron microscopy (LEEM). I will describe the conceptual design for this instrument and the phase-contrast mechanisms that provide sensitivity to the interfacial topography. Recent results for the orthoclase (001) surface obtained with this instrument will be presented. Future opportunities and challenges of applying this approach to the *in situ* studies of interfacial processes in real time and with sensitivity to molecular-scale features will be discussed.

This work was supported by the U.S. Department of Energy (BES, Division of Chemical Sciences, Geosciences, and Biosciences) under contract number W-31-109-ENG-38 at Argonne National Laboratory. This work was done at BESSRC (sector 12) of the Advanced Photon Source.

This work was done in collaboration with Changyong Park, Zhan Zhang, Jeffrey Catalano (Chemistry Division, Argonne) and Steve Wang (Xradia). We thank Wenbing Yun and Michael Feser (Xradia) for help in the initial design of the instrument and the BESSRC staff for their assistance.

WK4

Controlled Synthesis of Novel Epitaxial Films of Si-Based Magnetic Semiconductors

Frank Tsui
Department of Physics and Astronomy, University of North Carolina at Chapel Hill, Chapel Hill, NC 27599-3255

One of the most exciting prospects of materials science is the ability to tailor materials and properties through atomic-scale synthesis. By employing combinatorial molecular beam epitaxy (MBE) techniques, promising epitaxial films and heterostructures of group IV–based magnetic semiconductors have been synthesized by using complementary co-doping of metal dopants [1,2]. We have shown that two transition-metal dopants can be used to control the energetics and kinetics of epitaxial growth of group IV–based magnetic semiconductors. Specifically, the use of two dopants, one with a larger atomic radius and another one smaller than that of the group IV host, and at an appropriate atomic ratio between the two, can compensate for the internal stress caused by the individual dopants in the host lattice, and thus stabilize epitaxial growth at doping concentrations as high as 15 at. % [2], a level several times higher than those from using a single dopant. Our results also indicate that the presence of a second dopant can significantly reduce the tendency for clustering of the individual dopants. While the concentration of magnetic ions can be controlled by the transition metal dopants, a third metal dopant, e.g., a group III element, can also be used to fine-tune the electronic structure, particularly carrier concentration, which in turn controls the magnetic ordering. These findings provide a general approach for synthesizing high-quality epitaxial films of magnetic semiconductors with tailored magnetic and electronic properties, including high Curie temperature and high spin polarization. Our ability for combinatorial MBE synthesis when combined with the advanced instrumentation for x-ray microbeams experiments developed at the APS have made it possible for systematic studies of MBE synthesis and structural and chemical properties of these novel materials.

[1] F. Tsui, L. He, L. Ma, A. Tkachuk, Y. S. Chu, K. Nakajima, T. Chikyow, *Phys. Rev. Lett*. **91**, 177203 (2003).

[2] F. Tsui, L. He, A. Tkachuk, S. Vogt, Y. S. Chu, *Phys. Rev*. B **69**, 081304R (2004).

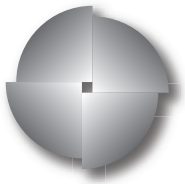
The work has been carried out in collaboration with Dr. Liang He and Mr. Brian Collins at UNC-CH, and Dr. Yong Chu and co-workers at beamline 2-BM of the APS. The work at UNC-CH is supported in part by NSF DMR-0441218 and DOE BES DE-FG02-05ER46216. Use of the APS was supported by DOE BES W-31-109-ENG-38 and one of us (BC) received half-time support by an APS/DOE Subcontract No. 5F-00428.

WK4

X-ray Imaging with Ultra-Small-Angle X-ray Scattering as a Contrast Mechanism

G. G. Long[1] and L.E. Levine[2]
[1] *Argonne National Laboratory, Argonne, IL 60439*
[2] *National Institute of Standards and Technology, Gaithersburg, MD 20899-8553*

Small-angle x-ray scattering is a technique well-known for its ability to provide direct statistical volume-averaged microstructural information from bulk speciments. However, it provides almost no information on how the scattering objects are distributed. USAXS imaging uses ultra-small-angle x-ray scattering (USAXS) as a contrast mechanism. In conjunction with small-angle scattering, it can provide quantitative and qualitative three-dimensional information on the distribution and the microstructural parameters of scattering objects, and it sometimes provides contrast in cases where radiography and other forms of phase-contrast imaging are unsuccessful. USAXS imaging greatly extends the size range over which microstructural information can be obtained, reaching to well over a micrometer. Remarkably, imaging as a function of scattering vector enables the direct determination of shape and size information even when the scattering objects are smaller than the spatial resolution of the images.



WK4

Structure and Dynamics in Functional Materials

Paul G. Evans
University of Wisconsin-Madison, Madison, WI 53706

The electronic properties of functional materials are in many cases intimately linked to subtle structural effects. Often, electronic phenomena are linked not only to the static structure of materials but to large transient structural perturbations. These structural changes have a large role in the electronic properties of materials and also serve as diagnostics for physical phenomena in electronic materials. We will discuss two cases in which transient changes in the structure of materials can be probed at the appropriate length and time scales by x-ray techniques. In the quasi-one-dimensional charge density wave material $NbSe_3$, the electronic properties at high electric fields are dominated by the transition at which the charge density wave depins and begins to contribute to electrical conduction. X-ray microdiffraction allows the structural signature of the depinning transition to be resolved as across micron-scale features. In ferroelectric materials, the electronic and structural effects associated with polarization switching can be probed dynamically using time-resolved microdiffraction techniques that match the lengths and times applicable to domain wall propagation [2]. Domain wall velocities and piezoelectric effects can be observed microscopically and connected to the operation of devices and to the fundamental properties of ferroelectric materials.

[1] A. F. Isakovic, P. G. Evans, J. Kmetko, K. Cicak, Z. Cai, B. Lai, and R. E. Thorne, "Shear Modulus and Plasticity of a Driven Charge DensityWave," Phys. Rev. Lett. **96**, 046401 (2006).

[2] A. Grigoriev, D.-H. Do, D. M. Kim, C.-B. Eom, B. Adams, E. M. Dufresne, and P. G. Evans, "Nanosecond domain wall dynamics in ferroelectric $Pb(Zr,Ti)O_3$ thin films," Phys. Rev. Lett., in press.

WK4

Fluctuation X-ray Microscopy and What It Tells Us about Medium-Range Order in Self-Assembled Materials

M. M. J. Treacy[1], D. Kumar[1], D. J. Paterson[2], L. Fan[3], I. McNulty,[3] and J. M. Gibson[3]
[1] *Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85284*
[2] *Australian Synchrotron, Clayton, Victoria 3168, Australia*
[3] *Advanced Photon Source, Argonne National Laboratory, Argonne IL 60439*

Fluctuation microscopy is a hybrid imaging-diffraction technique that is sensitive to medium-range order in disordered materials. It is significantly more sensitive than either imaging or pure diffraction on its own. The technique examines the scattering fluctuations between small volumes of disordered samples. The normalized variance of the diffracted intensity, as a function of scattering vector and image resolution, reveals the presence of the medium-range order. The sensitivity to medium-range order comes about because the normalized variance depends on four-body correlations, whereas pure diffraction depends on two-body correlations.

The technique was originally developed for transmission electron microscopy (TEM) studies of amorphous tetrahedral semiconductors, since the TEM provides ready access to both imaging and diffraction information. Fluctuation electron microscopy (FEM) has been successful in studying changes in medium-range order in these materials. Fluctuation x-ray microscopy (FXM) is now practical with recent advances in x-ray microscopy and coherent scattering. We demonstrated FXM on self-assembled nanophase materials at the Advanced Photon Source 2-ID-B soft x-ray beamline.

In this talk, I will present an overview of the fluctuation microscopy method, and will describe our FXM results for disordered assemblies of latex spheres and mesoporous silica gels. I will also describe ideas for enhancing the sensitivity of the technique, and for improving quantitative estimates of medium-range order length scales.

WK4

Nondestructive Three-Dimensional Imaging of Nanostructured Materials by Using Coherent X-rays

Jianwei Miao
Department of Physics and Astronomy and the California Nanosystems Institute, University of California, Los Angeles, CA 90095-1547

Because x-rays have a longer penetration depth than electrons and x-ray wavelengths are on the order of the size of atoms, there exists the prospect for atomic-resolution x-ray microscopes that could visualize arrangement and dynamics of atoms in three dimensions—without the requirement for periodicity (e.g., crystals). X-rays, however, are much more difficult to focus than electrons. By using Fresnel zone plates, the smallest focal spot currently achievable is 50 nm for hard x-rays and 15 nm for soft-x-rays. With third-generation synchrotron radiation, we have developed a lensless microscope, which is based upon coherent x-ray scattering in combination with a method of direct phase recovery called oversampling. By using this novel x-ray microscope, we have investigated nanostructured materials in three dimensions. Our work opens a door for nondestructive imaging of 3D morphology and 3D internal structures of a wide range of samples, including porous materials, semiconductors, quantum dots and wires, inorganic nanostructures, and biomaterials, at nanometer resolution.

WK4

First Results on Ultrafast Coherent Diffraction Imaging Using a Free-Electron Laser

Stefano Marchesini
Lawrence Livermore National Laboratory, Livermore, CA 94539

By avoiding the use of lenses, coherent diffraction imaging promises a resolution limited only by radiation damage. Theoretical studies predict that pulses faster than the damage process could enable the imaging of single molecules without the need for crystals. We present the first experimental verification of the principle of flash diffraction imaging using a soft x-ray free-electron laser. Experiments aimed at the characterization of the dynamics of organic matter under intense radiation and validation of theoretical models will be discussed.

Workshop 5
Nanopatterning

Organizers: John Rogers, University of Illinois at Urbana-Champaign
Leonidas Ocola, Argonne National Laboratory

Tuesday, May 2
Bldg. 402, Room E1100/E1200

1:30 pm – 5:30 pm

Progress in nanoscience and nanotechnology depends strongly on the capabilities of techniques for patterning and chemically modifying materials at the nanoscale. The methods that are currently available span a very wide range, from those that have emerged from research and development in the microelectronics area (e.g., electron beam lithography, photolithography, etc.) to newer classes of low-cost techniques designed to enable manipulation of soft materials. This workshop covers, in a broad way, recent developments in nanopatterning with applications. Speakers will address the general topics indicated below.

1:30 – 1:55	Electron beam lithography *Don Tennant, Lucent Technologies*
1:55 – 2:20	Holographic 3D lithography *Shu Yang, University of Pennsylvania*
2:20 – 2:45	Patterned organic semiconductors *Zhenan Bao, Stanford University*
2:45 – 3:00	Block copolymer lithography *Paul Nealey, University of Wisconsin*
3:00 – 3:25	Break, Atrium and Gallery
3:25 – 3:50	Robocasting *Jennifer Lewis, University of Illinois at Urbana-Champaign*
3:50 – 4:15	Colloidal lithography *Paul Braun, University of Illinois at Urbana-Champaign*
4:15 – 4:40	Nano xerography *Heiko Jacobs, University of Minnesota*
4:40 – 5:05	Self-assembly *Ned Bowden, University of Iowa*
5:05 – 5:30	Wrap-up discussion

Workshop 6

Quantum Nanomagnetism

Organizers: Eric Isaacs and Axel Hoffmann, Center for Nanoscale Materials, Argonne National Laboratory

Tuesday, May 2
Bldg. 401, Room A5000

1:30 – 5:30 pm

1:30 – 2:05	Quantum Tunneling of Spin Density Wave Domain Walls in Chromium *Oleg Shpyrko, Argonne National Laboratory*
2:05 – 2:40	Self-Organization in a Quantum Spin Liquid *Thomas F. Rosenbaum, The University of Chicago*
2:40 – 3:15	Artificial Spin Ice: Frustration in a Patterned Array of Nanoscale Ferromagnetic Islands *Peter Schiffer, Pennsylvania State University*
3:15 – 3:35	Break, Atrium and Gallery
3:35 – 4:10	Soliton Dynamics in Nanomagnets *Kristin Buchanan, Argonne National Laboratory*
4:10 – 4:55	Mesoscopic Fluctuations in Cobalt Nanoparticles *Dragomir Davidovic, Georgia Institute of Technology*
4:55 – 5:30	Highly Anisotropic Nanomagnets *Alejandra Lukaszew, University of Toledo*

WK6

Quantum Tunneling of Spin Density Wave Domain Walls in Chromium

Oleg Shpyrko
Argonne National Laboratory

We present coherent x-ray speckle measurements of the dynamics of domain walls separating microscopic regions with different orientations of the spin- (charge-) density waves in bulk Cr samples. Nanoscale domain wall fluctuations slow down as the sample temperature is lowered, consistent with the classical thermal activation model. Below 30K, however, the characteristic switching frequency remains constant, pointing to the possibility of quantum mechanical tunneling dominating "frozen out" thermally driven fluctuations. Our measurements provide insight into the role played by quantum fluctuations in spin dynamics of Cr and possibly other spin density wave materials.

WK6

Self-Organization in a Quantum Spin Liquid

Thomas F. Rosenbaum
The University of Chicago

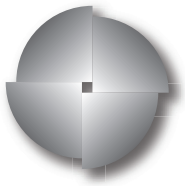
Quantum fluctuations combined with disorder can lead to physics dominated by locally organized spin clusters, which can be used to encode and manipulate information. The nonlinear response is also exquisitively sensitive to the ground state character for systems with competing order parameters. I will report results on spin liquids stabilized to the lowest temperatures by quantum fluctuations [$Li(Ho,Y)F_4$] and by geometrical frustration [GGG]. We are able to burn holes in the spectral response in analogy to optical bleaching, but we use a wire loop at 1 Hz rather than a laser at terahertz frequency, with an excitation field a fraction of the earth's magnetic field! The resulting spin excitations retain phase coherence with the pump pulse for many seconds after the pump is turned off, returning to their ground state via a process paralleling free induction decay in NMR experiments. Holes can be burned simultaneously at different frequencies, making it possible to encode independent bits of information in the noninteracting eigenstates of the system. Finally, we show that it is possible to quantum-mechanically project and tune the admixture of up and down spin states using kOe laboratory fields.

WK6

Artificial Spin Ice: Frustration in a Patterned Array of Nanoscale Ferromagnetic Islands

Peter Schiffer
Pennsylvania State University

Geometrical frustration among spins in magnetic materials can lead to exotic low-temperature states, including "spin ice," in which the local moments mimic the frustration of hydrogen ion positions in frozen water. We have performed extensive studies of spin ice materials and related compounds and recently have begun study of an artificial geometrically frustrated magnet which shares many of the properties of the spin ice materials. This artificial frustrated system is based on an array of lithographically fabricated single-domain ferromagnetic islands. The islands are arranged such that the dipolar interactions between them are analogous to those in spin ice. Images of the magnetic moments of individual elements in this correlated system allow us to study the local accommodation of frustration. We see both ice-like short-range correlations and an absence of long-range correlations, behavior that is very similar to the low-temperature



state of spin ice. These results shed light on the nature of frustration in patterned arrays and correspondingly demonstrate that artificial frustrated magnets can provide a rich new arena in which to study the physics of frustration.

J. Snyder *et al*. (PRL 2003 and PRB 2004).

R. F. Wang *et al*. (Nature 2006).

G. Lau *et al*. (Nature Physics 2006).

This research was supported by the Army Research Office and the National Science Foundation.

WK6

Soliton Dynamics in Nanomagnets

Kristin Buchanan
Argonne National Laboratory

The dynamic properties of patterned nanomagnets are of fundamental interest and also determine their suitability for high-speed applications. Magnetic vortices, observed in a wide variety of magnetically-soft structures, possess a topological singularity that controls much of the interesting physics. Thus they are a type of soliton, a phenomenon observed in many physical systems. Magnetic vortices in confined geometries provide a model system for exploring the rich physics of soliton dynamics. Elliptical nanodots can take on a single vortex or vortex pair magnetization state, providing a convenient model system for investigating the effects of both geometric confinement and dynamic vortex interactions. Experimentally we have measured the eigenmodes of Permalloy ellipses (2 x 1 μm^2 and 3 x 1.5 μm^2, 40 nm thick), fabricated using CNM lithography facilities, using a microwave reflection technique. We find that the frequency of the single-vortex translational mode (~100 MHz), a characteristic mode that corresponds to spiral-like motion of the vortex core, can be tuned by varying the ellipse geometry or by applying a magnetic field. This tuneability can be understood in terms of the shape of the vortex energy profile extracted from micromagnetic simulations. The vortex pairs exhibit a much richer excitation spectrum [1]. Dynamically, the relative polarizations of the vortex cores (parallel or antiparallel) play a defining role in spite of their small size (~20 nm).

[1] K. S. Buchanan, P. E. Roy, M. Grimsditch, F. Y. Fradin, K. Yu. Guslienko, S. D. Bader, and V. Novosad, *Nature Physics* **1**, 172-176 (2005).

WK6

Mesoscopic Fluctuations in Cobalt Nanoparticles

Dragomir Davidovic
Georgia Institute of Technology

We present the first measurements of mesoscopic resistance fluctuations in ferromagnetic nanoparticles as a function of magnetization reversal processes. Mesoscopic fingerprints are extremely sensitive to domain wall nucleation process; a domain wall induces an electron wavefunction phase shift of $\approx 5\pi$. The phase shift is not caused by the Aharonov-Bohm effect; we explain how it arises from the spin-mistracking effect, where electron spins lag in orientation with respect to the moments inside the domain wall. Dephasing time in Co at 0.03K is short, $\tau_\phi \sim$ ps, which we attribute to the strong magnetocrystalline anisotropy. Alternative physical origins of dephasing and fingerprints in mesoscopic ferromagnets will be discussed.

WK6

Highly Anisotropic Nanomagnets

Alejandra Lukaszew
University of Toledo

Nanomagnetism is very active area of research because of fundamental interest in material-size-property correlations, as well as novel applications. One of the driving applications is the development of new perpendicular magnetic media that can satisfy the demands for ever-increasing data strorage density. These new media for perpendicular magneto-recording must satisfy three important requirements: (1) the material must have large magnetic anisotropy to withstand the superparamagnetic limit upon size reduction; (2) the magnetic anisotropy must be perpendicular to the surface; and (3) these two conditions must persist upon nanopatterning. Nature offers particular phases of the FePt and FePd binary alloy systems that are possible candidates; namely, the $L1_0$ phase exhibits significant magnetic anisotropy, and thin films and nanostructures can be prepared with perpendicular anisotropy axis. Two main approaches are currently been investigated by several groups in order to achieve nanopatterning while preserving the two other conditions using these alloys. "Top-down" methods involve self-assembly of alloy magnetic nanoparticles, while in "bottom-up" methods the material is deposited and nanopatterning is induced during growth. In my talk I will present two variations of the bottom-up approach applied to FePd and FePt alloyed thin films and nanostructures. First I will show correlated structural and magnetic properties studies on epitaxial FePd films grown on (001) MgO. This particular system does exhibit perpendicular magnetic anisotropy and it is also nanostructured, because of a three-dimensional growth mode on the chosen substrate. The highly ordered nanostructures embedded in the chemically disordered alloy film are magnetically coupled, and we find that capping the films with a thin MgO layer significantly lowers interparticle coupling. For the FePt system, we have performed x-ray rapid thermal annealing (XRTA) treatments at the APS on partially ordered alloy thin films to enhance the chemical order while performing real-time structure characterization. We have demonstrated that it is possible to enhance the chemical ordering without significantly affecting the nanoparticle size. Finally, I will show our latest experiments where we have exploited ion-implantation of Fe ions on epitaxial Pt films followed by thermal treatments. In this latter case, we have achieved controlled nanopatterning of uncoupled $L1_0$ FePt nanoclusters with large perpendicular magnetic anisotropy.

Funding for this research was provided by NSF, ACS, and the Research Corporation.

Workshop 7

Texture and Strain Mapping with X-rays, Neutrons, and Electrons

Organizers: Dean R. Haeffner, Advanced Photon Source, Argonne National Laboratory
James W. Richardson, Jr., Intense Pulsed Neutron Source, Argonne National Laboratory
Dean J. Miller, Electron Microscopy Center, Argonne National Laboratory

Thursday, May 4
Bldg. 401, Room A1100

8:30 am - 12:00 pm
1:30 pm - 5:00 pm

Information on the orientation of grains and the way that macroscopic strain is distributed among grains is crucial for understanding the mechanical properties of polycrystalline materials. For composite systems, knowledge of the behavior of the different constituent materials is often difficult or impossible to obtain with macroscopic experimental methods. For material that is formed into mechanical components, a part's shape can lead to strong strain gradients under applied load.

Diffraction measurements with x-rays, neutrons, or electrons can determine the texture and strain as a function of position in a material, with each probe having its own particular advantages. Recent improvements in the capabilities of synchrotron light sources, neutron sources, and electron microscopes have led to increased numbers of experiments that use texture and strain mapping. This workshop will feature talks in a wide variety of scientific areas, using one or more of the three probes for mapping studies.

8:30 – 8:40	Welcome
8:40 – 9:10	Strain Pole Figures *Tom Holden, Northern Stress Technologies*
9:10 – 9: 40	Quantitative Strain Analysis Using Synchrotron X-ray Data *Matt Miller, Cornell University*
9:40 – 10:10	Electron Backscatter Diffraction and Orientation Imaging Microscopy for Strain, Texture, and Microstructural Characterization *Matt Nowell, EDAX Inc. / TSL*
10:10 – 10:30	Break, Atrium and Gallery
10:30 – 11:00	Through-Thickness Grain Structure in $YBa_2Cu_3O_7$ Films Using EBSD *Matthew Feldmann, University of Wisconsin-Madison*
11:00 – 11:30	Temperature and Direction Dependence of Internal Strain and Texture Evolution during Deformation of Uranium *Don Brown, Los Alamos National Laboratory*
11:30 – 12:00	High-Energy X-ray Scattering and Mapping in Mineralized Tissue: Sea Urchin Teeth and Bones under Load *Stuart Stock, Northwestern University*
12:00 – 1:30	Lunch, Bldg. 402, Lower Level, Tent

1:30 – 2:00 New Scientific Opportunities in Engineering Studies at the Spallation Neutron Source
Xun-li Wang, Oak Ridge National Laboratory

2:00 – 2:30 Title unavailable
Amit Goyal, Oak Ridge National Laboratory

2:30 – 3:00 Strain Mapping at the APS 1-ID Beamline
Jon Almer, Argonne National Laboratory

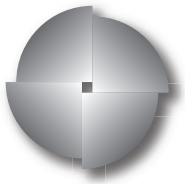
3:00 – 3:20 Break, Atrium and Gallery

3:20 – 3:50 Performance and Initial Studies at the New ORNL Neutron Residual Stress Mapping Facility
Cam Hubbard, Oak Ridge National Laboratory

3:50 – 4:20 Depth-Resolved Stress Profiling in Nanocrystalline Films with Sub-Micron Resolution
Gang Chen, Argonne National Laboratory

4:20 – 4:50 3D Texture and Strain Mapping with Polychromatic Micro/Nano Beams
Gene Ice, Oak Ridge National Laboratory

4:50 – 5:00 Wrap-up



WK7

Quantitative Strain Analysis Using Synchrotron X-ray Data

Matt Miller
Sibley School of Mechanical and Aerospace Engineering, Cornell University

Progress made in the area of quantitative texture analysis (QTA) has enabled a significant amount of understanding and model development to occur in the area of deformation-induced internal structure evolution in polycrystalline materials. This talk describes a "quantitative strain analysis" methodology that parallels the QTA process of pole density figure inversion to produce an orientation distribution function (ODF). We measure lattice strain pole figures (SPFs) and have developed an inversion algorithm for determination of a lattice strain distribution function (LSDF). This tensor field of elastic crystal strains can be used along with elastic moduli to determine the average crystal stress, orientation by orientation. This information is fundamental to understanding the anisotropic and inherently multiaxial response that occurs on the crystal scale during elastic-plastic deformation. Coupled with crystal-based simulations of material behavior, these data create a powerful processing and performance prediction tool for metallic systems. We have developed a method for conducting *in situ* mechanical tests/synchrotron x-ray diffraction experiments for measuring SPFs and their evolution on various single-phase and multiphase alloys. Results from these tests will be presented, along with their numerical analogs. Recent work employing cyclic loading will also be presented.

WK7

Through-Thickness Grain Structure in $YBa_2Cu_3O_7$ Films Using EBSD

Matthew Feldman
University of Wisconsin-Madison

Second generation superconducting wires, or coated conductors, consist of a YBCO layer (0.3 - 3.0 microns thick) deposited on an oxide buffered metal tape. Using ion milling and EBSD serially, 2D EBSD scans may be taken through the thickness of these films. In this way the grain structure of the YBCO may be studied through the film thickness and compared to the grain orientations of the underlying texture template. Using this procedure, a loss of epitaxy in some thicker YBCO films will be demonstrated, along with different degrees of epitaxial growth resulting from different YBCO deposition methods.

WK7

Temperature and Direction Dependence of Internal Strain and Texture Evolution during Deformation of Uranium

D. W. Brown, M. A. M. Bourke, B. Clausen, D. Korzekwa, R. Korzekwa, T. A. Sisneros, and D. F. Teter
Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Depleted uranium is of current programmatic interest at Los Alamos National Laboratory because of its high density and nuclear applications. At room temperature, depleted uranium displays an orthorhombic crystal structure with highly anisotropic mechanical and thermal properties. For instance, the coefficient of thermal expansion is roughly 20 x 10^{-6}/°C in the **a** and **c** directions, but negative in the b direction. The innate anisotropy combined with thermo-mechanical processing during manufacture result in spatially varying residual stresses and crystallographic texture, which can cause distortion, and failure in completed parts, effectively wasting resources. This paper focuses on the development of residual stresses and textures during deformation at room and elevated temperatures with an eye on the future development of computational models based on the known micromechanical deformation mechanisms of the material.

WK7

High-Energy X-ray Scattering and Mapping in Mineralized Tissue: Sea Urchin Teeth and Bones under Load

Stuart R. Stock
Northwestern University

Different organisms employ varied materials designs for mechanical structures employing mineralized tissues. Sea urchins form these tissues from calcium carbonate (high magnesium calcite) and produce complex geometries of single crystalline material millimeters in dimension. Vertebrates base their skeletons on a discontinuously reinforced composite design: nanocrystallites of calcium phosphate (carbonated apatite) in a collagen matrix. Contrasting the structures in these two phyla gives some indication of the design space for calcium-based biostructures. Spatial mapping with high energy x-ray scattering has been used to study sea urchin teeth and mammalian bones. Results on calcite magnesium composition changes with position in sea urchin teeth are presented and illustrate one reinforcement strategy in this animal. In mammalian bones under applied load, internal stress/strain measurements illustrate how this biocomposite functions.

WK7

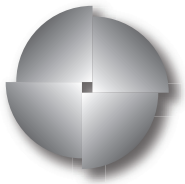
New Scientific Opportunities in Engineering Studies at the Spallation Neutron Source

Xun-Li Wang[1], T. M. Holden[2], A. D. Stoica[1], P. K. Liaw[3], H. Choo[3], and C. R. Hubbard[4]
[1] *Spallation Neutron Source, Oak Ridge National Laboratory, Oak Ridge, TN 379831-6474, USA*
[2] *Northern Stress Technology, Deep River, ON KOJ1P0, Canada*
[3] *Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996-2200*
[4] *Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 379831-6064, USA*

A dedicated engineering diffractometer is under construction at the Spallation Neutron Source (SNS) in Oak Ridge, Tennessee. The instrument is named VULCAN, after the Roman god of fire and metalworking. Design goals for VULCAN include the following:

- rapid volumetric (3-dimensional) mapping with a sampling volume of ~mm^3 and a measurement time of minutes
- very high spatial resolution (0.1 mm) in one direction
- ~20 well-defined reflections for *in situ* loading studies
- ability to study kinetic behaviors in sub-seconds
- simultaneous characterization capabilities, including dilatometry, weight, and microstructure.

Special sample environments, such as load-frame and furnaces, will be provided as an integrated part of the instrument. With the unprecedented flux provided by the SNS and improved instrumentation, it will be possible to conduct *in situ*, time-resolved measurements under real thermal-mechanical deformation conditions. A state-of-the-art, event-based data acquisition system will greatly facilitate the analysis of time-resolved data. Simultaneous measurements of diffraction and small angle scattering data will prove to be particularly useful for investigating the kinetics of complex phase transformations where the structure is evolving at multiple length scales. New scientific opportunities with VULCAN and the complementarity with synchrotron-based instruments will be discussed. The instrument is on schedule to be commissioned in the spring of 2008.



WK7

Performance and Initial Studies at the New ORNL Neutron Residual Stress Mapping Facility

C.R. Hubbard, W. B. Bailey, F. Tang, K. An, H. Choo, E.A. Payzant, and T. Watkins
Oak Ridge National Laboratory, Oak Ridge, TN 37830

The installation and testing of the new, second generation neutron residual stress mapping facility at HFIR is nearing completion and productive user and laboratory studies are being conducted. The facility at beam line HB-2B of HFIR consists of the following major components: (1) twin silicon multi-wafer, doubly focusing monochromator which provides six different wavelengths; (2) large capacity sample XY and rotation stage with two optionally mounted Z-stages; (3) a seven-position sensitive detector array; and (4) precision interchangeable incident and diffracted beam slits for definition of the gage volume. Accessories include a uniaxial tension/compression mechanical properties load frame and a Huber phi-chi sample orienter. LabView software packages NRSF2-MAP, NRSF2-LOAD, and NRSF2-VIEW work together and provide robust, easily learned, real-time data collection, calibration, and analysis tools.

The facility has been used for a number of industrial, academic, and laboratory projects. Research projects conducted on NRSF2 during the commissioning and testing period fall into the following three classifications:

(1) Mapping of residual strains in welds, compact tension fatigue specimens, quenched rods, cast and machined cylinder liners, nuclear reactor tubes, and cylinders deformed in tension and torsion.
(2) Materials deformation behavior under applied load in aluminum metal matrix composites, aluminum alloys, porous ceramic filter materials, and of compaction of granular quartz particles
(3) Phase and hydrogen content mapping in Zircalloy using both Bragg peak and incoherent background intensity
(4) Dynamic real time experiments such as real-time mechanical property loading and a moving hot spot simulating the heat input from friction stir welding

The High Temperature Materials Laboratory User Program (www.html.ornl.gov), sponsored by DOE-EERE Office of FreedomCAR and Vehicle Technologies, provides nonproprietary and proprietary research opportunities for industry, university and other laboratory engineers and scientists.

WK7

Depth-Resolved Stress Profiling in Nanocrystalline Films with Sub-Micron Resolution

Gang Chen[1], Dileep Singh[1], Osman Eryilmaz[1], Ali Erdemir[1], Jules Routbort[1], Bennett Larson[2], and Wenjun Liu[3]
[1] *Energy Technology Division, Argonne National Laboratory*
[2] *Materials Science and Technology Division, Oak Ridge National Laboratory*
[3] *Advanced Photon Source, Argonne National Laboratory*

Thin-film hard coatings (a few microns in thickness) are used for various applications for protecting bulk substrates from abrasion, corrosion, or wear or to provide low friction. Performance and adhesion of the coatings is closely related to the residual stresses throughout the film and into the substrate. Therefore, understanding the residual stresses is essential for developing excellent coatings with desired properties.

Typical stress-measurement techniques provide averaged information over the entire depth of thin films, providing little information of the stress profile. Microdiffraction with sub-micron x-ray beam size is a powerful tool to fill this gap, i.e., to measure depth-resolved stress profile in thin films with high spatial resolution.

Materials selected for this study are hard Mo and MoN thin films on Si substrates, which are fabricated by physical vapor deposition. These crystalline films have a dense columnar structure with average grain

size of 10–50 nm. By using sub-micron beam x-ray diffraction on XOR/UNI 34-ID beamline, we obtained depth-resolved stress profiles in the as-deposited and annealed films. The effect of annealing on the stress profile of the thin films is discussed. Our results demonstrate that microdiffraction can be used for studying depth-resolved stresses in nanocrystalline thin films with sub-micron resolution.

WK7

3D Texture and Strain Mapping with Polychromatic Micro/Nano Beams

Gene Ice
Oak Ridge National Laboratory

Polychromatic microdiffraction -with differential aperture microscopy- offers a nondestructive probe of local texture that can determine subgrain orientations with submicron resolution in three dimensions. In addition, precision measurements can determine the local elastic distortions of the unit cell and can be used to determine the hydrostatic strain with an additional measurement. These new capabilities offers promise for unraveling the key mechanisms that drive mesoscale evolution of materials. To reach the full potential of the method, new hardware and software are now under development to accelerate the data acquisition and analysis. The application of polychromatic methods to a wide range of long-standing materials problems will be discussed. In addition, applications to specific high-performance materials systems will be presented.

Workshop 8
Inelastic X-ray Scattering

Organizers: Esen Ercan Alp, Advanced Photon Source, Argonne National Laboratory
John Hill, Brookhaven National Laboratory
Steve Heald, PNC-CAT, Advanced Photon Source, Argonne National Laboratory
Guoyin Shen, HP-CAT, Advanced Photon Source, Argonne National Laboratory

Thursday, May 4
Bldg. 402, Room E1100/E1200

8:30 am – 12:20 pm
1:15 pm – 5:00 pm

This workshop will focus on various inelastic x-ray scattering (IXS) techniques currently practiced at the APS. There are now half a dozen beamlines with dedicated or semi-dedicated inelastic x-ray spectrometers, and our goal is to bring the science being done at these beamlines, as well as their new capabilities like the dedicated Sector 30 IXS-CDT beamline to the attention of the APS Users. Also, the workshops participants will have a chance to visit these beamlines and learn their capabilities and meet with the responsible scientists.

8:50 – 9:00 Welcome

Session 1 – Chair: Wolfgang Sturhahn, Argonne National Laboratory

9:00 – 9:30 Opportunities for Inelastic X-ray Scattering in Condensed Matter Research
George A. Sawatzky, University of British Columbia

9:30 – 9:55 The New IXS Beamline at Sector 30
Harald Sinn, Argonne National Laboratory

9:55 – 10:20 Low-Energy Charge Dynamics Studied by Inelastic X-ray Scattering: From Simple to Strongly Correlated Materials
Yong Q. Cai, National Synchrotron Radiation Research Center, Taiwan, and SPring-8

10:20 – 10:35 Break, Atrium and Gallery

Session 2 – Chair: John Hill, Brookhaven National Laboratory

10:35 – 11:05 Probing Local Excitations with Angular Dependence of Large-q Nonresonant IXS: Sensitivity to Weak Electronic Symmetry Breaking in NiO and CoO
Wei Ku, Brookhaven National Laboratory

11:05 – 11:30 Resonant Inelastic X-ray Scattering Investigation of Cuprates
Young-June Kim, University of Toronto, Canada

11:30 – 11:55 Polarization Dependence of L- and M-Edge Resonant Inelastic X-ray Scattering in Transition-Metal Compounds
Michel van Veenendaal, Northern Illinois University and Argonne National Laboratory

11:55 – 12:20 Novel Spectrometer for High-Resolution Inelastic X-ray Scattering
Simo Huotari, ESRF, France

12:20 – 13:15 Lunch, Bldg. 402, Lower Level, Tent

Session 3 – Chair: Steve Heald, Pacific Northwest Laboratories

13:15 – 13:45 LERIX: A Multielement Spectrometer for Efficient Measurement of the Momentum Transfer Dependence of Inelastic X-ray Scattering
Gerald T. Seidler, University of Washington

13:45 – 14:10 Dynamics of Atoms and Molecules in Clathrate Hydrates
Dennis D. Klug, National Research Council of Canada

14:10 – 14:35 Nuclear Resonance Vibrational Spectroscopy:
A Quantitative Picture of Iron Dynamics in Heme Proteins and Model Compounds
Bogdan M. Leu, Northeastern University

14:35 – 15:00 Phonon Confinement in Nanoscaled Metallic Multilayers and Low-Dimensional Vibrational Dynamics on Size-Selected ^{57}Fe Nanoparticles
Beatriz Roldan-Cuenya, University of Central Florida

15:00 – 15:20 Break, Atrium and Gallery

Session 4 – Chair: Guoyin Shen, The University of Chicago

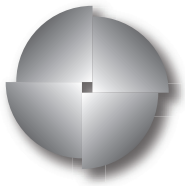
15:20 – 15:50 Phonon Dispersions in Metals at High Pressure Investigated by Inelastic X-ray Scattering
Daniel Antonangeli, Lawrence Livermore National Laboratory

15:20 – 16:05 Pressure-Induced Electronic Mott Transition in MnO
Choong-Shik Yoo, Lawrence Livermore National Laboratory

16:05 – 16:30 High-Frequency Dynamics in Molten Titanium
Ayman Said, Argonne National Laboratory

16:30 – 16:55 High-Resolution IXS Study of the Metal-Insulator Transition in VO_2
Sung C. Chang, Ames Laboratory

17:00 – 18:00 Beamline visits to Sectors 3, 9, 13, 16, 20, and 30



WK8

Opportunities for Inelastic X-ray Scattering in Condensed Matter Research

George A. Sawatzky
University of British Columbia

I will present some examples of research opportunities in inelastic x ray scattering. I will concentrate on strongly correlated electron systems, including transition metal compounds as well as organic conductors. I will discuss mainly elementary excitations in solids, such as the *d-d* and charge transfer excitons (including also the orbitons, which basically are *d-d* transitions) in transition metal compounds, as well as the Frenkel excitons in conducting polymers and oligomers of interest in molecular electronics. The importance here is in determining the dispersion of these excitations, which are related to the superexchange interaction in transition-metal compounds. In the molecular systems, the exciton dispersion can be quite large even for Frenkel excitons because of the large overlap of wave functions between neighboring monomers in the polymer. Information about the dispersion is extremely important in determining the magnitude of both on site and nearest neighbour coulomb interactions.

WK8

The New IXS Beamline at Sector 30

Harald Sinn
Argonne National Laboratory

Two new inelastic spectrometers at the the Advanced Photon Source, MERIX and HERIX, will start a general user program in 2007. MERIX will allow studies on weakly and strongly correlated electron systems, with an energy resolution of 100 meV, in the energy range of 5–12 keV. A 2-m-long arm spectrometer will have the capability to scan in the vertical as well as horizontal plane. Phonons and collective ionic excitations can be investigated with an energy resolution of about 1 meV with the HERIX spectrometer, a 9-m-long instrument that will have 9 analyzers located at different q-points. Both MERIX and HERIX spectrometers will offer state-of-the art, dedicated microfocusing capabilities. The expected performance of the spectrometers in relation to existing capabilities and the current status of the commissioning activities will be presented. User operation modes will be discussed.

WK8

Low-Energy Charge Dynamics Studied by Inelastic X-ray Scattering: From Simple to Strongly Correlated Materials

Yong Q. Cai
National Synchrotron Radiation Research Center, Taiwan

Inelastic x-ray scattering (IXS) is a powerful experimental technique that provides energy- and momentum-resolved information on lattice and charge dynamics of a variety of condensed matters, and offers in particular unique strengths in the study of the low-energy charge excitations of correlated electron systems and the electronic properties of materials under extreme thermal dynamical conditions such as high pressure and extreme temperature. The technique has been widely implemented in third-generation synchrotron radiation sources around the world that produce the tunable and highly intense and bright hard x-rays necessary for the practical application of the technique. The Taiwan Inelastic X-ray Scattering Beamline (BL12XU) at SPring-8 is designed for both resonant and nonresonant experiments on electronic excitations with variable energy resolutions from 10 to 1000 meV and energy ranges covering the K (L) absorption edges of most $3d$ ($4f$) metals. In this presentation, we will focus on the low-energy charge dynamics of a few archetypal materials, from collective excitations in simple systems to dd excitations in Mott insulators, recently studied on the beamline and discuss some of the interesting physics that can be unraveled using the technique.

WK8

Probing Local Excitations with Angular Dependence of Large-q Nonresonant IXS: Sensitivity to Weak Electronic Symmetry Breaking in NiO and CoO

Wei Ku
Brookhaven National Laboratory

A nonconventional approach of probing local excitations is introduced, by analyzing the strong angular dependence of the large-q nonresonant IXS inside the Mott gap of NiO and CoO. A "new selection rule" is shown to derive from the point group symmetry of the energy-resolved local Wannier states that form the particle-hole pair of the excitation. This new approach turns out to be very sensitive to the weak electronic symmetry breaking induced by magnetic order of the system and is expected to provide further insights in many other strongly correlated systems (e.g., direction of pseudo-spin of manganites).

WK8

Resonant Inelastic X-ray Scattering Investigation of Cuprates

Young-June Kim
Physics Department, University of Toronto

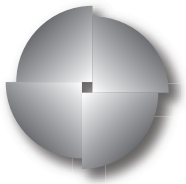
Extremely bright photons generated at the new generation of synchrotron light sources have made a huge impact on various scientific disciplines ranging from biology to materials science. One of the exciting new developments is the use of x-rays in the field of solid-state spectroscopy. Inelastic x-ray scattering, analogous to the well-known inelastic neutron scattering, is a powerful tool for studying momentum-dependent electronic excitations and phonons. In particular, resonant inelastic x-ray scattering in the hard x-ray regime has been widely used to study the momentum dependence of various electronic excitations in strongly correlated electron systems. For example, by tuning the incident photon energy to the Cu K-edge, one can gain a large intensity enhancement as well as element specific knowledge of the electronic excitations in various copper oxide compounds. In particular, recent improvements in instrumentation have allowed us to observe a new mode in the mid-infrared frequency region. We will discuss the momentum dependence of these excitations in prototypical cuprate superconductors, $La_{2-x}Sr_xCuO_4$, and also examine the evolution of such excitations as charge carriers are doped into the system.

WK8

Polarization Dependence of L- and M-Edge Resonant Inelastic X-ray Scattering in Transition-Metal Compounds

Michel van Veenendaal
Department of Physics, Northern Illinois University, and Advanced Photon Source, Argonne National Laboratory

The resonant inelastic x-ray scattering (RIXS) cross section at the L- and M-edges of transition-metal compounds is studied using an effective scattering operator. The intensities of the elastic peak and for spin-flip processes are derived. It is shown how the polarization dependence can be used to select transitions. The differences between RIXS in the soft (L- and M-edges) and hard (K-edge) x-ray regions are discussed. Theoretical calculations are compared with experiments. In particular, a detailed analysis of the polarization and angular dependence of L- and M-edge RIXS for divalent copper compounds, such as the high-T_c superconductors, is given.



WK8

Novel Spectrometer for High-Resolution Inelastic X-ray Scattering

S. Huotari, Gy. Vanko, R. Verbeni, F. Albergamo, and G. Monaco
ESRF, France

It has been generally accepted that diced analyzer crystals are not usable in resonant inelastic x-ray scattering (RIXS) applications, because the finite size of the individual cubes causes a large contribution to the resolution function. This effect could only be diminished by choosing Bragg angles very close to 90°, which is not generally possible in RIXS except perhaps for the sole case of Ge(733) reflection at the Cu K-edge. It is shown here that the cube-size contribution is not inevitable but can easily be overcome with a position-sensitive detector to record not only the intensity but also the position of the photons within the analyzer focus. RIXS spectrometers operating at the K-edges of 3*d* metals can thus have a resolution below 100 meV in a 1-m Rowland geometry. Examples of data recorded using a diced Si(553) crystal at the Cu K-edge (Bragg angle 77.5°) will be shown. The spectrometer resolution was 80 meV and when convoluted with the incident bandwidth, a total resolution of 110 meV was achieved.

WK8

LERIX: A Multielement Spectrometer for Efficient Measurement of the Momentum Transfer Dependence of Inelastic X-ray Scattering

Gerald T. Seidler
University of Washington

Non-resonant x-ray Raman scattering (XRS) is the inelastic scattering of hard x-rays from the K shell of low-Z elements or the less tightly bound shells of heavier elements. In the limit of low momentum transfer, q, XRS is determined by the same transition matrix element as is measured by x-ray absorption spectroscopies (XAS). However, XRS at higher q can often access higher-order multipole transitions, which help separate the symmetry of various contributions to the local density of states. The main drawback of XRS is its low cross section—a problem that is compounded for a q-dependent study. To address this issue, we have constructed a multielement spectrometer to simultaneously measure XRS at ten different values of q. By means of example, we report new measurements of the XRS from multiple edges in numerous borate minerals, from the L- and K-edges of Mg, from the Li K-edge in LiC_6, from the carbon K-edge of bucky onions and other nanoforms of carbon, and from the oxygen K-edge of ice. We will also discuss the forthcoming upgrade of the instrument to include 19 independent measurement channels, the availability of the instrument to general users of the APS at sector 20 PNC-XOR, and the possibility that the instrument can frequently be moved to other sectors to enable (for example) measurements in diamond anvil cells. Our instrument provides an important extension to the APS's HERIX and MERIX spectrometers (high and medium energy resolution inelastic x-ray scattering), which each have too high an energy resolution to be optimized for XRS measurements. For purposes of conformity, we have thus named our instrument LERIX (lower energy resolution inelastic x-ray scattering).

WK8

Dynamics of Atoms and Molecules in Clathrate Hydrates

Dennis D. Klug
Institute for Molecular Sciences, National Research Council of Canada

Clathrate hydrates are inclusion compounds consisting of atoms or molecules located in cages formed by hydrogen-bonded water molecules. The thermal conductivity of clathrate hydrates is similar to that of amorphous or glassy solids, although the clathrate hydrates are crystalline materials. To provide an understanding of this, the molecular and atomic vibrations in clathrate hydrates have been the subject of many recent

studies. In this talk, I will describe our research on clathrates using both theory and experiment, with an emphasis on recent work that combined nuclear resonant inelastic scattering, performed at the Advanced Photon Source, with incoherent inelastic neutron scattering and molecular dynamics simulations. The characterization of the dynamics of guest atoms and the cage molecules in clathrates resulting from this work may have wide implications for understanding the unusual thermal properties of clathrate hydrates.

WK8

Nuclear Resonance Vibrational Spectroscopy: A Quantitative Picture of Iron Dynamics in Heme Proteins and Model Compounds

Bogdan M. Leu
Department of Physics and Center for Interdisciplinary Research on Complex Systems, Northeastern University

We use synchrotron radiation to probe the vibrational dynamics of the ^{57}Fe atom in NO- and CO-ligated heme proteins and porphyrins designed to mimic the active site of heme proteins. This technique—nuclear resonance vibrational spectroscopy (NRVS)—is a highly selective and uniquely quantitative tool that yields the frequencies and amplitudes of all Fe vibrations. Measurements on oriented crystals reveal the direction of Fe motion and directly identify in-plane and out-of-plane modes.

NRVS data confirm vibrational predictions based on density functional theory (DFT) calculations for Fe(TPP)(NO) and Fe(TPP)(1-MeIm)(CO). Together, the experimental and computational approaches characterize vibrations of Fe against its nearest neighbors, as well as reactive modes. Many of these modes are clearly identified for the first time due to the absence of the selection rules and interfering vibrational signals that plague less selective techniques.

In collaboration with Marek Z. Zgierski[1], Nathan J. Silvernail[2], Graeme R. A. Wyllie[2], Mary K. Ellison[2], W. Robert Scheidt[2], Jiyong Zhao[3], Wolfgang Sturhahn[3], E. Ercan Alp[3], and J. Timothy Sage[4] at (1) Steacie Institute for Molecular Science, National Research Council of Canada, (2) Department of Chemistry and Biochemistry, University of Notre Dame, (3) Advanced Photon Source, Argonne National Laboratory, and (4) Department of Physics and Center for Interdisciplinary Research on Complex Systems, Northeastern University.

WK8

Phonon Confinement in Nanoscaled Metallic Multilayers and Low-Dimensional Vibrational Dynamics on Size-Selected ^{57}Fe Nanoparticles

Beatriz Roldan-Cuenya
Dept. of Physics, University of Central Florida

Investigations of vibrational dynamics in nanostructures are technologically relevant since they are basic for the understanding of materials properties such as heat capacity, thermal expansion, thermal conductivity, vibrational entropy, and phonon-assisted surface chemical reactions that might be an important issue in understanding the catalytic properties of metal nanoparticles. The use of nuclear resonant inelastic x-ray scattering (NRIXS) to study size-dependent changes in the Fe- and Sn-projected phonon density of states [DOS, g(E)], of nanoscaled metallic $[^{57}Fe/X]_n$ multilayers (X = Ag, Cr, Pt, Cu, Ru, Pd, etc.), semiconducting $[^{119}Sn/Si]_n$ superlattices, and ^{57}Fe nanoparticles will be reported. The multilayers have been grown by molecular beam epitaxy under ultrahigh vacuum, while the nanoparticles were synthesized by inverse micelle encapsulation using PS-P2VP diblock copolymers. While confinement of optical phonons in nanoscaled semiconductors is well known, work on metallic systems is scarce. Phonon confinement in superlattices arises from the limited film thickness, combined with energy mismatch of g(E) of the two materials. This work will demonstrate the systematic suppression of longitudinal acoustic modes within nanoscaled ^{57}Fe

films confined by metals (e.g., Ag, Pd,…) for which g(E) = 0 in the same energy region. Dramatic differences between the DOS of bulk bcc-Fe and that of size-selected ^{57}Fe nanoparticles have been found. In particular, an enhancement of the number of vibrational modes observed at low energies will be displayed. Such features have been previously attributed to new degrees of freedom existent in the nanocrystalline solids. Their physical nature is still controversial because linear, nonlinear, and Debye-like behavior of the phonon DOS [g(E) vs. E^n] at low energies have been previously reported.

In collaboration with Ahmed Naitabdi and Luis Ono, Department of Physics, University of Central Florida; ***R. Peters,*** *E. Schuster, and W. Keune, Fachbereich Physik, Universität Duisburg-Essen; and W. Sturhahn, J. Zhao, T. Toellner, and E.E. Alp, Advanced Photon Source, Argonne National Laboratory.*

WK8

Phonon Dispersions in Metals at High Pressure Investigated by Inelastic X-ray Scattering

Daniel Antonangeli
Lawrence Livermore National Laboratory

Traditionally, experimental determination of lattice dynamics is the domain of inelastic neutron scattering, but the restrictions on sample size imposed by the technique relegated the achievable information to low or at most moderate pressures (~10 GPa). Characterizing the effect of pressure on the propagation of elastic waves is singularly important for understanding elasticity, mechanical stability of solids, material strength, inter-atomic interactions, phase transition mechanisms, and to constrain models of the Earth and planetary interior. The elastic properties and the sound wave anisotropy of hcp metals at high pressure have been experimentally investigated by inelastic x-ray scattering (IXS). This technique allows the collection of the phonon dispersion curve and is particularly well suited for extreme conditions. The case of hcp iron, the main constituent of the Earth's inner core, will be addressed, followed by the case of hcp cobalt. The latter was chosen as an analogue to iron because it has similar mechanical and thermal properties but with the advantage of being available as a single crystal, so the complete elastic tensor has been determined and a comparative study of the single-crystalline and aggregate elastic properties has been performed. The case of molybdenum will be also discussed, as an example of a combined experimental and theoretical study of lattice dynamics at high pressure. In this regard, compression provides a very convenient way to probe the inter-atomic potential, allowing the study of the nature of anharmonic contributions, as well as electron-phonon interactions.

WK8

Pressure-Induced Electronic Mott Transition in MnO

Choong-Shik Yoo
Lawrence Livermore National Laboratory

Mott's seminal work on how insulating character may arise out of electron-electron repulsion used a 3*d* transition-metal monoxide, NiO, as an example and suggested its pressure-induced metallization, the Mott transition [1]. Yet for 55 years the Mott transition in these archetypal Mott insulators (MnO, FeO, CoO, and NiO) has eluded detection at room temperature because of the high pressures required. Recent resistivity measurements using "designer" diamond anvils, however, have recorded this insulator-metal transition in MnO, a decrease in resistance of five orders of magnitude between 90 and 106 GPa [2]. We present x-ray emission spectroscopy and x-ray diffraction data [3] which suggest that the Mott transition in MnO is a far richer phenomenon than just the onset of metallization. The data further suggest that the Mott transition

exhibits both profound similarities to transitions in the lanthanides and actinides as has been predicted [4] as well as subtle differences that challenge theory to gain general understanding of electron-correlation driven phase transitions.

[1] M. F. Mott, *Proc. Phys. Soc. A* **62**, 416 (1949), *Metal Insulator Transitions* (Taylor and Francis, London, 1990).

[2] J. R. Patterson et al., *Phys. Rev. B* **69**, 220101 (2004).

[3] C. S. Yoo et al., *Phys. Rev. Lett.* **94**, 115502 (2005).

[4] B. Johansson, *Phys. Rev. B* **15**, 5890 (1977).

This work has been supported by the LDRD-04-ERD-020 and PDRP programs at the LLNL, University of California, under the auspices of the U.S. DOE under Contract No. W-7405-ENG-48.

WK8

High-Frequency Dynamics in Molten Titanium

Ayman Said
Argonne National Laboratory

We have used a conical levitation setup to melt liquid titanium and study the collective atomic excitations using high-resolution inelastic x-ray scattering (IXS). The experiments took place at the IXS beamline at sector 3. The IXS data show well-defined sound excitations with a long lifetime comparable to the dynamics observed in liquid alkaline metals. Within the framework of generalized hydrodynamics, the structural relaxation can be expressed in terms of a generalized viscosity. This was approximated by a simple version of mode-coupling theory, yielding the damping functions of density fluctuations. The inputs for the calculations are the number density and static structure factor, and no adjustable parameters are used. A remarkable agreement between the experimental and theoretical results is demonstrated.

WK8

High-Resolution IXS Study of the Metal-Insulator Transition in VO_2

Sung C. Chang
Ames Laboratory

The nature of the metal-insulator transition (MIT) in VO_2 at T_C = 340 K has been a matter of debate for more than 50 years. The main point of controversy is the relative importance of the electron-lattice and electron-electron interactions as driving mechanisms for the MIT. In other words, is the transition a Peierls or Mott type? One major stumbling block to resolving this issue is that the ability to study the lattice dynamics in VO_2 using inelastic neutron scattering is severely hampered by the predominantly incoherent scattering of neutrons by vanadium. Recent advances in high-resolution inelastic x-ray scattering techniques provide an alternative path to studying lattice dynamics. Here we report on phonon dispersion measurements of VO_2 using the 3-ID-C beamline of the Advanced Photon Source. The effects of the MIT on the phonon dispersion are dramatic and reflect changes in both crystallographic and electronic structures which occur at the MIT. We will show that the transition is not a Peierls-type transition in the strict sense. However, electron-lattice interactions are likely not insignificant. A possible role played by them in the MIT will be discussed.

In collaboration with Y. Janssen, P. C. Canfield, J. W. Kim, B. Sieve, A. I. Goldman, and R. J. McQueeney, Iowa State University, and H. Sinn and A. Alatas, Argonne National Laboratory.

Workshop 9

X-ray Spectromicroscopy: A Tool for Environmental Science?

Organizers: Jürgen Thieme, Institute for X-ray Physics, University of Göttingen, Göttingen, Germany
Ian McNulty, Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois, USA
David Paterson, Australian Synchrotron, Melbourne, Australia
Stefan Vogt, Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois, USA

Thursday, May 4
Bldg. 402, Lecture Hall

8:30 am – 12:10 pm
1:30 pm – 6:00 pm

Environmental science is an extremely diverse field impacting many facets of science, technology, industry, and everyday life. Because of the complexity of environmental processes and the many mutual interactions involved, competence is required in more than one area of expertise and a multidisciplinary approach is often necessary to understand them. Experts in geochemistry, hydrology, microbiology, atmospheric, and soil sciences, to name a few, are essential in this regard. For example, the complex roles of carbon, nitrogen, phosphorus, and sulfur cycles in the environment are incompletely understood, not to mention their mutual interactions. Similarly, our knowledge of uptake and metabolism of trace metals by biological organisms, and their influence on the environment, is far from complete. Elemental transport, redox processes, microbial activity, and anthropogenic influences all affect these processes. Knowledge of each individual piece of the puzzle contributes to a better understanding of the overall picture. X-ray spectromicroscopy is a powerful tool for addressing key questions in the environmental sciences because of its high spectral and spatial resolution. This combination of high-resolution microscopy with spectroscopic capabilities allows determination of elemental composition as well as chemical speciation and identification of trace elements on length scales extending to the nanoscale. X-ray spectromicroscopy has already been applied to a very diverse range of problems in a multitude of scientific areas. It is now timely to define the most important problems and research directions in environmental science for which the latest x-ray spectromicroscopy methods would be indispensable and have the greatest impact.

The aim of this workshop is to bring together experts in all fields of the environmental sciences to formulate and discuss these key issues. A secondary aim is to improve accessibility to these methods to researchers in these fields. The workshop scope is limited to x-ray energies ranging from the K-absorption edge of carbon around 0.28 keV to that of iron around 7.2 keV and to spatial dimensions on the scale of a few microns extending to the nanoscale. This energy range covers many environmentally important elements and the working ranges of existing and planned x-ray microscopes with spectroscopic capabilities. This size range is where the behavior of particles such as colloids are determined not by their bulk properties but by their surfaces, and where the interface between local chemistry and macroscopic processes often occurs. The expected impact of this workshop is, however, much broader: a clear definition of outstanding problems in environmental science could serve as a roadmap for research activities worldwide.

08:30 – 08:45 Welcoming address

08:45 – 09:00 Introductory remarks
Juergen Thieme, Institute for X-ray Physics, University of Goettingen, Germany

09:00 – 09:30 Probing Structures and Processes in Soil Chemistry using X-ray Spectromicroscopy
Dean Hesterberg, Dept. of of Soil Science, North Carolina State University

09:30 – 10:00 Molecules, Cells, and Organisms: Environmental Spectromicroscopy of Biological Systems
Ingrid Pickering, Dept. of Geological Sciences, University of Saskatchewan, Canada

10:00 – 10:20 Formation of Minerals Inside and Near Single Bacterial Cells: Elemental Content and Valence State at the Sub-Micron Scale
Maxim Boyanov, Biosciences Division, Argonne National Laboratory

10:20 – 10:50 Break, Atrium and Gallery

10:50 – 11:20 X-ray Spectromicroscopy: A Tool for Advanced Understanding of Aqueous Colloidal Systems
Laurent Michot, Laboratoire Environnement et Minéralurgie, Nancy, France

11:20 – 11:50 Scanning Transmission X-ray Microscopy of Actinide Materials
David Shuh, Chemical Sciences Division, Lawrence Berkeley National Laboratory

11:50 – 12:10 Spectromicroscopy Analysis of Spore-Forming Bacteria
Bjorg Larson, Dept. of Physics & CEMS, SUNY Stony Brook

12:10 – 13:30 Lunch, Bldg. 402, Lower Level, Tent

13:30 – 14:00 Anaerobic Geomicrobial Effects on the Fate and Transport of Heavy Metals and Radionuclides
John Coates, Dept. of Plant and Microbial Biology, University of California, Berkeley

14:00 – 14:30 Metal Cycles
Satish Myneni, Dept. of Geosciences, Princeton University (tentative)

14:30 – 14:50 Ocean Phosphorus Cycling: New Insights from X-ray Spectromicroscopy
Ellery Ingall, School of Earth and Atmospheric Sciences, Georgia Institute of Technology, Atlanta

14:50 – 15:20 Break, Atrium and Gallery

15:20 – 15:50 Spectromicroscopy in the 1–4 keV Region: New Opportunities for Environmental Science
David Paterson, Australian Synchrotron, Clayton, Australia

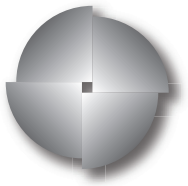
15:50 – 16:20 Colloidal Structures from the Environment Explored by X-ray Spectromicroscopy
Juergen Thieme, Institute for X-ray Physics, University of Goettingen, Germany

16:20 – 16:40 Break

16:40 – 17:00 Imaging Trace Metals in Biological Systems with Hard X-Ray Fluorescence Microscopy
Stefan Vogt, X-ray Science Division, Argonne National Laboratory

17:00 – 17:20 Spectral Deconvolution of Full-Spectral SXRF Imaging Data Aimed at Real-Time Quantitative Imaging Using the X-ray Fluorescence Microprobe
Chris Ryan, CSIRO Exploration and Mining, Clayton, Australia

17:20 – open Open Discussion
Ian McNulty, Advanced Photon Source, Argonne National Laboratory



WK9

Probing Structures and Processes in Soil Chemistry Using X-ray Spectromicroscopy

Dean Hesterberg
Department of Soil Science, North Carolina State University

Following a long history and a continuing need for soils as the primary medium supporting agriculture, soils are becoming increasingly recognized for their role in regulating the movement of chemicals between land, water, and air. One challenge of soil chemists in an era of urbanization is to increase the precision of controlling the solubility and movement of environmentally-hazardous chemical contaminants in soils, while maintaining adequate nutrition for plant growth. This task is particularly difficult because of the spatial and temporal heterogeneity of soils, which are open, living systems that undergo continuous change. Spatially-resolved x-ray techniques resolve fine-scale details of soil, and as such can transcend the larger-scale heterogeneity of the soil matrix. Such techniques have proved useful for characterizing both trace contaminants and their host matrix, yielding a greater degree of specificity to contaminant speciation analysis. In addition, these tools can potentially reveal the connection between micro-scale speciation and micro-scale chemical processes involving these species. One of the bigger challenges faced by scientists is to develop ways to utilize the micro-scale information for making quantitative, field-scale predictions of the short- and long-term impacts of chemical contaminants on the environment.

WK9

Molecules, Cells, and Organisms: Environmental Spectromicroscopy of Biological Systems

Ingrid J. Pickering
Department of Geological Sciences, University of Saskatchewan, Saskatoon, SK S7N 5E2, Canada

An important focus of environmental sciences is to understand how perturbations in the environment impact organisms, and ultimately humans. The use of synchrotron light gives unique opportunities for investigating living systems, from their molecular components, through intact cells, and even to whole complex organisms. Examples in this talk will focus on sulfur, an element which is ubiquitous and essential for life. Sulfur is intimately involved in many mechanisms used by organisms to counteract heavy metal toxicity, such as the phytochelatins produced by plants. It is also an element which is "spectroscopically silent" in that there is a dearth of analytical tools which can be used to investigate its chemical form *in situ*. The near-edge spectra of the sulfur K-edge show large chemical shifts and are valuable in identifying different chemical forms *in vivo*. The talk will demonstrate how this sensitivity can be used to image for the different chemical forms of sulfur in living systems, and will speculate on the pitfalls and benefits to environmental studies of a probe with resolutions down to the nanoscale.

WK9

Formation of Minerals Inside and Near Single Bacterial Cells: Elemental Content and Valence State at the Sub-Micron Scale

Maxim I. Boyanov[1], Barry Lai[2], Susan Glasauer[3], Matthew J. Marshall[4], S. Langley[5], Alice C. Dohnalkova[4], James K. Fredrickson[4], T. J Beveridge[3], and Kenneth M. Kemner[1]
[1] *Bioscienes Division, Argonne National Laboratory*
[2] *Advanced Photon Source, Argonne National Laboratory*
[3] *University of Guelph, Guelph, Ontario N1G 2W1, Canada*
[4] *Pacific Northwest National Laboratory, Richland, Washington 99354, USA*
[5] *University of Ottawa, Ottawa, ON K1N 6N5, Canada*

Using the current capabilities of the x-ray microscopy beamline at sector 2-ID-D at the Advanced Photon Source, the functioning of cells and their biogeochemical interactions can be studied on the 150-nm length scale. This spatial resolution is necessary because of the steep chemical gradients present near cells, resulting in unexpected metal transformations.

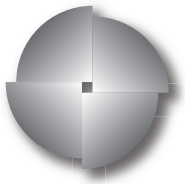
The growth of *Shewanella putrefaciens* CN32 on solid-phase Fe(III) results in the formation of abundant 40 nm sized intracellular granules. Elemental maps showed the presence of typical cellular elements (P, Ca, Cl, S), together with high Fe concentrations at the locations of high electron density on TEM micrographs. Selected area Fe K-edge XANES measurements showed that the internal Fe precipitates were more oxidized than Fe associated with other parts of the cell, but more reduced than magnetite. The valence state of extracellular precipitates in the vicinity of the cell was consistent with magnetite, a result also seen by bulk characterization techniques. In another study, TEM micrographs of *Shewanella oneidensis* MR-1 incubated in the presence of U(VI) showed the presence of uniform uraninite nanoparticles along thin (<100 nm in thickness) fiber-like structures. The x-ray elemental analysis revealed co-localization of the uraninite particles with Fe and P, pointing to the bacterial origin of these structures. Heme staining and antibody co-localizations corroborated the finding and demonstrated for the first time extracellular localization of two decaheme cytochromes in direct association with UO_2 nanoparticles.

WK9

X-ray Spectromicroscopy: A Tool for Advanced Understanding of Aqueous Colloidal Systems

Laurent J. Michot[1], Emmanuelle Montargès-Pelletier[1], Bruno S. Lartiges[1], Delphine Vantelon[2], and Jürgen Thieme[3]
[1] *Laboratoire Environnement et Minéralurgie, INPL-ENSG-CNRS UMR 7569, Avenue du Charmois, BP40, 54501 Vandoeuvre CEDEX, France*
[2] *SOLEIL Orsay, Swiss Light Source, Paul Scherrer Institut, CH-5232 Villigen, Switzerland*
[3] *Institut für Röntgenphysik, Universität Göttingen, Geiststrasse 11, 37073 Göttingen, Germany*

Because of their high reactivity, colloidal particles play a major role in the function of natural aquatic systems and water treatment installations. Numerous measurements indicate that suspended materials and sediments are the mains carrier of trace elements, and xenobiotic molecules, thus controlling the mobility of such contaminants In most cases, these materials are complex bio-organo-mineral assemblies whose structure and composition is highly variable and evolves upon changes in the physico-chemical conditions. A better understanding of these complex heterogeneous systems requires on one hand, advanced in situ characterization techniques of the size, structure, composition and speciation of the various components involved and on the other hand, the development of relevant model systems at the frontier between environmental sciences and soft condensed matter research. On the basis of literature data and of some of our own results on river systems, water treatment processes and model colloidal systems, we will try to illustrate the strong potential of x-ray spectromicroscopy in various energy ranges (water window, soft x-rays, intermediate x-rays) for obtaining new insight on colloid-mediated environmental processes.



WK9

Scanning Transmission X-ray Microscopy of Actinide Materials

D. K. Shuh[1], T. Tyliszczak[2], H. J. Nilsson[1,3], R. E. Wilson[1,2], P. Nico[1], and L. Werme[3,4]
[1] *Lawrence Berkeley National Laboratory, One Cyclotron Road, Berkeley, CA 94720 USA*
[2] *Advanced Light Source, Lawrence Berkeley National Laboratory, One Cyclotron Road, Berkeley, CA 94720 USA*
SKB, Stockholm, Sweden
Department of Physics, Uppsala University, Uppsala, Sweden

The scanning transmission x-ray microscope at the Advanced Light Source Molecular Environmental Science (ALS-MES) Beamline 11.0.2 has been utilized to investigate actinide materials. The ALS-MES scanning transmission x-ray microscope permits near-edge x-ray absorption fine structure (NEXAFS) spectroscopy and imaging of actinide particles with 30 nm resolution. The results from studies of U, Np, and Pu oxides will be presented, demonstrating the capabilities and limitations of soft x-ray STXM spectromicroscopy for investigations of actinide systems. The actinide 4d edges are employed for both imaging and for oxidation state determination. Of particular importance is the capability to directly probe the edges of light elements by NEXAFS, such as the oxygen K-edge, that are integral constitutents of many actinide materials. Actinide sample preparation methods, as well as sample radiation damage considerations, will be described.

WK9

Spectromicroscopy Analysis of Spore-Forming Bacteria

Bjorg Larson[1,2], Chris Jacobsen[1,2], Holger Fleckenstein[1], Jeff Gillow[2,3], and Mirna Lerotic[4]
[1] *Department of Physics and Astronomy and*
[2] *Center for Environmental Molecular Science, Stony Brook University, Stony Brook, NY 11794-3800, USA*
[3] *Department of Environmental Science, Brookhaven National Laboratory, Upton, NY*
[4] *Department of Computing, Imperial College London*

Soft x-ray spectromicroscopy provides the means for studying chemical speciation at the 30-50 nm resolution scale, and it is finding wide use in studies in biology, environmental science, astrobiology, polymer research, and other fields. For a specimen that can be characterized in terms of a set of known spectra, a variety of approaches [1,2] can be used for compositional mapping.

However, in more complex systems, as are found in biology and environmental science, the signature spectra are rarely known in advance. Cluster analysis provides a way to find the signature spectra that exist in a specimen and forms compositional maps based on these "discovered" spectra. Following preliminary work [3], we have carried out a systematic development [4] of this approach to soft x-ray spectromicroscopy analysis, and have extended it with methodologies aimed at classifying only based on compositional variations rather than specimen thickness [5].

Recent developments will be reviewed and illustrated with studies of soil bacteria. These bacteria are capable of changing soil chemistry by altering the oxidation-reduction potential of their environment which affects metal speciation. They produce organic metabolites and exudates, and can biodegrade organic contaminants. Changes in chemistry of subcellular features, such as spores, can be studied using these analysis techniques.

[1] X. Zhang et al., *J. Struc. Bio.* **116,** 335 (1996)

[2] C.J. Buckley in XRM 1999 proceedings, p. 33 (1999)

[3] C. Jacobsen et al., *Journal de Physique IV* **104**, 623 (2003).

[4] M. Lerotic et al., *Ultramicroscopy* **100**, 35 (2004).

[5] M. Lerotic et al., *J. Electron Spectr. Rel. Phenom.* (in press).

We gratefully acknowledge support from the NIH under contract R01 EB00479-01A1, and the National Science Foundation under grants CHE-0221934 and OCE-0221029.

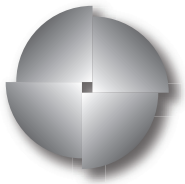
WK9

Anaerobic Geomicrobial Effects on the Fate and Transport of Heavy Metals and Radionuclides

John D. Coates[1], Karrie Weber[1], Juergen Thieme[2], Kenneth Kemner[3], and Michelle Scherer[4]
[1] *University of California, Berkeley*
[2] *Universitaet Goettingen*
[3] *Argonne National Laboratory*
[4] *University of Iowa*

Both the iron mineral and natural organic matter content of soils and sediments in the subsurface are important factors in controlling the fate and transport of heavy metals/radionuclides (HMR). This is because insoluble iron hydrous oxides rapidly adsorb and immobilize a broad range of soluble cations while, in contrast, soluble humic materials associated with the natural organic matter have hydrophilic characteristics and can alternatively competitvely bind and mobilize these adsorbed cations. As such, the net extent of transport of HMR in any environment is a function of the relative immobilizing effects of FeOOH and the re-mobilizing effects of humic substances. As a further complicating aspect microorganisms can significantly affect both of these processes through independent redox reactions especially in anoxic environments. Studies in our laboratory have demonstrated that under anaerobic conditions with nitrate as the electron acceptor, many microorganisms can oxidize both aqueous and mineral phase Fe(II) resulting in the rapid precipitation of Fe(III) minerals. These studies demonstrated that a broad range of Fe(II) phases including the structural Fe(II) in nesosilicate (almandine and staurolite) minerals were subject to direct nitrate-dependent microbial oxidation Mössbauer spectroscopy and X-Ray diffraction analysis indicated that this metabolism resulted in the formation of crystalline mixed Fe(II)/Fe(III) mineral phases including green rust, maghemite, and magnetite. In the case of green rust (GR), X-Ray fluorescence spectroscopy identified the association of chloride and phosphate with the mineral product. Anaerobic biological oxidation of the Fe(II) amended with soluble U(VI) or Co(III) resulted in the rapid removal of the metals from solution. Comparison of the energy-aligned and step-height-normalized XANES data from the uranium content of the biogenically-formed iron oxides with those from UO_2 (U(IV)) and UO_3 (U(VI)) standards indicated that the uranium was present only in the oxidized U(VI) state while x-ray spectroscopy (XAFs) studies indicated that uranyl formed bidentate inner-sphere complexes with the biogenic iron (hydr)oxide surfaces. More cobalt (81% of the initial 100 μM) than uranium was removed from solution as a result of biological Fe(II) oxidation. When a similar experiment was performed with cadmium 69% of the initial 100 μM was bound by the biogenically-formed iron minerals. Comparison of the atomic radii of these ions (R_U=1.75 Å, R_{Cd}=1.55 Å, R_{Co}=1.35 Å) to the atomic radius of Fe (1.4 Å) suggests that the amount of U, Cd, and Co removed from solution is inversely dependent on the similarity of their size to that of the Fe.

Similarly to the effects on iron, the microbial redox cycling of the quinone moieties of humic acids (HA) also resulted in significant morphological and geochemical alterations. X-ray microscopy analysis of microbially reduced and oxidized humic acids indicated that the conformational structure of the humic colloids was significantly altered as a direct result of the redox change. In the reduced state, the humic acids appeared as small dense particles, while on reoxidation, large loose aggregates were formed. Similarly the redox alteration had a significant effect on the geochemical characteristics of the HA. Surface tension measurements of an aqueous solution of the HA indicated that the reduced HA showed higher values at all HA concentrations tested which is indicative of a more hydrophilic and less hydrophobic solute. On re-oxidation, the surface tension values reverted back to values similar to those obtained for the untreated oxidized HA. These data indicate that the hydrophobicity of the HA is altered on biological reduction of the HA and that this alteration is reversible. In support of this the reduced HA demonstrated a 15% higher affinity for HRM such as trivalent cobalt than the oxidized HA. In addition to increasing the binding capacity of HA for heavy metals, the reduction of the HA also decreased the bioavailability and toxicity of bound heavy metals such as chromium. When incubated in the presence of Cr(III) and HA, cells of *Escherichia coli* grew much



more rapidly in the presence of the reduced HA suggesting that the higher metal binding capacity of the reduced humic substances resulted in a removal of the Cr(III) from solution and hence reduced its bioavailability and toxicity.

Overall, these studies demonstrate that the fate and transport of HRM in the environment is not only a function of the iron and natural organic matter content of soils and sediments but also the redox state of these components which is in turn regulated by the geomicrobiology of the environment.

WK9

Ocean Phosphorus Cycling: New Insights from X-ray Spectromicroscopy

Ellery Ingall[1], Jay A Brandes[2], David Patterson[3], and Martin de Jonge[4]
[1] *School of Earth and Atmospheric Sciences, Georgia Institute of Technology, Atlanta, GA, USA*
[2] *Skidaway Institute of Oceanography, Savannah, GA, USA*
[3] *Australian Synchrotron, Clayton, VIC, Australia*
[4] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA*

Phosphorus plays a critical role in regulating terrestrial primary productivity, and exists in a wide variety of organic, inorganic and mineral forms in the environment. The response of P cycling to shifting redox conditions has been a subject of many studies, yet the controlling mechanisms remain elusive. We investigated the effect of redox conditions on sedimentary phosphorus (P) composition at sites overlain by oxic and anoxic bottom waters in Effingham Inlet, a fjord located on the west coast of Vancouver Island. Scanning tandem fluorescence and transmission x-ray microscopy, together with phosphorus near edge x-ray fluorescence spectroscopy (P-NEXFS), were used to examine P composition in these samples on sub-micron scales Examination of sediments using x-ray techniques revealed a heterogeneous P distribution characterized by several micron-diameter P-rich regions associated with polyphosphates as well as regions associated with different inorganic calcium phosphate and magnesium aluminum phosphate containing mineral phases. Such P-rich regions comprised roughly 50% of the total P within the sediments. Mineral phosphates exhibited a considerable range of P-NEXF spectra, from poorly-structured phases resembling brushite to spectra closely matching crystalline hydroxyapatite. Intriguingly, no enriched region appeared to be associated with iron phosphates, and there was no evidence for a physical association between polyphosphate and mineral P regions. Although polyphosphate regions were common in surface oxic sediments, they were rare in surface anoxic sediments and absent in deeper (~20 cm) sediments in both locations. The results support recently published papers suggesting that redox-sensitive formation and remineralization of polyphosphates plays a central role in regulating the formation of phosphorites (apatite) in marine sediments. Although apatite minerals do not appear to form in close proximity to polyphosphates, the release of polyphosphate pools within anoxic sediments can provide a local increase in porewater dissolved P concentrations, leading to formation of, and sequestration within, mineral phases. Coupled cycling of P and iron is not associated with distinct iron phosphate minerals, rather it may be associated with iron coatings on particulates distributed throughout the sample matrix.

WK9

Spectromicroscopy in the 1–4 keV Region: New Opportunities for Environmental Science

David Paterson
Australian Synchrotron, 800 Blackburn Road, Clayton, VIC 3168, Australia

Unique opportunities with x-ray microscopy in an increasingly important energy region for biological and environmental sciences are available. Using brilliant, focused x-ray beams at the Advanced Photon Source, spectromicroscopy with 60 nm resolution can be achieved in the 1–4 keV region. Phosphorus and sulfur are key elements in biological and environmental studies. Chemical state analysis using near-edge spectroscopy

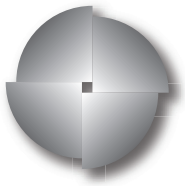
combined with high spatial resolution allows probing of new questions. Phosphorus occurs in dissolved and particulate marine organic materials and is a significant, but poorly understood, source of bioavailable phosphorus in many aquatic environments. Sulfur is an indispensable nutrient in soil for plants and microorganisms. The speciation of sulfur in soils is intimately linked with the chemical state of the soils, such as redox potential and acidity. Changes in the speciation of sulfur in soils can result in considerable changes of soil fertility. Knowledge of the chemical speciation of phosphorus and sulfur at the submicron level and *in situ* is critical and can be provided by x-ray spectromicroscopy.

WK9

Colloidal Structures from the Environment Explored by X-ray Spectromicroscopy

Jürgen Thieme
Institute for X-ray Physics, University of Göttingen, Friedrich-Hund-Platz 1, 37077 Göttingen, Germany

X-ray microscopy achieves a much higher resolution than light microscopy due to the much shorter wavelength of x-rays compared to visible light. The smallest structures that can be seen in an x-ray microscope at present are less than 20 nm in size. In addition, x-ray microscopy is capable of imaging specimens directly in aqueous media. Especially, x-radiation within the energy range of E = 250 eV to E = 540 eV, which equals the wavelength range of λ = 4.43 nm to λ = 2.34 nm resp., is very well suited for x-ray microscopy studies of hydrated specimens. By choosing the x-ray wavelength appropriately, it is possible to perform spectromicroscopy studies. Comprising, x-ray microscopy is a tool very well suited to study structures showing colloidal dimensions in the environment. Due to their surface activity these structures are principally involved in various chemical and physical processes. Substances can be bound and immobilized or transported, colloids can attach to microorganisms building up microhabitats, and organic substances as humic substances can flocculate due the interaction with metals. A great variety of these structures have been studied with x-ray microscopy using its high spatial resolution as well as its high spectral resolution. Dispersions extracted from soils and groundwater aquifers have been imaged to visualize first of all the appearance of the colloidal structures. The effect of changing chemical conditions in an aqueous environment has been studied extensively. The change in the appearance of colloidal structures has been imaged and evaluated. Using spectromicroscopy, the distribution of organic and inorganic soil colloids has been studied. The obtained spectra have been analyzed for major chemical components. A large fraction of these are humic substances. Spectra have been taken from humic substances with and without a coagulation agent. Different functional groups have been identified and changes have been mapped. Clay dispersions and microhabitats as well as morphological effects of biologically induced redox changes of humic substances have been imaged tomographically. Tilt series of images have been obtained with an x-ray microscope; the specimen was then reconstructed from these images. These reconstructions convey a detailed three-dimensional presentation of the specimen structure.



WK9

Imaging Trace Metals in Biological Systems with Hard X-Ray Fluorescence Microscopy

Stefan Vogt, Barry Lai, and Jörg Maser
X-ray Science Division, Argonne National Laboratory

Trace elements, in particular metals, are essential to most biological functions, as numerous proteins require the binding of metals such as Ca, Fe, Cu, or Zn, to modulate their structure, function, or activity. At the same time, the dys-regulation or uncontrolled uptake of these elements is suspected to play a role in many diseases. Additionally, environmental exposure to metals such as Cr, As, Cd, Hg, and Pb can lead to toxic and long term adverse health effects. The mechanisms of these toxicities are often not yet fully understood, but techniques such as x-ray fluorescence microscopy (XRF) are increasingly being used to address these questions.

XRF enables studies of inter- and intra-cellular distributions of elements from Al to Zn and above, with accurate quantification at very high sensitivity (e.g., ~3 attograms of Zn can be detected in 1s). The elemental content is measured directly by using the characteristic fluorescence of atoms excited by the microfocused x-ray beam, without the need for element-sensitive dyes. To determine metal speciation, micro-XANES (x-ray absortion near edge spectroscopy) can be employed. Currently, a spatial resolution of 200 nm can be achieved routinely in mapping aplications, and next-generation microprobes with a resolution limit of 30 nm are being planned, constructed and tested.

We will report on the capabilities of existing microprobes, approaches in sample preparation and data processing, in the context of biological specimens and environmental sciences, and present examples of scientific applications.

Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

WK9

Spectral Deconvolution of Full-Spectral SXRF Imaging Data Aimed at Real-Time Quantitative Imaging Using the X-ray Fluorescence Microprobe

C.G. Ryan[1,2], B.E. Etschmann[1], R. Hough[1], D.P. Siddons[3,4], S. Vogt[5], G. Moorhead[6], P. Dunn[6], A. Dragone[3],and G. de Geronimo[3]
[1] *CSIRO Exploration and Mining, Clayton VIC, Australia*
[2] *Physics Department, University of Melbourne, Parkville VIC, Australia*
[3] *Instrumentation Division, Brookhaven National Laboratory, Brookhaven NY, USA*
[4] *National Synchrotron Light Source, Brookhaven National Laboratory, Brookhaven NY, USA*
[5] *Advanced Photon Source, Argonne National Laboratory, Argonne IL, USA*
[6] *CSIRO Manufacturing and Infrastructure Technology, Preston VIC, Australia*

Energy dispersive spectra from synchrotron x-ray fluorescence (SXRF) analysis of minerals can display severe multi-element overlap that can hamper imaging approaches that rely on regions of interest. A method called dynamic analysis (DA), developed at the CSIRO, builds a matrix transform that is applied to full-spectral pixel data to unfold the contributions of overlapping elements, rapidly performing the effect of a linear least squares fit to each pixel spectrum [1]. Projection of companion variance images aid in tracking uncertainty contributions. Once constructed, the transform can be applied in real-time to image elemental distribution in other sample areas with similar elemental components.

Application of the technique to geological and environmental problems using full-spectra data collected at sectors 2 (XOR, 2-ID-E) and 20 (PNC-CAT, 20-ID-B) of the APS show the value of the technique to unfold complex overlapping sequences of elemental components to extract data on metal distribution (e.g. Au) in

the regolith and ore systems. User access to the technique is via the GeoPIXE software [2] installed at sectors 2, 13, and 20 of the APS.

The DA approach lends itself to real-time processing of fluorescence events. A detector development collaboration between the NSLS and Brookhaven Instrumentation Division and the CSIRO is working to combine BNL detector array technology with CSIRO machine vision processing approaches to achieve high throughput real-time processing of SXRF image data. The BNL detector comprises a large 384 element planar Si detector array and custom front-end chips for pulse-shaping, peak-detecting derandomization and time-over-threshold (ToT) measurement [3,4,5]. The CSIRO processor (HYMOD) uses a 150 MHz FPGA and co-processor and an embedded implementation of the DA algorithm for pile-up rejection (using ToT) and image projection. Tests of the processor have demonstrated throughput (deconvoluted SXRF and projected elemental images in ppm-flux units) at 10^8 events per second.

[1] C.G. Ryan, *International Journal of Imaging Systems and Technology* **11** (2000) 219.

[2] http://nmp.csiro.au/GeoPIXE.html

[3] D.P. Siddons, *et al*., Proc. of SRI-2003 Conference, *AIP Conference Proceedings* **705** (2004) 953.

[4] G. De Geronimo *et al*., *IEEE Trans. Nucl. Sci*. **50** (2003) 885

[5] A. Dragone *et al*., *IEEE Trans. Nucl. Sci., in press*.

This work was supported by the Australian Synchrotron Research Program, funded by the Commonwealth of Australia under the Major National Research Facilities Program. Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

Workshop 10

Diffuse Scattering: Emerging Opportunities with Advanced X-ray and Neutron Sources

Organizer: Gene Ice, Oak Ridge National Laboratory

Thursday, May 4
Bldg. 401, Room A5000

8:30 am – 12:00 pm

Diffuse scattering is a powerful probe of local correlations with sensitivity to defects, surfaces, dynamics, interfaces, and other structures with short-range periodicity. The development of intense x-ray sources and the development of more powerful neutron sources has created new opportunities with the potential to revolutionize our understanding of short-range correlations in materials. In this workshop we present prototype experiments that demonstrate the range of science that can now be addressed with diffuse scattering and that highlight major opportunities for materials discovery now possible. Talks will include a discussion of the ability to study dynamics of atomic motion (thermal-diffuse scattering) in-situ, in real time and in combinatorial studies. This work is only possible due to the development of efficient x-ray area detectors and intense x-ray beams. Another talk will highlight the role of modeling to extract key features of atomic dynamics and local structure in materials and will discuss new opportunities made possible by this approach. Combined diffuse/microbeam experiments will also be discussed. Here the ability to make intense x-ray micro/nanobeams that can measure weak diffuse scattering in very small samples offers enormous--and as yet largely untapped--potential for studying materials where large homogeneous samples are hard to make, where large samples may be dangerous, or where naturally occurring samples are polycrystalline by nature. Other talks will discuss surface and interface scattering and how diffuse scattering can be used to characterize structure with limited range in one dimension. It is the goal of this workshop, to both teach the range of new science now emerging and to develop an understanding of where better instrumentation and/or software can enable the user community to access diffuse scattering as an essential tool of materials discovery.

8:30 – 8:40	Welcome and Introduction *Gene Ice, Oak Ridge National Laboratory*
8:40 – 9:20	Diffuse Scattering and Models of Disorder: New Opportunities for Diffuse X-ray and Neutron Scattering *Richard Welberry, Australian National University*
9:20 – 10:00	Diffuse Scattering: Study of Bulk and Near-Surface Microstructure of Alloys *Bernd Schönfeld, ETH Zürich*
10:00 – 10:15	Break, Atrium and Gallery
10:15 – 10:50	Microbeam Measurements of Defect Distributions *Bennett Larson, Oak Ridge National Laboratory*
10:50 – 11:25	Studies of Short-Range Order and Atomic Displacements in a Null Matrix $^{62}Ni_{0.52}Pt_{0.48}$ Crystal *Jose Abelardo Rodriguez, University of Houston*
11:25 – 12:00	X-Ray Diffuse Scattering Studies of Phonons and Phase Transitions *Tai-Chang Chiang, University of Illinois at Urbana-Champaign*

WK10

Diffuse Scattering and Models of Disorder: New Opportunities for Diffuse X-ray and Neutron Scattering

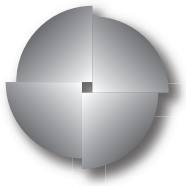
Richard Welberry
Australian National University

We will review some of our recent work involving diffuse scattering in two contrasting areas of research. The first involves diffuse scattering and disorder in flexible molecular systems and has been undertaken with a view to understanding the occurrence of polymorphism, particularly in pharmaceuticals. The second is aimed at trying to understand the complex nanoscale structure that occurs in the relaxor ferroelectrics PZN and PNM. In both cases, we use Monte Carlo simulation of large model crystals to interpret and analyse extensive three dimensional diffuse scattering data obtained with either x-rays at the APS or neutrons at the ISIS (UK) spallation neutron source.

Despite extensive attempts to make *ab initio* predictions of polymorphic structures, there has, to date, been little progress in this important field. Diffuse scattering analysis gives a detailed picture of local structure and dynamics, rather than the *average* structure yielded by conventional crystallography. This new information can give valuable insights into how and why some compounds form different polymorphs and others do not.

Although PZN, PMN, and numerous related materials have been studied extensively over a long period, a detailed understanding of the exact nature of their polar nanostructure has still not emerged. We show how the detailed information contained in full three-dimensional diffuse scattering patterns, together with a little chemical knowledge, can be used to construct a detailed picture of the nanoscale structure of these important materials.

Professor Richard Welberry, *Australian National University in Canberra, is a pioneer in the application of least-squares Monte Carlo methods for direct fitting of diffuse x-ray and neutron scattering. He has extensive experience with collecting and interpreting x-ray and neutron diffuse scattering and in the study of structures in materials with length scales on the order of 0.1–20 nanometers. These nanoscale structures are inherently inaccessible using conventional crystallography. His applications of diffuse scattering have included studies of functional material such as solid-state ionic conductors, relaxor ferroelectrics, guest/host inclusion compounds, and measurements aimed at understanding the importance of nanoscale structure to materials properties. His talk will review recent diffuse scattering applications based on the use of synchrotron and neutron beams.*



WK10

Diffuse Scattering: Study of Bulk and Near-Surface Microstructure of Alloys

Bernd Schönfeld
ETH Zürich, Switzerland

The local atomic arrangement of binary alloys has been repeatedly analysed from x-ray and neutron diffuse scattering. While local order at elevated temperature and the evaluation of effective pair interaction parameters for states of thermal equilibrium are the domain of neutron scattering, the strength of x-rays lies in the determination of the species-dependent atomic displacements. Here the tunabilty of the energy of synchrotron radiation has been elegantly exploited to improve data quality. Current partially controversial issues related to short-range order in bulk Ni-Au and Fe-Al will be presented and discussed. The near-surface microstructure requiring measurements at elevated temperature under UHV conditions is a newly evolving area. In investigations of the Pt-Rh (110) and (111) surfaces at the Swiss Light Source, it was found that the microstructure depends on the type of surface. Improvements in experimental set-ups to go beyond diffuse in-plane scattering will be discussed.

Professor Bernd Schönfeld, *Institut für Angewandte Physik, ETH Zürich, is a master researcher, respected for his careful sample preparation methods designed to achieve reproducible diffuse scattering measurements. His comparison of alternative data analysis methods have shed light on best practices for determining local correlations in materials. His recent work on local structure near surfaces is only possible because of intense synchrotron radiation.*

WK10

Microbeam Measurements of Defect Distributions

Bennett Larson
Oak Ridge National Laboratory, Oak Ridge, TN, USA

Submicron resolution x-ray beams have been used for depth-resolved diffuse scattering investigations of the size, concentration, type, and depth dependence of ion-irradiation induced defect clusters in silicon irradiated with 10-MeV Si ions at 300°C. Diffuse scattering measurements in the asymptotic (Stokes-Wilson) diffuse scattering region near Bragg reflections have been carried out in cross-section, as a function of depth in <100> oriented Si, using x-ray microbeams focused to 0.7 μm by Fresnel zone plate optics. The microbeam diffuse scattering measurements were analyzed in terms of the size distributions for vacancy and interstitial loops as a function of depth. The cross-section diffuse scattering technique will be compared to alternative measurements for studying ion-implantation-induced defects.

Dr. Bennett Larson, *Oak Ridge National Laboratory, is a long-time pioneer in the field of diffuse scattering from defects and is pioneering new science in the area of micro- and nanodiffraction. His work on ion-implanted materials demonstrates that spatial resolution combined with advanced single-crystal diffuse scattering methods can provide important new information about defects and their distributions in materials.*

WK10

Studies of Short-Range Order and Atomic Displacements in a Null Matrix $^{62}Ni_{0.52}Pt_{0.48}$ Crystal

Jose Abelardo Rodriguez
University of Houston, Houston, TX, USA

The best known exception to the Heine-Samson and Bieber-Gautier arguments for ordering effects in transition metal alloys (similar to the Hume-Rothery rules) is a NiPt alloy, where the phase diagram is similar to the CuAu system. Using the disk chopper spectrometer at NIST, we have investigated a null-matrix crystal, $^{62}Ni_{0.52}Pt_{0.48}$, (^{62}Ni has a negative scattering length, nearly equal in magnitude to Pt). Its composition has therefore been chosen so that all effects depending on the average lattice scattering vanish. The only remaining contributions to the diffuse scattering are the short-range order and size effect terms, as will be discussed. Such data permit the extraction of the short-range order parameters (concentration-concentration correlations) as well as the displacement parameters (concentration-displacement correlations). Using the Krivoglaz-Clapp-Moss theory, we obtained the effective pair interactions (EPI) between the several neighbors in the alloy. The results can be used to model the alloy in the context of electronic theory of alloy phase stability, including an evaluation of the potentially important aspect of charge transfer and ionicity.

Dr. Jose A. Rodriguez, *University of Houston, has been working with Prof. Simon C. Moss on diffuse scattering from disordered alloys. Dr. Rodriguez will discuss the application of diffuse x-ray and neutron scattering to understand subtle reciprocal space influences on local real-space structure and order in NiPt alloys using the null-matrix technique.*

WK10

X-Ray Diffuse Scattering Studies of Phonons and Phase Transitions

Tai-Chang Chiang
Department of Physics, University of Illinois at Urbana-Champaign, IL, USA

X-ray diffuse scattering is a powerful technique for probing lattice vibrations, or phonons, in solids. Measurements of the scattering intensity derived from thermally populated phonons yield information about phonon dispersion relations. The development of intense synchrotron radiation sources and efficient detectors has made it possible to make rapid measurements of small samples, thus opening up opportunities for detailed systematic studies of material systems and phase transitions. This talk will focus on some recent technical developments with examples drawn from measurements of elemental materials including Si, Nb, Pu, $TiSe_2$ (a charge density wave compound), and $SrTiO_3$ (a complex oxide). This talk will also address challenges important for further advancement of the technique, including calculation of high-order scattering corrections and development of simple direct methods for data reduction.

Dr. Tai-Chang Chiang, *University of Illinois at Urbana Champaign, has done seminal work on bulk, surface, and interface states of metals and semiconductors. He has pioneered rapid, in situ x-ray studies of TDS in single crystals. These methods offer hope for studies of atomic across phase transitions and during processing for combinatorial studies of differences in atomic motions due to differences in processing and/or alloy composition.*

Workshop 11

Beam Line Controls at the Advanced Photon Source

Organizers: Pete Jemian and John Maclean, Advanced Photon Source, Argonne National Laboratory

Thursday, May 4
Bldg. 401, Room A5000

1:30 – 5:30 pm

After ten years of operations and experience, a variety of beam line control systems are in use at the APS. The majority of beam lines base their controls on EPICS, while others have developed their own control protocols or adopted commercial control solutions. The aim of this workshop is to present the current state of beam line controls at the Advanced Photon Source as well as to outline future directions. Speakers will address the general topics indicated below.

1:30 – 1:35	Welcome and Introduction *Pete Jemian*
1:35 – 1:50	synApps: A software tool kit *Tim Mooney, Beamline Controls and Data Acquisition Group, Advanced Photon Source*
1:50 – 2:05	APS sector 8 conversion to EPICS & synApps *Joe Sullivan, Beamline Controls and Data Acquisition Group, Advanced Photon Source*
2:05 – 2:20	The EPICS brick *David Kline, Beamline Controls and Data Acquisition Group, Advanced Photon Source*
2:20 – 2:35	Support for spec at the APS *Xuesong Jiao, Beamline Controls and Data Acquisition Group, Advanced Photon Source*
2:35 – 3:00	Controls at ESRF *Vicente Rey-Bakaikoa, European Synchrotron Radiation Facility*
3:00 – 3:30	Break, Atrium and Gallery
3:30 – 3:45	The CCD image server—An EPICS-based data acquisition system for area detectors *Brian Tieman, Beamline Controls and Data Acquisition Group, Advanced Photon Source*
3:45 – 4:10	EPICS detector and feedback software *Mark Rivers, GSE-CARS, Advanced Photon Source*
4:10 – 4:35	From sample to structure: Automation at SER-CAT *Jim Fait, SER-CAT, Advanced Photon Source*
4:35 – 4:50	Beam line controls at SGX *Steve Wasserman, SGX-CAT, Advanced Photon Source*
4:50 – 5:10	X-ray software initiative *Ken Evans, Controls Group, Advanced Photon Source*
5:10 – 5:30	AccessGrid at SER-CAT (tentative) *John Rose, SER-CAT, Advanced Photon Source*
5:30	Adjourn

Workshop 12

Microdiffraction in Structural Biology

Organizers: Stephen C. Harrison, Harvard University
Steven E. Ealick, Cornell University

Friday, May 5
Bldg. 402, Lecture Hall

9:00 am – 11:45 am
1:30 pm – 4:15 pm

Microdiffraction is an increasingly important tool for the elucidation of structures of biological macromolecules. Microdiffraction is defined as the study of crystals with at least one dimension less than 20 μm. Microdiffraction places special demands on beamline optics and endstation instrumentation. First developed at European facilities such as the ESRF and SLS, demand for microdiffraction is growing in the United States. This one-day workshop on microdiffraction of biological macromolecules includes a survey of capabilities at the APS, descriptions of existing microdiffraction beamlines at the European Synchrotron Radiation Facility (ESRF) and Swiss Light Source, a description of the microdiffractometer and automounter developed at the ESRF, and plans for microdiffraction beamlines at the Advanced Photon Source.

9:00 – 9:30	Current and Planned Capabilities in Microdiffraction at the APS *Qun Shen, Advanced Photon Source, Argonne National Laboratory*
9:30 – 10:15	Microdiffraction Capabilities at the Swiss Light Source *Ehhmke Pohl, Swiss Light Source, Paul Scherrer Institute*
10:15 – 10:45	Break, Atrium and Gallery
10:45 – 11:15	Structures of Amyloidogenic Peptides Obtained from Microcrystals at ESRF ID13 Microfocus Beamline *Michael Sawaya, University of California, Los Angeles*
11:15 – 11:45	ID23-2: The New Dedicated MX Microfocus Beamline at the ESRF *David Flot, European Molecular Biology Laboratory*
11:45 – 1:30	Lunch, Bldg. 402, Lower Level, Tent
1:30 – 2:15	Quickly Getting the Best Data from your Macromolecular Crystals with a New Generation of Beamline Instruments *Florent Cipriani, European Molecular Biology Laboratory*
2:15 – 2:45	NE-CAT Plans for Microdiffraction *Kanagalaghatta Rajashankar Raj, NE-CAT, Advanced Photon Source; Cornell University*
2:45 – 3:15	Break, Atrium and Gallery
3:15 – 3:45	Diamond Beamline I24: An Instrument for Macromolecular Microcrystallography *Gwyndaf Evans, Diamond Light Source*
3:45 – 4:15	Plans for Microdiffraction at the Structural Biology Center *Gerd Rosenbaum, Structural Biology Center, Argonne National Laboratory; SER-CAT, University of Georgia*

WK12

Current and Planned Capabilities in Microdiffraction at the APS

Qun Shen
X-ray Microscopy and Imaging Group, Advanced Photon Source (APS), Argonne National Laboratory, Argonne, IL

Microdiffraction is a significant part of research activities at the Advanced Photon Source (APS) and at all third-generation synchrotron sources. These activities can be grouped into three categories—monochromatic microdiffraction, microbeam Laue, and microcrystallography. In the first category, a scanning microprobe is used in standard monochromatic x-ray diffraction experiments to study nonuniform and inhomogeneous structural properties in relatively large specimens such as semiconductor thin-films. In the second category, a microfocused polychromatic beam is used on polycrystalline specimens to investigate grain orientation, strain field, and grain boundary structures in alloys. In the third category, a monochromatic microbeam is used to study crystallographic structures of small micron-sized, isolated single-crystal specimens. This talk will provide an overview of current capabilities in all three areas of microdiffraction at the APS, with an outlook into the future.

The Advanced Photon Source 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.

WK12

Microdiffraction Capabilities at the Swiss Light Source

Ehmke Pohl, Claude Pradervand, Roman Schneider, Takashi Tomizaki, Armin Wagner, and Clemens Schulze-Briese
Swiss Light Source, Paul Scherrer Institute, CH-5232 Villigen, Switzerland.

The Swiss Light Source (SLS) is a 2.4-GeV, medium-energy synchrotron with a circumference of 288 m that generates hard x-rays using small-gap in-vacuum undulators. The macromolecular crystallography group at the SLS currently operates two undulator beamlines for protein crystallography.

Both beamlines share a similar optical design consisting of a sagitally focusing Si(111) double-crystal monochromator followed by a vertically focusing mirror. This setup allows an energy range of 6–20 keV. The first beamline (X06SA) is equipped with two separate endstations, the high-resolution diffractometer (HRD), equipped with a Mar225 mounted in an A-frame, and the EMBL/ESRF microdiffractometer (MD2) mounted on an optical table equipped with a Mar165 and an Actor sample changer. The HRD is optimized to study large unit cells and weakly diffracting crystals. The beam is highly parallel and can be focused to 85 µm x 10 µm. In contrast, the minimal focus on the MD2 is 25 µm x 5 µm, allowing data collection on crystals less than 10 µm in size. The second beamline, designated X10SA, was inaugurated in February 2005. It features the same HRD as X06SA with a beam size of 50 µm x 10 µm at the sample position.

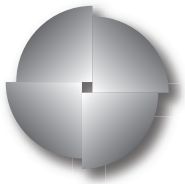
In this talk an overview of the optical system will be given, with particular emphasis on microfocusing. Furthermore, our experience with the microdiffractometer and recent examples of successful data collections on microcrystals will be summarized. The presentation will conclude with an outlook into future developments in the area of beamline automation.

WK12

Structures of Amyloidogenic Peptides Obtained from Microcrystals at ESRF ID13 Microfocus Beamline

Michael Sawaya
Institute for Genomics and Proteomics, UCLA, Los Angeles, CA

The crystal structure of a seven-residue peptide from yeast Sup35 prion was recently solved after five years of struggling for accurate and sufficient data. Although well ordered, the crystals are exceedingly small, the largest being 50 x 2 x 2 µm. Early efforts at data collection focused on powder methods. It was routinely



possible to record diffraction of x-rays to 1.3 Å on an in-house source; however, the problem of overlapping reflections limited the size of the data set to too few reflections. Molecular replacement methods using structure factors extracted from powder data produced feasible solutions but could never be refined to an R-factor below 35%. Single crystals were capable of diffracting electrons to ultra-high-resolution (0.5 Å); however, dynamic scattering effects limited the usefulness of these data. Finally, it was possible to collect single-crystal x-ray diffraction data for the very largest of these microcrystals at the microfocus beamline ID13 at ESRF. Anomalous scattering from a well-ordered zinc ion was sufficient to produce a readily interpretable electron density map using the SAD method of phasing. About ten other peptide structures have since been solved using data collected at ESRF ID13 in three separate data collection trips. Rate-limiting steps include beamline accessibility, quick loss of diffraction due to radiation damage to the crystal, inability to judge crystal quality before the diffraction experiment, and length alignment procedures.

WK12

ID23-2: The New Dedicated MX Microfocus Beamline at the ESRF

D. Flot[1], T. Mairs[2], E. Mitchell[2], M. Guijarro[2], V. Rey[2], D. Nurizzo[2], and S. McSweeney[2]
[1] EMBL, 6, rue Jules Horowitz, BP181 38042 Grenoble Cedex 9, France
[2] ESRF, 6 rue Jules Horowitz, BP220 38043 Grenoble Cedex 9, France

In the early nineties, on the ID13 microfocus beamline at ESRF, it was demonstrated that it was possible to collect diffraction data from crystals of micrometer size (microcrystals) [1-3]. Initially devoted to small-molecule crystallography, pioneering work was carried out to see if it was possible to collect data from very small protein crystals. This pioneering work allowed one of the first structures of membrane proteins to be solved [4] and the advantages of such a method to be evaluated [5]. To take advantage of the experience gained in these early days, it was decided to design a new diffractometer allowing easier crystal alignment, on-axis viewing, etc., in collaboration with the EMBL-Grenoble outstation [6]. Complementary to ID13 and the other six ESRF macromolecular crystallography beamlines, a new beamline (ID23-2) fully dedicated to protein crystallography has been built at ESRF, in order to make available to the macromolecular crystallography community a dedicated tool for protein microcrystals.

ID23-2 is a fixed-wavelength beamline using a single-bounce Si[111] monochromator. The beam is focused down to less than 10 µm in diameter (FWHM) by Pt-coated silicon mirrors in a Kirkpatrick-Baez (KB) geometry. The experimental setup is composed of a MD2M diffractometer (constructed by MAATEL, under an EMBL license), a sample changer (SC3 type), and a MarMOSAIC 225 CCD detector. The beamline has been open to the user community since mid-November 2005. Instrumentation developments and the first results will be presented and discussed.

[1] R.W. Broach, R.L. Bedard, S.G. Song, J.J. Pluth, A. Bram, C. Riekel, H.-P. Weber. *Chem. Mater.* 1999, **11(8)**:2076-2080.

[2] D. Madsen, M. Burghammer, S. Fiedler, H. Müller. *Acta Cryst.* 1999, **B55**:601-606.

[3] R.B. Neder, M. Burghammer, T. Grasl, H. Shulz, A. Bram, S. Fiedler, C. Riekel. *Z. Kristallogr.* 1996, **211(11):**763-765.

[4] E. Pebay-Peroula, G. Rummel, J.P. Rosenbush, E.M. Landau. *Science* 1997, **277**:1676-1681.

[5] S. Cusack, H. Belrhali, A. Bram, M. Burghammer, A. Perrakis, C. Riekel. *Nat. Struct. Biol.* 1998, **5**:634-637.

[6] A. Perrakis, F. Cipriani, J.C. Castagna, L. Claustre, M. Burghammer, C. Riekel, S. Cusack. *Acta Crystallogr. D Biol. Crystallogr.* 1999, **55**:1765-1770.

WK12

Quickly Getting the Best Data from your Macromolecular Crystals with a New Generation of Beamline Instruments

F. Cipriani[1], F. Felisaz[1], B. Lavault[1], S. Brockhauser[1], R. Ravelli[1], L. Launer[2], G. Leonard[3], and M. Renier[3]
[1] *EMBL-Grenoble, www.embl.fr/groups/instr*
[2] *MRC-France/ESRF-BM14, www.bm14.ac.uk*
[3] *ESRF, www.esrf.fr*

An important step forward is being accomplished on improving the quality and throughput of the macromolecular x-ray beamlines of the EMBL/ESRF Joint Structural Biology Group. A new diffractometer generation (MD2x) and a sample changer robot (SC3) that have been developed to perform the most demanding experiments in an automated environment.

The MD2x diffractometer generation is designed to process routinely crystals down to 10x10x10 μm. Constructed around an air-bearing goniometer, it features a sphere of confusion of less than 3 μm, less than 1 milli-degree error at 20°/s and 230°/s maximum speed. It benefits from an on-axis beam-viewing video microscope for precise crystal-to-beam alignment. The air scattering is minimised by a capillary beamstop, ensuring that weak high-resolution spots can be recorded against a minimal background. An additional miniKappa goniometer head allows crystals to be reoriented with minimum risk of collision. Its simplified design benefits from the unique SPINE sample holder length (www.spineurope.org). Tiny crystals can be processed without stability problems, even in fast, multi-pass scans. New data collection strategies will be possible using the STAC software package that is under development, which includes modules for calibration, crystal reorientation calculation, and automatic recentering, as well a multi-pass strategy calculation. The integrated automatic crystal alignment software based on our C3D crystal detection package makes theMD2x diffractometers a primary choice for automated beamlines.

Complementing the MD2x diffractometers, the SC3 sample-changer robot has been designed to automatically handle prefrozen crystals mounted on standard SPINE sample holders and vials. An automatically refilled Dewar can hold up to 50 samples placed in 5 pucks. Any sample in the SC3 is transferred in few seconds to and from a host goniometer by a compact Cartesian transfer arm. The SC3 exploits the DataMatrix codes of the SPINE sample holders to ensure safe management of the sample flow. The sample changer can be operated locally through a touchpad screen or remotely by using a graphical user interface or a device server. Although the SC3 was primarily designed to increase the throughput of beamlines, it has also been shown to be efficient in improving the quality of the data collected. Strategies in screening a large number of crystals and selecting the best, or collecting a full data set from several reoriented microcrystals can now be run with minimum time and effort.

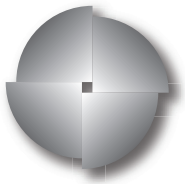
The MD2M diffractometer associated with the SC3 sample changer (ESRF ID14-4).

WK12

NE-CAT Plans for Microdiffraction

Kanagalaghatta Rajashankar
NE-CAT and Cornell University, Argonne, IL 60439

At the NE-CAT beamline at the APS (24-ID-C), we recently collected a 3.5Å SAD data from a ~10x10x100-μm-sized crystal of an enzyme. We were able to determine the structure using the anomalous signal from twelve Se atoms. This is one example of several "smaller" sized crystals that were exposed at 24-ID-C by various user groups, with varying success. Synchrotron beamline scientists are seeing such "smaller" samples at their facilities more often. Obviously, demand for beamlines providing x-ray beams and hardware that are optimized for collecting complete data sets from crystals smaller than 30 μm is increasing. There is also demand for developing optimal strategies to collect complete data sets from such



crystals, keeping radiation damage under acceptable levels. To meet these goals, we have started implementing microdiffraction capability at one of the NE-CAT ID beamlines. This plan and the motivation behind it will be discussed during the workshop.

WK12
Diamond Beamline I24: An Instrument for Macromolecular Microcrystallography

Gwyndaf Evans, Armin Wagner, Kawal Sawhney, and Lucia Alianelli
Diamond Light Source, Diamond House, Chilton OX11 0DE, UK

Beamline I24 at Diamond is being designed to meet the challenges of structural biology of membrane proteins and large macromolecular complexes. Crystals of these molecules are notoriously difficult to grow to sizes appropriate for use on standard third-generation beamlines where beam sizes are typically 30–100 μm. To address problems where crystals of only 30 μm or less are available, a dedicated microfocus macromolecular crystallography beamline is being constructed at Diamond. The beamline will be capable of delivering a focused beam of <5–100 μm onto a crystal sample with high positional and high flux stability. The double K-B pair optical design permits the focal plane to be maintained at the sample position for beam sizes of <5–40 μm. The scientific aims and the beamline design will be presented, along with proposed solutions for improving the stability and quality of the focused x-ray beam at the sample.

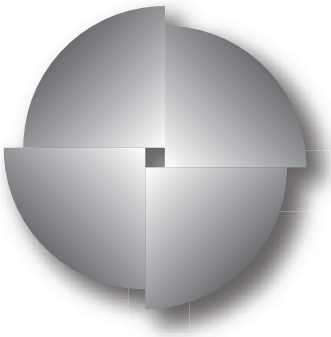
WK12
Plans for Microdiffraction at the Structural Biology Center

Gerd Rosenbaum
Structural Biology Center, Biosciences Division, Argonne National Laboratory; SER-CAT, Department of Biochemistry, University of Georgia

The Structural Biology Center (SBC) at Argonne National Laboratory is planning to more than double its capacity for macromolecular crystallographic structure determination by adding two undulator beamlines (in addition to the existing beamline 19-ID), each with its own undulator source in the straight section of sector 19. One of the new beamlines will have a horizontally deflecting double-crystal monochromator with an output beam offset of 2 m from the incident beam. The other will have a vertically deflecting monochromator with an output beam offset of 1 m. This beam will be transported above the instrumentation of the existing ID beamline 19-ID to an elevated endstation enclosure downstream of the existing endstation of 19-ID. In addition to increasing capacity, the SBC upgrade plans call for one beamline to be equipped with microdiffraction capability. We have chosen the vertically offset beamline because it will be the longest beamline, making it most suitable for a two-stage optics needed to achieve the very high demagnification required in the horizontal direction. Microfoci are needed for very small sample crystals to minimize scatter from nondiffracting material around the crystal. Already now, a sizable fraction of samples brought to 19-ID have a smallest dimension as low as 5 μm. Similarly, microfoci are needed for consecutively exposing different volumes of the sample for a certain range of rotation, e.g., needle-shaped crystals, or for starting each exposed volume at a different starting angle for radiation damage equalization. Further, with microfoci it is possible to reduce radiation damage in the exposed volume (at the expense of nonexposed neighboring volumes that receive a higher doses). The latter requires a spot size in the direction of polarization (the direction in which photoelectrons are preferentially ejected) of 1–2 μm depending on the photon energy. In the direction orthogonal to the rotation axis of the sample (horizontal = plane of polarization), the spot size can be 2–3 times as large because, due to the rotation, a slice much taller than the beam is exposed anyway. Imaging the horizontal source of 650 μm FWHM into a 1-μm spot in one step would result in an impractically short optic-to-sample distance. Instead we will first image the source on a slit by means of a sagittally focusing second crystal stage. The slit of adjustable width will then be imaged on the sample. The very much smaller vertical source size of 20 μm can be demagnified in one step. Since the focal requirement

in the vertical direction is less stringent, we will use a plane mirror at 45° with the horizontal plane to flip the large horizontal source size into the vertical direction. This will allow larger width of the effective horizontal source defined by the slits and larger flux into the focus. Current plans for the focusing optics are mirrors. At micrometer focal sizes, the surface figure error of the mirrors is limiting and requires short image distances. Therefore, the horizontally focusing mirror will be closest to the sample, about 0.5 m, preceded by the 45° mirror, preceded by another horizontally focusing mirror for the vertical focus. These mirrors, the scatter guard slits, the sample goniostat, and the sample alignment microscope will be mounted on one solid base to achieve micrometer-stable positional correlation. Feedback from beam position monitors with micrometer sensitivity will be used to stabilize the optical elements and the beam.

This work was supported by National Institutes of Health Grant GM62414-01 and by the U.S. Department of Energy, Office of Biological and Environmental Research, under contract W-31-109-Eng-38.



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Poster Abstracts

001

Topological Changes in Glassy $GeSe_2$ under Pressure

Q. Mei[1,2], C.J. Benmore[1,3], R.T. Hart[1], E. Bychkov[4], P.S. Salmon[5], C.D. Martin[6], F.M. Michel[6], S.M. Antao[6], P.J. Chupas[7], P.L. Lee[3], S.D. Shastri[3], J.B. Parise[6], K. Leinenweber[2], S. Amin[2], and J.L. Yarger[2]
[1] *Intense Pulsed Neutron Source, Argonne National Laboratory Argonne, IL, USA*
[2] *Arizona State University, Tempe, AZ, USA*
[3] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA*
[4] *LPCA, UMR 8101 CNRS, Université du Littoral, 59140 Dunkerque, France*
[5] *Department of Physics, University of Bath, Bath BA2 7AY, United Kingdom*
[6] *Center for Environmental Molecular Science (CEMS), Department of Geosciences, Stony Brook University, Stony Brook, NY, USA*
[7] *Materials Science Division, Argonne National Laboratory, Argonne, IL, USA*

Monochromatic high-energy x-ray diffraction measurements employing micro-focusing optics were performed on glassy $GeSe_2$ in a diamond anvil cell at pressures up to 9.3 GPa. In addition, the method of isotopic substitution in neutron diffraction was applied to samples that had been densified by 4% via pressurization to 10 GPa in a multi-anvil device and subsequently recovered to ambient conditions. The results reveal a steady increase with pressure of the average coordination number of Ge from 4.0(2) under ambient conditions to 4.5(2) at 9.3 GPa. With increasing pressure, the first sharp diffraction peak in the measured diffraction patterns at ~1.0 $Å^{-1}$ decreases in intensity and almost disappears while the amplitude of the peaks beyond the nearest neighbor in the measured total pair distribution functions gradually increases. Equation of state measurements show a gradual density increase of 33% from ambient pressure to 8.5 GPa, which is in good agreement with molecular dynamics simulations. The results are consistent with the occurrence of two densification processes for glassy $GeSe_2$, namely a conversion from edge-sharing to corner-sharing tetrahedra and a gradual increase in the average local coordination number with increasing density.

002

Wide Angle X-ray Scattering Reveals Crowding-Induced Suppression Protein Breathing

Lee Makowski[1], Diane J. Rodi[1], David Minh[2], Suneeta Mandava[1], David B. Gore[3], and Robert F. Fischetti[1,4]
[1] *Biosciences Division, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439*
[2] *Department of Chemistry and Biochemistry, University of California, San Diego, CA 92093*
[3] *Bio-CAT, Illinois Institute of Technology, 3300 South Federal Street, Chicago, IL 60616*
[4] *GM/CA-CAT, Biosciences Division, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439*

Wide angle x-ray scattering (WAXS) from proteins in solution contains multi-scale structural information which provides dynamic information about protein structure not accessible through crystallographic studies. WAXS studies carried out over a wide concentration range indicates that at low concentrations hemoglobin and avidin undergo structural fluctuations that are suppressed at higher concentrations. This macromolecular crowding effect was not seen in studies performed with lysozyme. These data were applied as restraints on models quantitatively applicable to hemoglobin fold structure. The models were then used to assess the scale of fluctuations in the protein conformation as a function of volume fraction of protein. Increases in protein concentration increasingly restrict protein internal motions among the hemoglobin molecules but not the lysozyme molecules, suggesting that hemoglobin in non-physiologically dilute solutions undergoes structural fluctuations that explore the full range of configuration space in a manner consistent with maintenance of the hydrogen bonding in the protein structure. At physiologically relevant concentrations this range of explored fluctuations becomes suppressed, with concomitant stabilization of the native conformation of the protein. This novel application of WAXS to the quantitation of protein breathing illustrates the importance of non-crystallographic solution-based methodology to the study of protein structure.

003

Self-Assembled Nanostructures in Copolymer Blends

J. Holoubek[1], J. Baldrian[1], S. Malkova[2], and J.Lal[2]
[1] Institute of Macromolecular Chemistry, Academy of Sciences of the Czech Republic, Heyrovsky, Sq. 2, Prague, Czech Republic
[2] Argonne National Laboratory, Argonne, IL 60439-4814, USA

"Bottom-up" nanofabrication uses directed assembling of molecular-scale entities to create larger scale ones with engineering properties. Self-assembly of block copolymers or functionalized nanostructures can generate a rich variety of nanomorphologies (lamellar, cylindrical, gyroid, various cubic, etc.).

Phenomena associated with the order-disorder transition (ODT), microdomain morphology, and phase behavior of two block copolymer systems have been studied by small-angle x-ray scattering (SAXS), small-angle neutron scattering (SANS), and scanning and transmision electron microscopy (SEM, TEM).

1. The microdomain morphology and phase behavior in copolymer blends of A-b-B/A-b-C, i.e., with one block in common (deuterated PS-b-PMMA / deuterated PS-b-PI), were investigated by SAXS, SANS, and TEM. The studied almost symmetric copolymers differ essentially in microdomain morphology. One of them is in disordered microdomain state, while the other displays lamellar morphology at ordinary temperatures. Self-assembled structures in blends were investigated as a function of concentration of the added microphase-separated copolymer and temperature.

2. Self-assembled structures in binary mixtures of A-b-B/A (deuterated PS-b-PMMA/ PS homopolymer) were studied as a function of molecular weight and concentration of the added polymer. A criterion for "wet and dry brush" behaviour respecting different solubilization of the homopolymer in microdomain morphology has been applied to explain the changes in microdomain morphology.

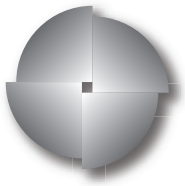
004

X-ray Scattering Studies of Gold and Platinum Nanoparticles Formed by Atomic Cluster Deposition

S. Vajda[1,2], R.E. Winans[1,3], G.E. Ballentine[1], J.W. Elam[4], B. Lee[3], M.J. Pellin[5], S. Seifert[3], G.Y. Tikhonov[1], and N.A. Tomczyk[1]
[1] Chemistry Division, [2] Center for Nanoscale Materials, [3] Experimental Facility Division, [4] Energy Systems Division, and [5] Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

GISAXS is a powerful tool for the determination of the size and shape of nanoparticles dispersed on surfaces or embedded into thin films. It allows for real-time monitoring of the evolution of particle size and shape with temperature and to study the kinetics of particle aggregation under vacuum conditions or when exposed to reactive gases. This technique was used for the characterization of sub-nm to several nm size platinum and gold particles produced by deposition of atomic metal clusters from molecular beams on technologically relevant oxide surfaces. As an example, small Pt clusters supported on Al_2O_3 did not undergo sintering in vacuum and when exposed to hydrogen during a lengthy heat treatment reaching 400°C; Au clusters on SiO_2 remained stable up to 350°C. These temperatures are considerably higher than those characteristic for the onset of the catalytic activity of these clusters. Heat-induced isomerization of clusters and examples of their catalytic performance will be presented and the structure of the oxide surfaces discussed. In summary, these studies made it possible to identify cluster-support combinations for (for example) catalysis-oriented research.

This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Chemical Sciences, Geosciences, and Biosciences under contract number W-31-109-ENG-38.



005

Protein Self-Association Analyzed by Solution X-ray Scattering and Singular Value Decomposition

Tim E. Williamson[1], Bruce A. Craig[2], Elena Kondrashkina[3], Chris Bailey-Kellogg[4], and Alan M. Friedman[1]
[1] *Departments of Biological Sciences and* [2] *Statistics, Purdue University, West Lafayette, IN 47907*
[3] *BioCAT, Argonne National Laboratory, Argonne, IL 60439*
[4] *Department of Computer Science, Dartmouth College, Hanover, NH 03755*

The self-association of a protein existing in a monomer-dimer-tetramer equilibrium was studied using solution x-ray scattering data simulated from the sub-units of a homo-tetramer of known atomic structure. The simulated curves were linearly combined according to fixed association constants to yield curves representing a mixture of oligomers. Gaussian noise was also introduced to approximate counting error arising during the collection of experimental data. Singular value decomposition (SVD) was used to extract scattering curves representing each oligomer from the noisy data. The SVD analysis identified the presence of three states and a two-dimensional grid search was employed to identify the optimal values of the association constants of both oligomerization events. A methodology was developed that makes use of this information to reconstruct the scattering curves of each oligomer present in the heterogeneous data. These curves are then available for further low-resolution structural analysis.

006

Observation of Fe Vibrational Modes in Heme Using Orientated Single Crystal Nuclear Resonance Vibrational Spectroscopy

Nathan J. Silvernail[1], W. Robert Scheidt[1], Bogdan M. Leu[2], Alexander Barabanschikov[2], J. Timothy Sage[2], Jijong Zhao[3], Wolfgang Sturhahn[3], and E. Ercan Alp[3]
[1] *University of Notre Dame, Department of Chemistry and Biochemistry, Notre Dame, Indiana 46556, USA*
[2] *Department of Physics and Center for Interdisciplinary Research on Complex Systems, Northeastern University, Boston, Massachusetts 02115, USA*
[3] *Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, USA*

Nuclear resonance vibrational spectroscopy (NRVS) arises from the nuclear excitation of a probe nucleus at its resonant energy plus an additional quantum of energy resulting in fluorescent photons associated with the vibrational relaxation of the target nucleus. With sufficient high-resolution monochomators, and a necessary high flux photon source, these vibrational bands may be observed with a resolution of less than 1.0 meV. This technique allows the observation of all vibrational modes containing Fe motion. Furthermore the excitation is orientationally dependent so that only vibrational modes along the incident x-ray vector (or a projection onto that vector) are observed. This allows additional selectivity to small-molecule single crystals because of their ability to be oriented, thereby enhancing either in-plane or out-of-plane vibrational modes.

Spectra obtained from oriented single crystals of hemes, the iron-containing prosthetic group of heme proteins, display enhancements of modes associated with biologically relevant phenomena such as protein activation and ligand loss. Single crystals of six-coordinate heme diatomics of the general formula [Fe(Porph)(Im)(XO)] (X = C, N, O) have been studied. Low-frequency modes, including Fe-Im stretching and heme doming modes, have been observed. An ongoing study of the interaction of diatomic ligands with heme will be presented highlighting the use of single-crystal NRVS in the observation of modes associated with protein activation and ligand loss.

007

Nanometer Focusing of Hard X-rays with Linear Zone-Plate Multilayer Laue Lenses

Chian Liu[1], R. Conley[1], A. T. Macrander[1], J. Maser[1,2], H. C. Kang[2], and G. B. Stephenson[2]
[1] *X-ray Science Division, Argonne National Laboratory*
[2] *Center for Nanoscale Materials and Materials Science Division, Argonne National Laboratory*

A multilayer Laue lens (MLL) is a new type of linear zone plate, made by sectioning a planar depth-graded multilayer and used in Laue transmission diffraction geometry, for nanometer-scale focusing of hard x-rays [1-4]. To produce an MLL, a depth-graded multilayer consisting of thousands of layers with thicknesses determined by the Fresnel zone-plate law is first grown on a flat substrate. A thin cross section of the multilayer is then polished down to ~10 to 25 μm, yielding a diffracting grating. To achieve efficient focusing to the diffraction limit, the multilayers need to have good mechanical and thermal properties, sharp interfaces, and accurate layer placement for all the layers. In this presentation, the material system of dc magnetron sputtered WSi_2/Si multilayers is compared with that of W/Si in terms of growth characteristics, interface sharpness, and film stress. Multilayers with precise zone-plate structures have been successfully fabricated by using the WSi_2/Si system. Layer thicknesses and layer positions of zone-plate multilayers were analyzed with SEM images. The growth-rate decay during prolonged depositions was studied using x-ray reflectivity measurements on test periodic multilayers grown before, during, and after a zone-plate multilayer growth. An iterative process was used to perfect the growth with predetermined growth-rate corrections to achieve a desired zone-plate multilayer structure. A nearly perfect multilayer consisting of 728 layers with 12.43-μm total thickness and 10-nm outermost zone width has produced a diffraction limited line-focus of 30.6 nm with an efficiency of 44% at an x-ray energy of 19.5 keV. Another multilayer of 1588 layers, 13.25-μm total thickness, and 5-nm outermost zone width has also been tested with even better line-focus.

[1] J. Maser, G. B. Stephenson, S. Vogt, W. Yun, A. T. Macrander, H. C. Kang, C. Liu, and R. Conley, *Proc. SPIE* **5539** (2004) 194.

[2] H. C. Kang, G. B. Stephenson, C. Liu, R. Conley, A. T. Macrander, J. Maser, S. Bajt, and H. N. Chapman, *Appl. Phys. Lett.* **86** (2005) 151109.

[3] C. Liu, R. Conley, A. T. Macrander, J. Maser, H. C. Kang, M. A. Zurbuchen, and G. B. Stephenson, *J. Appl. Phys.* **98** (2005) 113519.

[4] H. C. Kang, J. Maser, G. B. Stephenson, C. Liu, R. Conley, A. T. Macrander, and S. Vogt, "Nanometer linear focusing of hard x-rays by a multilayer Laue lens," *Phys. Rev. Lett.*, in press.

This work 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.

008

Spectromicroscopy Studies to Characterize Sulfur Species in Oxic and Anoxic Forest Soils

Nora A. Tyufekchieva[1], Jörg Prietzel[1], Jürgen Thieme,[2] David Paterson[3], and Ian McNulty[3]
[1] *Lehrstuhl für Bodenkunde, Technische Universität München, 85350 Freising-Weihenstephan, Germany*
[2] *Institut für Röntgenphysik, Georg-August-Universität, 37073 Göttingen, Germany*
[3] *Argonne National Laboratory, Argonne, IL, USA*

Synchrotron-based x-ray absorption near edge fine structure spectroscopy (XANES) at the K-absorption edge (2465 eV to 2495 eV) of sulfur (S) was used for the speciation of different S forms in anoxic topsoil horizons. The analyses were conducted directly on freeze-dried and ground soil samples. The measurements were carried out at the Advanced Photon Source with an intermediate energy scanning x-ray microscope (SXM). The study was conducted on four different soil types in the Lehstenbach catchment in the Fichtelgebirge (Germany). The soils differ in the degree of groundwater influence.

Increasing soil depth and hydromorphology increased the part of reduced S-species (anorganic and organic sulfides) at total Horizons S-pool from 57% to 76% and simultaneously decreased the part of oxidized

S-species (sulfite, sulfone, sulfonate, and sulfate) from 33% to 21%. Evidently sulfur is fixed in humic top soils in the form of oxidized and intermediate sulfur compounds under oxidation conditions. Current methods of chemical analysis for sulfur speciation are highly unsatisfactory, in light of the enormous ecological relevance of S in soils. Wet chemical methods can only distinguish total soil S into operationally defined fractions rather than specific S species. Synchrotron-based x-ray spectromicroscopy (S K edge XANES) allows a quick, direct quantification of different sulfur species in oxic and anoxic soils.

009

The Atomic-Scale Structures of $Ba_xSr_{1-x}TiO_3$ (x = 1, 0.5, 0) and GaN Nanocrystals Studied with High-Energy X-ray Diffraction

M. Gateshki[1], V. Petkov[1], M. Niederberger[2], J. Choi[3], E. G. Gillan[3], and Y. Ren[4]
[1] *Department of Physics, Central Michigan University, Mt. Pleasant, MI 48859*
[2] *Max-Planck Institute of Colloids and Interfaces, Potsdam D-14424, Germany*
[3] *Department of Chemistry, University of Iowa, Iowa City, IA 52242*
[4] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439*

The atomic-scale structures of nanocrystalline $Ba_xSr_{1-x}TiO_3$ (x = 1, 0.5, 0) and GaN powders have been studied using high-energy x-ray diffraction, Rietveld refinement, and the atomic pair distribution function technique. The studies show that the $Ba_xSr_{1-x}TiO_3$ materials are well ordered at nanometer length distances. The three-dimensional atomic ordering in $Ba_{0.5}Sr_{0.5}TiO_3$ and $SrTiO_3$ nanocrystals may well be described by a cubic structure of the perovskite type, similar to that occurring in the corresponding bulk crystals. The three-dimensional atomic ordering in $BaTiO_3$ is more complex. It is cubic-like on average but locally shows slight distortions of a tetragonal-type. The GaN nanomaterials have very limited structural coherence yet possess well-defined local atomic arrangements resembling the wurtzite structure. The study demonstrates the power of x-ray diffraction and the PDF approach in determining the atomic-scale structure of nanocrystalline materials.

010

Interacting Magnetic Nanostructures

V. Rose[1,2], D.R. Lee[2], Y. Choi[2], G. Srajer[2], V. Metlushko[3], and J.W. Freeland[2]
[1] *Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439*
[3] *Department of Electrical & Computer Engineering, University of Illinois at Chicago, Chicago, IL 60607*

Nowadays the hunt for faster and particularly smaller electronic devices is of huge economic interest and stimulates research by clarifying how the needs of microchip technologies match our knowledge of fundamental processes on the nanoscale. While the properties of single magnetic nanostructures are intensively studied, interactions between these elements as a consequence of miniaturization lead to more complex switching mechanisms that need to be elucidated. In our study we present x-ray resonant magnetic scattering measurements of patterned magnetic single (Co and NiFe) and multilayer (Co/Cu/NiFe) heterostructures such as dots, rings, and squares, to extract the magnetization reversal both in terms of layer dependence and in different sections of the nanostructure. These results provide unique insight into the interactions within and between magnetic nanostructures.

Work at Argonne was supported by the U.S. Department of Energy, Office of Science, under Contract No. W-31-109-Eng-38.

011

Time-Resolved Study of Ordering between Quantum Well Islands

Hawoong Hong[1], L. Basile[1,2], P. Czoschke[1,2], A. Gray[1,2], and T.-C. Chiang[1,2]
[1] *Fredrick Seitz Materials Research Laboratory, University of Illinois at Urbana-Champaign, Urbana, Illinois 61801-2902*
[2] *Department of Physics, University of Illinois at Urbana-Champaign, Urbana, Illinois 61801-3080*

The growth of Pb quantum well islands on Si(111) was investigated in a time-resolved manner. The wide-area momentum space mapped in our x-ray diffraction techniques allows simultaneous observation of the lateral ordering and height distributions of Stranski-Krastanov (S-K) islands during deposition. There were two regimes of islands ordering. Below 170K, island ordering occurs with one consistent spacing between them. And above 170K, the diameter of the islands grows linearly with total deposited coverage, which was predicted for edge-mediated interaction between unstrained islands. The island ordering was stable against coarsening with time, at least within the experimental condition. Quantum well effect appears to have strong correlation to island ordering under 170K. This study suggests that the quantum well effect may provide a new mechanism for uniform self-assembled nanostructures.

012

Characteristics of the MBE1 End Station at PNC/XOR

R. A. Gordon[1], E. D. Crozier[1], D.-T. Jiang[2], J. Shoults[3], B. Barg[4], and P. S. Budnik[1]
[1] *Department of Physics, Simon Fraser University, Burnaby, BC, Canada V5A 1S6*
[2] *present address: Dept. of Physics, U. of Guelph, Guelph, Ontario, Canada N1G 2W1*
[3] *Science Technical Centre, Simon Fraser University, Burnaby, BC, Canada V5A 1S6*
[4] *present address: Australian Synchrotron Project, Level 17 Nauru House, Melbourne VIC 3000, Australia*

An end station for in situ characterization of thin films at the PNC/XOR undulator beamline, sector 20 of the Advanced Photon Source, is detailed. The ability to study films in situ on a beamline enables examination of surfaces and interfaces on freshly prepared films, without the influence of a capping layer. The MBE1 molecular beam epitaxy system was designed with this in mind. Now in routine operation and available for General Users on a collaborative basis, the primary function of MBE1 is to undertake polarization-dependent XAFS studies on fresh or stored films, but it also has the capability to do x-ray standing wave and reflectivity measurements. The characteristics of the MBE1 system—its ranges of motions and detector options—will be described in detail, with example data illustrating its functionality.

013

Inelastic X-ray Study of Plasmons in Oriented Single and Multi-Wall Carbon Nanotubes

D. M. Casa[1], M. H. Upton[2], T. Gog[1], J. Misewich[3], J. P. Hill[3], D. Lowndes[3], and G. Eres[3]
[1] *Argonne National Laboratory, Argonne, IL, USA*
[2] *Brookhaven National Laboratory, NY, USA*
[3] *Oak Ridge National Laboratory, Oak Ridge, TN, USA*

We report inelastic x-ray scattering measurements of plasmon dispersion as a function of wave vector in oriented multi- and single-walled nanotubes samples. The π and $\pi + \sigma$ plasmon dispersions are measured for wave vectors parallel to the nanotubes' axis and for wave vectors varying from parallel to perpendicular. The resulting spectra are then compared to plasmon dispersions in graphite. The $\pi + \sigma$ plasmons in both multi- and single-wall nanotubes exhibit systematic shift in energy with respect to graphite and to each other. Specifically, they have a lower energy in multi-wall nanotubes than in graphite, and a still lower energy in single-wall nanotubes. Since the plasma frequency is proportional to the square root of the electron density, we introduce a model to account for the energy variations based on different effective electron

densities. Due to the increasing electron wave function overlap for multiple graphene walls, the systematic variation in plasmon energy can be then explained by an effective lowering of the effective electron density in single- and multi-walled nanotubes.

014

Design and Testing of the Support/Mover System for the Undulators of the Linac Coherent Light Source

Jeff T. Collins, E. Trakhtenberg, and P. Den Hartog
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

The Linac Coherent Light Source (LCLS), a fourth-generation x-ray free-electron source, is being constructed at the Stanford Linear Accelerator Center (SLAC) at Stanford University. The Advanced Photon Source (APS) is significantly contributing to this effort in the area of undulator system development. A precision support and positioning system has been designed for the LCLS undulator systems, and a prototype system has been fabricated and tested for system performance. The LCLS will use a beam-based alignment technique to precisely align all of the 33 undulator segments contained within the 132-m-long undulator line. The requirement for overlap between the electron beam and the x-ray beam, in order to develop and maintain lasing, demands that each quadrupole must be aligned within a tolerance of ±7 μm and that undulator axes must be positioned within 5 μm vertically and 10 μm horizontally. Five cam movers will allow precise adjustment with five degrees of freedom of a 4,000-lb girder supporting structure used to rigidly mount the undulator, its vacuum chamber, a quadrupole, and a beam position monitor in relative alignment. An additional motion transverse to the beam axis allows removal of individual undulators from the beam path for diagnostic purposes. Details of the support/mover system design will be discussed, along with performance results for the precision positioning systems.

015

The EPICS Brick Development, Application, and Current Status

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

The EPICS brick (eBrick) is designed to be a low-cost solution for an IOC with soft real-time requirements. The current implementation is based on the PC104 Athena Single Board Computer from Diamond Systems Corporation. It consists of a Pentium III based processor clocked at 660Mhz, 128Mb of RAM, 4 serial ports, 2 USB (1.0) ports, VGA, mouse, and keyboard inputs, 16 ADCs at 16bit, 4 DACs at 12bit, 3 banks of 8bit digital IO, and a 40GB hard drive. This configuration provides a cost-effective solution with sufficient processing power and functionality for soft IOC real-time requirements. Furthermore, other COTS (commercial off-the-shelf) modules, as well as in-house-developed PC104 modules, can be incorporated.

The eBrick is distributed with EPICS base 3.14.7 and most synApps 5.1 components on top of the VectorLinux operating system (Slackware Linux derivative). VectorLinux is a lightweight, fast distribution that uses <2Gb of disk space and consists of most Linux utilities for EPICS development, as well as system management features.

Ready for deployment at this time, eBricks are being used in sector 26 (Nanoprobe) for controlling the white-beam slits, the Kohzu monochrometer, and other beamline controls. Soon it will be deployed in sector 32 as part of the USAXS–DEI instrument. Furthermore, sector 19 is evaluating two eBricks for their new controls.

016

Synthesis and Properties of Single-Crystalline FeSi and CoSi Nanowires

Andrew Schmitt[1], L. Zhu[1], F. Himpsel[2], D. Schmeier[3], and S. Jin[1]
[1] *Department of Chemistry, University of Wisconsin-Madison, Madison, WI 53706, USA*
[2] *Department of Physics, University of Wisconsin-Madison, Madison, WI 53706, USA*
[3] *Lehrstuhl Angewandte Physik-Sensorik, BTU Cottbus, 03046 Cottbus, Germany*

We report the chemical synthesis of free-standing single-crystal nanowires (NWs) of FeSi, the only transition metal Kondo insulator and the host structure for ferromagnetic semiconductor $Fe_xCo_{1-x}Si$. Straight and smooth FeSi nanowires are produced on silicon substrates covered with a thin layer of silicon oxide through decomposition of the single source organometallic precursor trans-$Fe(SiCl_3)_2(CO)_4$ in a simple chemical vapor deposition process. Unlike typical vapor-liquid-solid (VLS) NW growth, FeSi NWs form without the addition of metal catalysts, have no catalyst tips, and depend strongly on the surface employed. X-ray spectroscopy verifies the identity and the room-temperature metallic nature of FeSi NWs, which is seen in room-temperature electrical transport measurements. This general synthetic approach has also been applied to the formation of CoSi nanowires. Patterning using standard photolithographic techniques shows the possibility for selectively growing nanowires in place for future bottom-up assembly of devices. Investigations into the low-temperature physical properties of the first one-dimensional Kondo insulator and the possible new NW growth mechanism are underway. Additionally, we believe this unique synthetic approach to FeSi and CoSi NWs will apply to many other transition metal silicides.

017

White and Pink Beam Slits for the Hard X-ray Nanoprobe Beamline at the Advanced Photon Source

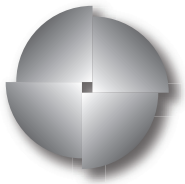
C. Benson[1], Y. Jaski[1], J. Maser[2], T. Powers[1], O. Schmidt[1], and E. Rossi[1]
[1] *Experimental Facilities Division, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA*

A new type of slit has been designed for use in the Hard X-ray Nanoprobe Beamline at the Advanced Photon Source (APS) [1]. The design incorporates monolithic GlidCop slit bodies mounted to commercially available x-y drive systems. Long tapered apertures with adjacent water cooling channels intercept the x-ray beam, removing the high heat load produced by two co-linear APS undulators. The apertures are L-shaped and provide both horizontal and vertical slits. The beam-defining edges, positioned at the end of the tapered surfaces, consist of two sets of tungsten blades. These blades produce an exit beam with sharp corners and assure a clean cut-off for the white beam edges. The slit assembly is designed to allow overlap of the slit edges to stop the beam.

The white beam slit design accommodates 3100 W of total power with a peak power density of 763 W/mm^2. The pink beam slit design accommodates 400 W of total power with a peak power density of 180 W/mm^2. Detailed thermal analyses were performed to verify the slits' accuracy under full beam loading.

[1] J. Maser, G.B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider "Conceptual design for a hard x-ray nanoprobe beamline with 30 nm resolution," *AIP Conf. Proc.* **705**, AIP (2004) 470 – 473.

Work supported by U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.



018

Texture and Residual Strain Measurement under a Mild Scratch on Inconel 600 Using Synchrotron Micro-Laue Diffraction

Marina Suominen Fuller[1], S. McIntyre[1], S. Ramamurthy[1], R. Klassen[2], A. Gerson[3], P. King[4], and W. Liu[5]
[1] *Surface Science, University of Western Ontario, London, Ontario, Canada N6A 5B7*
[2] *Department of Mechanical and Materials Engineering, University of Western Ontario, London, Ontario, Canada N6A 5B7*
[3] *Applied Centre for Structural and Synchrotron Studies, Mawson Lakes Campus, University of South Australia, South Australia, 5022*
[4] *Babcock & Wilcox, Cambridge, Ontario, Canada N1R 5V3*
[5] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

Pressurized water reactor (PWR)–type nuclear power stations are widely used throughout the world. As the nuclear power stations approach their design life, one of the greatest concerns to the owners/operators is the corrosion of steam generators (SGs). This can occur in several different forms: pitting, thinning and stress corrosion cracking (SCC). Nickel-based alloys, for instance Inconel 600 (I600), used in most of the older power stations, are more susceptible to these forms of corrosion. In fact, SCC of I600 tubing is the single most prevalent reason for the replacement of nuclear steam generators. Laboratory simulations suggest that under certain operating conditions, SCC could occur at locations that have sustained mechanical damage, such as fretting, dings and dent marks during manufacturing and/or operation.

The effect of mechanical strain fields, expressed through scratches in polycrystalline nickel alloy A600, has been studied using micro-Laue diffraction and differential aperture microscopy facilities on beamline 34-ID-E at APS. Results from the analyses of deep (0.04 mm) and shallow (0.002 mm) scratches on the polished metal alloy surfaces will be presented. For the deep scratch, material below the scratch was found to be nano-crystalline to the technique sampling depth. Grain structure was detected away from the scratch to depths of 0.06-0.1 mm into the metal, indicating that the stress associated with the deep scratch significantly deformed the metal grain structure. In contrast, for the shallow scratch, grain structure under the scratch was detected, except for a 0.03x0.03mm severely deformed area. The strain under the scratch region appears to be triaxial. Also, a discontinuity in one deviatoric strain component near a grain boundary was noted under the scratch region, as compared to the gradual variation across three grain boundaries in an area away from the scratch. As expected, the magnitude of the strain also appears to be the greatest at the outer surface.

019

XAFS Studies of Candidate Dilute Magnetic Semiconductor Materials

Steve M. Heald, S. A Chambers, T. C. Kaspar, and T. C. Droubay
Pacific Northwest National Laboratory, Richland, WA, USA

The search for a dilute magnetic semiconductor with a high Tc has attracted much interest and resulted in controversial and conflicting findings. These materials are needed for continued progress in spintronics as possible injectors of spin-polarized carriers. XAFS studies at sector 20 have proven to be extremely useful in characterizing candidate materials based on TiO_2-anatase, hematite, and ZnO. To convert these materials into possible magnetic semiconductors, they need to be doped, typically with transition metals at the few percent level. The element specificity and sensitivity of XAFS allows these dopants to be studied in epitaxial films as thin as 20 nm. The near edge can be used to determine the valence of the dopant atoms, important for understanding the charge balance and whether metallic nanoparticles are forming. The EXAFS can determine the local atomic environment, verifying that substitutional doping is occurring and quantifying the lattice distortion at the dopant site. This poster will present recent XAFS results for Co and Cr doped TiO_2-anatase, Ti-doped hematite, and Co-doped ZnO.

020

Synthesis and Physical Properties of Ferromagnetic Semiconducting EuO Nanorods

Matthew Bierman and S. Jin
Department of Chemistry, University of Wisconsin-Madison, Madison, WI 53706, USA

We report the synthesis, structural characterization, and physical properties of nanorods of ferromagnetic semiconducting EuO materials. A semiconductor with a bandgap of 1.1 eV, EuO becomes ferromagnetically ordered at a Tc of 69 K as an ideal Heisenberg ferro-magnet. We are developing rational chemical syntheses of one-dimensional nanoscale single-crystal building blocks of EuX (X=O, S) and other rare-earth chalcogenide materials to exploit them as building blocks for spintronic devices and investigate the size dependences of physical properties of these fascinating materials. We prepare the EuO nanorods via a facile aqueous solution synthesis of $Eu(OH)_3$ nanorods, followed by solid-state conversion to Eu_2O_3 and subsequent reduction to EuO while maintaining nanorod morphology. Reaction progress has been monitored, and the products characterized by powder x-ray diffraction and scanning electron microscopy. Preliminary physical property investigation, such as magnetic measurem ents, will be discussed for these novel nanoscale materials. Our preliminary results toward other rare-earth sulfide nanowire materials will also be reported.

021

Resonant Anomalous X-ray Reflectivity Studies of As(V) Adsorption on Iron and Aluminum Oxide Surfaces

J.G. Catalano, C. Park, Z. Zhang, and P. Fenter
Interfacial Processes Group, Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA

Adsorption of toxic elements onto mineral surfaces reduces their bioavailability and potential for transport. A fundamental understanding of adsorption processes is desired in order to predict the fate of contaminants in complexes systems and to generalize about the behavior of these elements in the environment. The adsorption of arsenate at pH 5 on the (012) surfaces of corundum and hematite was studied using resonant anomalous x-ray reflectivity (RAXR). Two distinct sorbed arsenate species were observed in roughly equal proportions: an inner-sphere species consistent with a bridging bidentate complex, and an outer-sphere species, presumably adsorbed via hydrogen bonding. The relative fraction of arsenate adsorbed in the outer-sphere complex was generally independent of the arsenate concentration in solution. These results suggest that outer-sphere arsenate adsorption may be a significant sequestration mechanism that has been largely overlooked in past studies and whose impacts on the fate and bioavailability of arsenic need further evaluation.

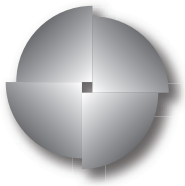
This work is supported by the Geosciences Research Program of the Office of Basic Energy Sciences, U.S. Department of Energy, through contract W-31-109-ENG-38 to Argonne National Laboratory.

022

Lens-Based Large Area CCD Detector for X-ray Crystallography

Timothy Madden[1], M. Molitsky[2], E. Westbrook[3], A. McArthur[4], and I. Naday[5]
[1] *Advanced Photon Source, Argonne National Laboratory*
[2] *Structural Biology Consortium*
[3] *Advanced Light Source*
[4] *Interstate Electronics*
[5] *Aviex Electronics*

An x-ray crystallography detector has been built based upon a Fairchild 486 back-illuminated CCD and custom lens system. The advantages of the lens system over more conventional fiber-optic tapers are improved point spread function, negligible spatial distortion, and lack of "chicken-wire" patterns. Also, the engineer-



ing is simpler because the CCD need not be bonded to the fiber-optic taper. Unique mechanical design has been employed to accurately focus the image on the CCD. The detector software is based on MATLAB and takes advantage of its powerful imaging and signal processing libraries. The CCD timing can be updated on-the-fly by using a "CCD controller language" to specify timing.

023

Marker Motion Measurements of Nanoparticles in Polymeric Matrices

Aleta Hagman[1,2], K. Shull[1], J. Wang[2], X. Li[2], and S. Narayanan[2]
[1] *Department of Materials Science and Engineering, Northwestern University, Evanston, IL, USA*
[2] *X-ray Operations and Research Division, Argonne National Laboratory, Argonne, IL 60439, USA*

Marker motion can be used to infer the ordering kinetics and nanoparticle dynamics in model polymer/metal nanocomposite thin films. In current experiments, the thermally evaporated gold particles (few nanometers in diameter) act as a marker layer between two polymer layers with different mobilities. The disparity in the mobility causes a marker movement towards the layer with the higher mobility. X-ray standing waves (XSWs), generated by total external reflection above a mirror surface, are used to monitor the time evolution of the gold nanoparticle distribution as the nanocomposite ultrathin films are heated above the polymer glass transition temperatures. Diffusion coefficients are obtained from the time dependence of the nanoparticle distribution evolution. Polymer/gold interactions have been shown to be system dependent, with pol(tert-butyl acrylate) (PtBa) showing weak interactions while poly(2-vinyl pyridine) (PVP) shows strong interactions as expected. Free surface effects relating to mobility will also be discussed.

024

Design and Application of CVD Diamond Windows for X-rays at the Advanced Photon Source

Yifei Jaski[1] and D. Cookson[2]
[1] *Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *The University of Chicago, Chicago, IL, USA*

Two types of directly cooled, 0.2-mm-thick, 8-mm-diameter clear aperture CVD diamond windows have been designed and successfully fabricated by two different vendors for use at the Advanced Photon Source (APS). Both windows contain a direct braze joint between the diamond and the cooled OFHC copper. One window was made with a high-quality optical-grade CVD diamond disk and is to be used as front-end exit window. Another window was made with a lower quality CVD diamond disk and to be used as white beam commissioning window. The front-end exit diamond window can be used to replace the front-end beryllium windows in high-heat-load applications and can be used as white beam windows for beamline operation. Small angle scattering (SAX) experiments have been carried out in the 15-ID beamline on the high-quality diamond window, the low-quality diamond window and a polished beryllium window to study the scattering effect on the x-ray beam. This paper presents the detailed design of the diamond windows, the thermal analysis of the diamond windows under different thermal load configurations, and the SAX data comparison on the different grade of CVD diamond and polished beryllium windows.

025

Application of *In Situ* XAFS: Structural Study of Lithium-Ion Intercalation in Battery Materials

Mali Balasubramanian[1], G. Jain[2], and J.J. Xu[2]
[1] *Sector 20, PNC-XOR, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *Department of Ceramic and Materials Engineering, Rutgers, The State University of New Jersey, Piscataway, NJ 08854, USA*

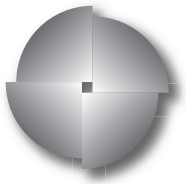
Lithium-ion batteries with high energy and power density are needed for a variety of new existing technologies. The materials that are used in the electrodes of these batteries have crystal structures that can accommodate and release lithium over a fairly large composition range. Over 95% of the present day commercial lithium-ion batteries use a $LiCoO_2$ cathode. Due to the high cost of cobalt, there is considerable interest in developing cheaper alternatives. Knowledge of the redox chemistry and changes in the structure during electro-chemical cycling is of paramount importance in designing new cathode materials with superior properties. Insights into the electronic and structural properties of the transition-metal elements in the cathode materials during charging/discharging can be gained using *in situ* XAFS. The element-specific nature of the XAFS technique and its sensitivity to the local chemical environment make it an ideal tool to study this class of materials. A few examples of *in situ* XAFS studies of battery materials will be presented.

026

Local Structure of Nanosized Dilute Magnetic Semiconductors $Zn_{1-x}Mn_xO$ Studied Using Extended X-ray Absorption Fine Structure

Soma Chattopadhyay[1], S. D. Kelly[2], T. Shibata[3], R. Viswanatha[4], S. Stoupin[3], M. Balasubramanian[5], C. U. Segre[3], and D. D. Sarma[4]
[1] *Physics Division, BCPS Department, Illinois Institute of Technology, Chicago, IL 60616, USA; Materials Research Collaborative Access Team (MRCAT), APS, Bldg 433B, Argonne, IL 60439, USA*
[2] *Biosciences Division, Argonne National Laboratory, Argonne, IL 60439, USA*
[3] *Physics Division, BCPS Department, Illinois Institute of Technology, Chicago, IL 60616, USA*
[4] *Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560012, India*
[5] *PNCCAT, APS, Bldg 435E, Argonne National Laboratory, Argonne, IL 60439, USA*

Diluted magnetic semiconductors (DMSs) are semiconductor solid solutions, where a small percentage of the cations are replaced by magnetic ions, such as Mn, Co, or Fe. In the dilute limit, magnetically doped nanocrystals form a unique class of systems that integrate semiconductor confinement effects arising out of the finite size effects and magnetic properties arising out of the DMS nature of the system. Nanoparticles of $Zn_{1-x}Mn_xO$, a DMS exhibiting a quantum confinement effect, can be used to finetune the band gap (by varying the concentration of Mn) to tune the absorption or emission energies for electronic, electro-optical, optoelectronic, or purely optical devices. The local atomic environment around the Mn atoms within $Zn_{1-x}Mn_xO$ nanocrystals is known to affect the macroscopic properties of the system. Hence it is necessary to accurately study the environment around the Mn atoms in these particles. Here we have synthesized nearly monodisperse Mn-doped (1 to 5 atomic percent) ZnO nanocrystals with an average diameter of 4 nm using a novel wet chemical method. The Mn K-edge x-ray-absorption fine structure (XAFS) and x-ray-absorption near-edge structure (XANES) measurements of bulk and nanocrystals of $Zn_{1-x}Mn_xO$ show that the local structure around the Mn atoms is different in the nanocrystals as compared to the bulk. The differences in the valence state and local atomic environment of Mn as a function of the Mn concentration (which affects the band gap properties of $Zn_{1-x}Mn_xO$), will be presented.



027

A High-Sensitivity, High-Spatial-Resolution CCD-Based Detector for Non-Crystalline Diffraction and SAXS Applications at Third-Generation Synchrotron Sources

I. Naday[1], A. Stewart[1], L. Guo[2], and T. C. Irving[2]
[1] *Aviex LLC, Naperville, IL, USA*
[2] *BioCAT, Department of BCPS, Illinois Institute of Technology, Chicago, IL 60616 USA*

Because of their high flux and excellent beam quality, small-angle instruments on undulator beamlines at third-generation synchrotron sources hold great promise for static and time-resolved studies of muscle and connective tissue fiber diffraction and small angle scattering for solutions of biological macromolecules (SAXS). Detectors have always been one of the major limiting factor for such studies. Multiwire proportional counters cannot operate at the count rates delivered by the APS, and commercially available CCD detectors have been optimized for protein crystallography and lack the sensitivity needed for SAXS. Here we report the results of initial commissioning and characterization studies of a AVIEX PCCD -16080 CCD detector, the first commercial CCD detector designed specifically for biological SAXS applications. It has a relatively large active area of 160 x 80 mm, 4000 x 2000, 38-micron pixels). It is also designed to be exceptionally stable. Asymmetric n x m binning is available to reduce the effect of read noise as well as allow a variety of exotic readout modes to allow ms-scale time-resolved measurements. The implications of the availability of this type of detector for non-crystalline diffraction and SAXS studies at third-generation synchrotron sources will be discussed.

028

Design of a Horizontal Bounce Double-Crystal Monochromator at the Advanced Photon Source

Soon-Hong Lee, T. Gog, M. Beno, and P. Den Hartog
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

At the Advanced Photon Source (APS), sector 12-ID beamlines will be upgraded to use APS dual-canted undulators (CU) and APS CU standard front-end (FE) components. To select a desired beam wavelength from the incident white beam and to split beamlines to double research capacity, a horizontal bounce double-crystal (HBDC) monochromator will be designed and installed in the first optical enclosures (FOE) for the sector 12-ID beamline. The first crystal of the HBDC monochromator actually provides the monochromatizing action, while the second crystal simply redirects the monochromatized beam parallel to the incoming beam with a horizontal plane offset of 750 mm and with a beam height of 1,400 mm for both crystals. In this paper, vacuum/motion design requirements and technical challenges associated with the HBDC monochromator will be presented.

029

Structure Analysis of Diesel Exhaust Fine Particulate Matter with Wide- and Small-Angle X-ray Scattering

Artur Braun[1,2], Frank E. Huggins[1], Gerald P. Huffman[1], Jan Ilavsky[3], Soenke Seifert[4], and Stephen N. Ehrlich[5]
[1] *Consortium for Fossil Fuel Sciences and Dept. of Materials and Chemical Engineering, University of Kentucky, Lexington KY 40506, USA*
[2] *Laboratory for High Performance Ceramics, EMPA – Swiss Federal Laboratories for Materials Testing & Research, CH-8600 Duebendorf, Switzerland*
[3] *UNICAT - Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*
[4] *BESSRC-CAT - Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*
[5] *National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973, USA*

We demonstrate the power of x-ray scattering for the study of diesel exhaust soot. Ultra-small-angle x-ray scattering (USAXS) data clearly resolve soot aggregates, primary particles and their subunits, but with a more robust statistical significance than electron microscopes can do. Compacticity studies allow for derivation of elasticity data on the soot. Wide-angle x-ray scattering (WAXS) data of soot from diesel and ferrocene-doped diesel revealed that adding ferrocene can generate diesel soot that almost entirely lacks in graphitic structure. SAXS and WAXS are hence techniques that permit highly quantitative studies on diesel soot, which is of interest for environmental scientists and combustion engineers.

A. Braun, J. Ilavsky, F.E. Huggins, et al. X-ray scattering and spectroscopy studies on diesel soot from oxygenated fuel under various engine load conditions, *Carbon* **43**, (12), 2588-2599 (2005).

A. Braun, J. Ilavsky, S. Seifert, P. R. Jemian. Deformation of diesel soot aggregates as a function of pellet pressure: A study with ultra-small-angle x-ray scattering. *J. Appl. Phys.* **98**, 073513 (2005).

A. Braun, S. Seifert, J. Ilavsky, et al. Size-range analysis of diesel soot powders and pellets with ultra small angle x-ray scattering. *Combustion & Flame* **137** (1/2), 63-72 (2004).

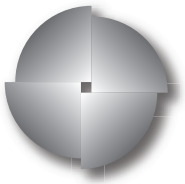
030

Ostwald-Ripening of Cobalt Precipitates in Silica Aerogels? An Ultra-Small-Angle X-ray Scattering Study

Artur Braun[1,2], Frank E. Huggins[2], Edward M. Eyring[3], Jan Ilavsky[4], and Pete R. Jemian[4]
[1] *Consortium for Fossil Fuel Sciences and Dept. of Materials and Chemical Engineering, University of Kentucky, Lexington KY 40506, USA*
[2] *Laboratory for High Performance Ceramics, EMPA – Swiss Federal Laboratories for Materials Testing & Research, CH-8600 Duebendorf, Switzerland*
[3] *University of Utah, Salt Lake City 84112, UT, USA*
[4] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA*

Monolithic silica aerogels with radial symmetry were synthesized by supercritical drying, doped to 2% and 10% with cobalt, and reduced with hydrogen. All samples were investigated with ultra-small-angle x-ray scattering. The nondoped aerogels have three populations of scatterers with radii of gyration of about 1, 4 and 6-7 nm. The doped aerogels show an additional structure with a radius of gyration ranging from 105 – 300 nm. This structure causes intensity oscillations, thus suggesting a relatively narrow size distribution. Scattering curves of the 10%-doped aerogels fitted well to a Lifshitz-Slyozov-Wagner particle size distribution, thus revealing that Ostwald ripening might have occurred during aerogel preparation. The same range also shows differences depending on whether the samples were reduced, or in their as-prepared condition. Scattering curves obtained from the cylinder-axis region were different from the scattering curves obtained from the sample boundary, indicating a process-dependent skin effect.

A. Braun, J. Ilavsky, B. C. Dunn, P.R. Jemian, F. E. Huggins, E. M. Eyring, G. P. Huffman. Ostwald-Ripening of Cobalt Precipitates in Silica Aerogels? An Ultra-Small Angle X-ray Scattering Study. *J. Appl. Cryst.* **38**, 132-138 Part 1 FEB 2005.



031

Nucleation of Glycine Crystals from Supersaturated Solutions Examined by Small-Angle X-ray Scattering

Deniz Erdemir[1], S. Chattopadhyay[2,6], L. Guo[3], J. Ilavsky[4], H. Amenitsch[5], C. U. Segre[2,6], and A. S. Myerson[1]
[1] *Department of Chemical Engineering, Illinois Institute of Technology, Chicago, IL 60616, USA*
[2] *MR-CAT, CSRRI-IIT, Building 433B, Advanced Photon Source, Argonne, IL 60439, USA*
[3] *APS/Bldg 435B/sector 18, Argonne National Laboratory, Argonne, IL 60439, USA*
[4] *Building 438E, XOR-APS, Argonne National Laboratory, Argonne, Illinois 60439, USA*
[5] *Institute of Biophysics and X-ray Research, Austrian Academy of Sciences, Schmiedlstrasse 6, A8042, Graz, Austria*
[6] *Biological, Chemical and Physical Sciences Department, Illinois Institute of Technology, Chicago, IL 60616, USA*

Solution crystallization is an important separation and purification method used in chemical, pharmaceutical and food industries. Hence it is necessary to control the crystallization process to obtain products with desired and reproducible properties, such as size and crystal structure. The early stages of crystallization, simply called nucleation, play a decisive role in determining these properties, so higher levels of control over crystallization can not be achieved without understanding the fundamentals of nucleation. However, an accurate description of this process is still missing. In this work, crystallization of the amino acid glycine is examined by small angle x-ray scattering (SAXS), in order to develop an experimentally based model that can accurately describe the nucleation of small organic molecules from supersaturated solutions. The analysis of the scattering data suggests that glycine molecules exist as dimers in pure aqueous solutions, and monomers in the presence of acetic acid additive. The system obeys power-law behaviour, indicating the presence of fractals in the solution. A transformation from mass fractal structure to surface fractal structure is observed, which could be the signature of a two-step nucleation mechanism, in which the formation of a liquid-like critical cluster is followed by the organization of such cluster into a lattice.

032

Quantitative Measurements of Piezoelectric Response in Ferroelectric Thin Films

D. Do[1], A. Grigoriev[1], R. Sichel[1], P. G. Evans[1], D. M. Kim[1], C. B. Eom[1], E. M. Dufresne[2], and Z. Cai[2]
[1] *Materials Science and Engineering, University of Wisconsin-Madison, Madison, WI 53706, USA*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

Ferroelectric oxide thin films have been widely studied for applications in micro-electronics and micromechanical devices. In order to predict device performance, it is necessary to measure piezoelectric coefficients precisely. Due to the clamping effects of the substrate piezoelectric response in thin films is smaller than in bulk. When the ferroelectric thin films are clamped by the substrate, it is expected that there is no change in a lateral size. Although the longitudinal piezoelectric coefficient (d_{33}) in ferroelectric thin films is measured using various methods, the transverse piezoelectric coefficient (d_{31}) is less known because of difficulties in the measurement.

Time-resolved synchrotron x-ray microdiffraction was used to study the piezoelectricity in Pb(Zr, Ti)O_3 (PZT) thin film capacitors. The longitudinal and transverse piezoelectric coefficients were determined by mapping of intensity of PZT (002) and (103) Bragg reflections. Precise measurements of changes in the lattice constant were performed on PZT capacitors subjected to an electric field. The mean value of d_{33} under unipolar pulses was 53 pm V^{-1}. The values of d_{33} measured with bipolar pulses were different in two polarization states. This can be explained with a model based on the coexistence of switchable and nonswitchable polarization domains.

033

USAXS Characterization of the Micro- and Nanostructure of Polyethylene Used in Orthopaedic Implants

Alessandro Bistolfi[1], K. Simis[2], L. A. Pruitt[2], T. S. Thornhill[1], and A. Bellare[1]
[1] *Department of Orthopaedic Surgery, Brigham & Women's Hospital, Harvard Medical School, Boston, MA, USA*
[2] *Department of Mechanical Engineering, University of California at Berkeley, Berkeley, CA, USA*

Ultra-high molecular weight polyethylene has been used as a bearing material in total joint replacement prostheses for over 40 years. In knee implants, the total lifetime of the polyethylene component is determined by *in vivo* wear and mechanical damage since it is subjected to cyclic stresses and wear. The implants usually last for 10-15 years depending on the patient's age and activity but can fail earlier in younger, more active patients. In this study, high-pressure crystallization was employed to improve the performance of polyethylene components in knee prostheses. High-pressure crystallization leads to an increase in the overall crystallinity and lamellar thickness of polyethylene via extended-chain crystallization. The USAXS camera of the UNICAT beamline at APS was used to measure lamellar spacing and lamellar thickness of polyethylene induced by high-pressure crystallization. *In vitro* fatigue and wear tests, showed that the increase in lamellar thickness and crystallinity associated with high-pressure crystallization led to an increase in fatigue strength of polyethylene without significantly affecting its tribological (wear) properties. The results of this study show that high-pressure crystallization has the potential to improve the lifetime of knee implants by decreasing mechanical damage without compromising wear performance.

034

APS USAXS Instrument—Capabilities and Examples of Science

Jan Ilavsky and P. Jemian
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

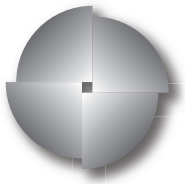
Ultra-small-angle x-ray scattering (USAXS) instrument at a third-generation synchrotron source has been proven to open up new areas of microstructure characterization in materials science by combining the high brilliance and small beam size with a highly flexible instrument design. We will present overview of current instrument capabilities of the APS USAXS instrument in both slit-smeared and 2D collimated geometries. Further we will present examples of science documenting the unique data that this instrument provides for a broad range of applications.

035

Monolayers, Wrinkling and Reversibility in Langmuir Films of Gold Nanoparticles

David G. Schultz[1], D. Li[2], X.-M. Lin[3], J. Gebhardt[4], M. Meron[4], and B. Lin[4]
[1] *University of Illinois at Chicago, Chicago, IL 60607-7059, USA*
[2] *University of Chicago, Chicago, IL 60637, USA*
[3] *ANL Chemistry and Center for Nanoscale Materials, Argonne, IL 60439, USA*
[4] *ChemMatCARS, APS sector 15, Argonne, IL 60439, USA*

Quantum size effects and high surface-to-volume ratio have induced many unique properties for particles with sizes of a few nanometers. Assembling these particles into larger structures offers the possibility of creating new optical, magnetic and electronic devices. The confinement of nanoparticles within a film offers a route for creating microscopically well-defined two-dimensional structures. In this study, the microscopic structure of Langmuir films of gold nanoparticles is characterized with x-ray scattering and *in situ* optical microscopy. We find that gold nanoparticles coated with a layer of dodecanethiol molecules readily adopt a hexagonally packed monolayer structure following compression on a liquid surface. This structure is



relatively insensitive to pressure variations within certain limits, suggesting a fairly rigid lattice. Further compression of the monolayer causes monolayer to fold. Under the right conditions these highly compressed, wrinkled films are found to be capable of unfolding upon relaxation of surface pressure. The ability of the film to return to the monolayer state upon expansion is found to be dependent upon the concentration of thiol molecules in the film.

036
New Controls for 8-ID

J. P. Sullivan, A. R. Sandy, S. Narayanan, T. Mooney, K. Goetze, R. Sluiter, and B.-C. Cha
Argonne National Laboratory, Argonne, IL 60439, USA

When XOR beamline 8-ID became part of the APS, its antiquated, PC-based control system was replaced with standard hardware and software tools from the AOD Beamline Controls and Data Acquisition (BCDA) Group, based on the Experimental Physics and Industrial Control System (EPICS) software toolkit. These changes led to improved functionality, improved ease of use, significantly faster data acquisition, and markedly greater beamline automation and reliability.

037
Compositional Studies of Transition-Metal-Substituted ZnO

S. Kolesnik[1], B. Dabrowski[1], and R. E. Cook[2]
[1] Department of Physics, Northern Illinois University, DeKalb, IL 60115, USA
[2] Electron Microscopy Center, Argonne National Laboratory, Argonne, IL 60439, USA

Theoretical predictions of carrier-induced room-temperature ferromagnetism in p-type Mn-substituted ZnO pointed to a possible novel application of this material in the field of spintronics. Recent studies have claimed that Mn-substituted ZnO can be obtained homogeneous and uniform from low-temperature 500-700°C ceramic processing. We have synthesized and studied structural, compositional and magnetic sproperties of low-temperature processed (Zn,Mn)O. The cation ratio in this material was determined by EDXS analysis. Our results demonstrate that this compound can be formed in air at temperatures higher than 900°C. Above this temperature, the expansion of the crystallographic axes, as well as the increase of the effective Mn content, can be observed. No bulk ferromagnetism was observed for the studied samples. Our results show the significance of compositional studies in the determination of the homogeneity of novel oxide materials. Further studies are required to introduce p-type doping in this material, which appears to be crucial for the existence of room-temperature ferromagnetic order.

Results shown here are derived from work at Argonne National Laboratory, which is operated by the U. of Chicago for the U. S. Dept. of Energy under contract W-31-109-Eng-38. Work was supported by NSF (DMR-0302617) and the U.S. Dept. of Education.

038
Detector Development in the XSD Beam Line Technical Support Group

John Weizeorick[1], T. Madden[1], J. Lee[1], A. Miceli[1], S. Ross[1], P. Fernandez[1], P. Denes[2], H. Padmore[3], D. Capatina[1], and M. Molitsky[4]
[1] Advanced Photon Source
[2] Lawrence Berkeley National Laboratory
[3] Advanced Light Source
[4] Structural Biology Center

This poster will describe several ongoing detector projects being developed by the XSD Beam Line Technical Support Group at the Advanced Photon Source. The first project is a large-area SAXS detector based on a two-by-two array of fiber-optic coupled Kodak CCDs and is being developed for the BESSRC beam lines at the Advanced Photon Source. Unique to this project is the use of extremely sensitive Kodak CCDs,

cryogenic cooling, and low-demagnification fiber-optic tapers yielding excellent detector performance. A second project is in collaboration with Lawrence Berkeley National Laboratory (LBNL). We are developing a Fast CCD (FCCD) detector using a new LBNL CCD with an almost-parallel readout structure, along with high-density custom-integrated circuit digitizers to achieve upwards of 100 frames per second while retaining extremely low readout noise. Finally an update will be given on the APS Detector Pool.

039

Ultra-Small-Angle X-ray Scattering from Dense Nanoparticle Suspensions

Eric Mock, B. Anderson, and C. Zukoski
University of Illinois at Urbana-Champaign

Dense colloid suspensions are encountered in numerous commercial and industrial applications, including adhesives, coatings, and electronic and optical materials. The microstructure of these suspensions is of great interest to the chemist and engineer, as it manifests itself in macroscopic properties such as viscosity, yield stress, conductivity, luminescence, etc. Here we discuss two systems where we have chosen to manipulate particle interactions with the intent of controlling microstructure and investigating the resulting changes in macroscopic properties.

The first system we have chosen to investigate is that of anisotropic colloid particles. As the concentration of these suspensions is increased, one would expect anisotropy to cause the particles to arrange in structures different from the cubic structures formed by concentrated spherical particles. We will present results of ultra-small-angle x-ray scattering on dense suspensions of a variety of anisotropic particles.

Next we explore polymer matrices filled with particulate matter. Changing the molecular weight of polymer that the particles are suspended in, as well as the particle concentration, affects the phase behavior and microstructure of the mixture through enthalpic and entropic contributions. We will present results of ultra-small-angle x-ray scattering on dense suspensions as polymer size is changed.

040

CCD Image Server: An EPICS-Based Server Program for Area Detector Acquisition

Brian Tieman
BCDA-APS

The increasing use of area detectors poses a problem for users of synchrotron light sources. An increasing number of manufacturers of area detectors—from cheap video cameras to expensive scientific grade systems—has brought with it an increasing number of vendor-supplied programs to control these systems. This situation places experimenters in the awkward position of having to learn a different control program for each area detector they may wish to use.

While there is a large variety of vendor interfaces, the basic control options for all these systems is the same. The CCD Image Server provides a common interface for this myriad of area detectors while adding the benefit of an underlying EPICS control interface. The underlying architecture of the CCD Image Server will be explored. The subsystems that allow for a flexible, expandable system that provides a common set of tools for a large selection of area detectors will be explained. Several examples of integrating an area detector into a synchrotron beamline will also be explored.

041

One Interface to Control Them All—The Integrated Tomography System at 2BM

Brian Tieman[1], F. DeCarlo[2], and C. Roehrig[2]
[1] *BCDA-APS*
[2] *XOR-APS*

Performing tomography at a synchrotron beamline, such as 2BM, can be a daunting task. At 2BM two particular problems have arisen. One, scientists must understand how to control the beamline, the detector, a robotic sample changer, and a parallel processing cluster. And two, the current system is capable of completing a single sample—from acquisition to reconstruction—every 15 minutes. This complexity and high throughput often requires that the scientist spend long hours at the beamline performing complicated, but menial, tasks. At 2BM, we've taken the approach of developing a single user interface to walk the user through all aspects of a tomography experiment. It is now possible to set up an entire shift's worth of samples to be run and reconstructed unattended, leaving the scientist free to focus on data analysis or even to sleep! The current status of automated tomography at 2BM will be presented. The details of how systems as diverse as beamline controls, data acquisition, parallel processing, and data archiving are integrated into a single easy-to-use interface will be illustrated.

042

Nuclear Resonance Vibrational Spectroscopy: A Quantitative Picture of Iron Dynamics in Proteins and Model Compounds

Bogdan M. Leu[1], M. Z. Zgierski[2], N. J. Silvernail[3], G. R. A. Wyllie[3], M. K. Ellison[3], W. R. Scheidt[3], J. Zhao[4], W. Sturhahn[4], E. E. Alp[4], and J. T. Sage[1]
[1] *Department of Physics and Center for Interdisciplinary Research on Complex Systems, Northeastern University, Boston, MA 02115, USA*
[2] *Steacie Institute for Molecular Science, National Research Council of Canada, Ottawa, Ontario, Canada K1A OR6*
[3] *Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN 46556, USA*
[4] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

We use quantitative experimental (nuclear resonance vibrational spectroscopy, NRVS) and theoretical (density functional theory, DFT) approaches to characterize the vibrational dynamics of the ^{57}Fe atom in CO- and NO-ligated proteins and porphyrins designed to mimic the active site of heme proteins. NRVS—a uniquely quantitative probe of the vibrational dynamics of reactive Fe sites in proteins and other complex molecules—yields the frequencies, amplitudes, and directions of the Fe vibrations. We discuss the character of normal modes involving Fe motion in the plane of the heme, Fe-Im modes, Fe-ligand modes, and reactive modes.

We report the observation of weaker two-quantum vibrational excitations. The predicted intensities depend strongly on the direction of Fe motion. We compare the observed features with predictions based on the observed fundamentals, using information on the direction of Fe motion obtained either from DFT predictions or from single-crystal measurements.

We use NRVS data on Fe(II) and Fe(III) cytochrome c to identify the Fe vibrational modes, some of them believed to be Raman inactive. We also investigate the previously reported different iron mean square displacements between the reduced and the oxidized states of this protein.

043

Electronic Excitations in Organic Semiconductor Copper Phthalocyanine

C. N. Kodituwakku[1,2], C. A. Burns[1], H. Sinn[2], A. Said[1,2], X. Wang[1], and M. Al-Amar[1]
[1] *Western Michgan Unversity, Kalamazoo, MI 49008-5252, USA*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

Organic semiconductors are of great interest for future technologies. These materials allow for a new generation of ultra-low-cost, light-weight and even flexible electronic devices. We have begun inelastic x-ray scattering (IXS) measurements of the room-temperature electronic excitation spectrum of single crystals of an organic semiconductor, copper phthalocyanine (CuPc). This molecule is essentially planar and has a monoclinic base-centered crystal structure with P2/n space group. We have carried out both resonant and non-resonant inelastic x-ray scattering on CuPc with a resolution of about 120 meV. We compare the IXS data with theoretical calculations.

044

Dewetting and Dynamics of Polymer Bilayer Films

Sarka Malkova[1], M. Mukhopadhyay[2], S. Narayanan[3], L. B. Lurio[4], and J. Lal[1]
[1] *Intense Pulsed Neutron Source, Argonne National Laboratory, Argonne, IL, USA*
[2] *Department of Physics, University of California, San Diego, CA, USA*
[3] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA*
[4] *Department of Physics, Northern Illinois University, DeKalb, IL, USA*

We report grazing incidence coherent x-ray measurements from polymer bilayers consisting of spun-cast layers of polystyrene (PS) and polybromostyrene (PBrS) supported on silicon (Si) wafers. For PS/PBrS/Si bilayers, the films are stable and we are able to probe equilibrium thermal surface height fluctuations using x-ray photon correlation spectroscopy (XPCS). When the layers are inverted to PBrS/PS/Si, the films dewet. In this geometry we can measure both the non-equilibrium evolution of the film structure using time-resolved surface diffuse x-ray scattering and quasi-equilibrium fluctuations of the dewetting film using XPCS.

045

Low-Energy XAFS at High Temperatures and Pressures: Ca^{2+} and Cl^- K-Edge Spectra of Supercritical Aqueous Solution

John L. Fulton[1], Y. Chen[1], S. M. Heald[1], and M. Balasubramanian[2]
[1] *Fundamental Sciences Division, Pacific Northwest National Laboratory, Richland, WA 99352, USA*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

By combining a new diamond window sample cell with improved optics at beamline 20-BM, it is now possible to measure XAFS at high pressures and temperatures for energies as low as the Cl K-edge. XAFS spectroscopy was used to measure the first-shell structure about Ca^{2+} and Cl^- in high-temperature aqueous solution. A focused, 150-µm-diameter x-ray beam is transmitted through the pressurized solution contained between two, 10-µm-thick diamond windows. XAFS spectra were acquired at both the Ca and Cl K-edges at temperatures up to 400°C and pressures up to 350 bar. A global model was used to fit the two independent sets of XAFS data. The results show, that above 250°C, a significant number of Ca-Cl direct contact ion pairs form in agreement with existing thermodynamic data for this system. Overall these measurements provide a structural basis for understanding solvation of Ca^{2+} in hydrothermal systems. These results also provide important new insights into the structural aspects of Ca^{2+} ion pairing that are the basis of many biological processes under ambient conditions.

046

XRD and EXAFS Study of Single-Phase $PbTi_{(1-x)}Mn_{(x)}O_3$ Perovskites at High Mn Concentration

Stanislav Stoupin, C. Segre, S. Chattopadhyay, and T. Bolin
Physics Division, Illinois Institute of Technology, Chicago, IL 60616, USA

We report observation of tetragonal $PbTiO_3$ structure in a $PbTi_{(1-x)}Mn_{(x)}O_3$ system. The new materials are prepared using a sol-gel method with various Mn concentrations. A range of concentrations starting from a relatively high value of x=0.1 is accessed in order to introduce considerable amount of magnetic sites into the system. Reduction in the tetragonal ratio (c/a) is observed by XRD with increase in x. EXAFS study confirms substitution of Ti with Mn in the perovskite unit cell. The effect of reduction of the tetragonal ratio is most likely caused by Jahn-Teller distortion due to removal of degeneracy in the non-zero occupied 3*d* levels. Future study is to determine ferroelectric and magnetic properties of the system.

047

NE-CAT 8-BM Upgrade

Jun Wang, C. M. Ogata, X. Yang, N. Sukumar, M. Capel, I. Kourinov, A.E. Lynch, K. Rajashankar, J. Unik, J. Withrow, and S. Ealick
NE-CAT, Cornell University

The bending magnet beamline (8-BM) is one beamline of the North East Collaborative Access Team (NE-CAT). It is for protein crystallography. The three main optical components of this beamline are a collimating mirror, a double-crystal monochromator with a sagittally bent second-crystal assembly and a vertically focusing mirror, which are used to obtain a monochromatic, focused beam in an experimental hutch located about 50 m away from the x-ray source. Recently, the beamline has undergone an upgrade of its x-ray optics, control system, and the addition of a robot automounter. The first crystal of the double-crystal monochromator was replaced by a new design offered by Oxford Danfysik with a micro-finned, direct water-cooled crystal assembly that would provide better cooling and reduced thermal distortion, pressure bulge and residual strain. X-ray optics characterization has been carried out. Besides the upgrade of the optical components, the installation of the Blu-Ice control system has been initiated at 8-BM, as well as the installation of an automated robotic sample-mounting system obtained from the ALS (Advanced Light Source). Preliminary results are presented.

048

Application of Bond Constraint Theory to the $Ge_2Sb_2Te_5$ Optical Memory Material

D.A. Baker, M.A. Paesler, and G. Lucovsky
North Carolina State University

EXAFS studies of the nearest-neighbor bonding of Ge, Sb, and Te in as-deposited $Ge_2Sb_2Te_5$ (GST) films are presented. Analysis of the Ge K1 EXAFS features indicated significant concentrations of both Ge-Ge and Ge-Te bonds. Additionally, analysis of the three EXAFS spectra is internally self-consistent yielding the following coordination numbers and bond lengths: (i) Ge-Te: 3.3±0.5, 0.263±0.003 nm, (ii) Ge-Ge: 0.6±0.2, 0.247±0.003 nm, (iii) Sb-Te: 2.8±0.3, 0.283±0.003 nm, (iv) Te-Ge: 1.2±0.3, 0.262±0.003 nm, and (v) Te-Sb: 1.2±0.3, 0.283±0.003 nm. These results, in particular, the presence of homopolar Ge bonds, suggest the following molecular-scale structure: $Ge_2Sb_2Te_5 = Ge_2Te_3 + Sb_2Te_3$, with ~17% of Te-atoms 3-fold, rather than 2-fold, coordinated. The average bonding coordination, <r>, and average number of stretching and bending constraints/atom, <n>, have been determined using bond-constraint theory (BCT). In applying BCT to GST, it is necessary to include broken bond-bending constraints for the tetrahedrally bonded Ge atoms in the Ge_2Te_3 arrangements. These broken constraints reduce <n> for Ge from 7 to 4.33. The inclusion of Ge-Ge bonding in Ge_2Te_3 groups provides the microscopic basis for the switching ability of GST.

049

Depth-Resolved Stress Profiling in Nanocrystalline Films with Submicron Resolution

Gang Chen[1], D. Singh[1], O. Eryilmaz[1], A. Erdemir[1], J. Routbort[1], B. Larson[2], and W. Liu[3]
[1] *Energy Technology Division, Argonne National Laboratory*
[2] *Materials Science & Technology Division, Oak Ridge National Laboratory*
[3] *Advanced Photon Source, Argonne National Laboratory*

Thin-film hard coatings (a few microns in thickness) are used for various applications for protecting bulk substrates from abrasion, corrosion, wear or to provide low-friction. Performance and adhesion of the coatings is closely related to the residual stresses throughout the film and into the substrate. Therefore, understanding the residual stresses is essential for developing excellent coatings with desired properties.

Typical stress-measurement techniques provide averaged information over the entire depth of thin films, providing little information of the stress profile. Microdiffraction with submicron x-ray beam size is a powerful tool to fill this gap, i.e., to measure depth-resolved stress profile in thin films with high spatial resolution.

Materials selected for this study are hard Mo and MoN thin films on Si substrates, which are fabricated by physical vapor deposition. These crystalline films have a dense columnar structure with average grain size of 10 -50 nm. By using submicron beam x-ray diffraction on the XOR/UNI 34-ID beamline, we obtained depth-resolved stress profiles in the as-deposited and annealed films. The effect of annealing on the stress profile of the thin films is discussed. Our results demonstrate that microdiffraction can be used for studying depth-resolved stresses in nanocrystalline thin films with submicron resolution.

050

In Situ Characterization of Catalysts and Adsorbents Using Synchrotron Powder Diffraction

Ning Yang,[1] J. Karapetrova,[1] P. Zschack,[1] R. Broach,[2] N. Greenlay,[2] M. Gatter[2], and S. Wilson[2]
[1] *Argonne National Laboratory, Argonne, IL, USA*
[2] *UOP, Des Plaines, IL, USA*

A new *in situ* reactor system was designed and built for use in characterization of industrial catalysts using synchrotron x-ray powder diffraction. This is an extremely versatile reactor system that allows heating and treatment of catalysts and adsorbents to high temperatures and high pressure under static or flowing gases and liquid chemicals. The uniqueness of the reactor system is its ability to easily handle a wide variety of sample types under various conditions that are close to commercial operating conditions, while also allowing collection of x-ray diffraction patterns with great angular precision and high intensity.

The structure changes occurring under different environments in an aluminophosphate molecular sieve, AlPO4-14, were investigated using the *in situ* reactor. AlPO-14 has been demonstrated to preferentially absorb propene over propane under some conditions indicating its potential applications in separation processes. Synchrotron data were collected in atmospheres of water, N_2, propane and propene at temperatures up to 300°C and pressures up to 90 psi. With water, dramatic structure changes were observed, presumably due to expansion of coordination sites around the Al atoms. Propane had no affect on the AlPO-14 structure under the conditions studied. Propene caused variable expansion of the unit cell lattice under different conditions. Rietveld refinement for conditions giving high propene loading revealed the locations of adsorbed propene. Using the structure data from the refinement, molecular dynamics calculations were performed to determine the absorption pathway.

051

Inelastic X-ray Scattering Study of Supercooled Liquid and Solid Silicon

A. Alatas[1], A. Said[1,2], H. Sinn[1], E. E. Alp[1], C. N. Kodituwakku[1, 2], M.-L. Saboungi[3], and D. L. Price[4]
[1] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *Western Michigan University, Kalamazoo, MI, 49008, USA*
[3] *CRMD, Orleans, France*
[4] *HFIR Center for Neutron Scattering, ORNL, Oak Ridge, TN 37831, USA*

We present high-resolution inelastic x-ray scattering measurements of the dynamical structure factor of silicon at the same temperature (T=1300°C) for supercooled liquid and solid phases. Two significant changes in the averaged longitudinal sound velocities and in the longitudinal modulus are observed. First, we observe a different longitudinal modulus in the polycrystalline hot-solid silicon compared to the extrapolated value obtained from the single-crystal measurement. This reduction of the modulus may be an indication of premelting. Second, the increase in the longitudinal modulus in the liquid upon supercooling is consistent with an increase in the degree of the directional bonding.

052

Dynamics of Strain and Polarization in Ferroelectrics

Alexei Grigoriev[1], D.-H. Do[1], D. M. Kim[1], C.-B. Eom[1], P. G. Evans[1], B. Adams[2], and E. M. Dufresne[2]
[1] *University of Wisconsin-Madison, WI, USA*
[2] *Argonne National Laboratory, Argonne, IL, USA*

Structural transitions in ferroelectrics, such as polarization switching, are fundamentally interesting for exploration of the atomic structure modification due to interactions with electric field. Studying these transitions is also important for development of electro-mechanical actuators and non-volatile memories. Although the physics of ferroelectric materials is relatively well developed, there is little known about the dynamics of polarization switching. The time and spatial scales of the dynamics are defined by the speed of the elastic wave propagation in a solid. This means that for micron-size scales, which are technically important and reasonable for applying electric fields of the magnitude adequate for driving polarization switching, the time scale of structural modifications is of the order of nanoseconds. Using time-resolved synchrotron x-ray microdiffraction, we have studied the crystal structure dynamics of a $Pb(Zr,Ti)O_3$ thin ferroelectric film. As a result, we have visualized the polarization switching process and the nucleation of domains and the propagation and velocity of domain walls at scales of microns and nanoseconds. The domain wall velocity scales with the electric field, and, using time-resolved x-ray microdiffraction, the structural dynamics can be studied at fields up to the breakdown fields of ferroelectric films.

053

New Capabilities of Probing Ion Adsorption at Solid-Liquid Interfaces with Resonant Anomalous X-ray Reflectivity

Changyong Park[1], P. A Fenter[1], N. C. Sturchio[2], K. L. Nagy, and J. R. Regalbuto[3]
[1] *Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *Department of Earth and Environmental Sciences, University of Illinois at Chicago, Chicago, IL 60607, USA*
[3] *Department of Chemical Engineering, University of Illinois at Chicago, Chicago, IL 60607, USA*

The ability to obtain element-specific information at solid-liquid interfaces is important for studying surface and colloid chemistry. We recently demonstrated the new capabilities of probing geometric and spectroscopic structures of aqueous metal complexes adsorbed at mineral-water interface simultaneously (C. Park et al., 2005, PRL **94**, 076104) using resonant anomalous x-ray reflectivity (RAXR). This new technique combines the merits of x-ray reflectivity (e.g., interface-specificity and structural sensitivity) and x-ray absorption spectroscopy (e.g., element-specificity and spectroscopic sensitivity). We demonstrate two new opportuni-

ties derived from this method: (1) the determination of model-independent ion distributions at the solid-liquid interface from derived amplitude and phase information of the RAXR spectra; and (2) the use of the RAXR signal to probe ion adsorption isotherms. A fundamental finding from these preliminary observations is that adsorption of multivalent cations is more complex than previously inferred based on classical theories.

Work performed under the auspices of the Office of Science, Geoscience Research Program, US-DOE under contract number W-31-109-ENG-38.

054
In Situ XAS Characterization of Aligned Carbon Nanotubes for PEM Fuel Cell Application

Junbing Yang[1], D.-J. Liu[1], and D. J.Gosztola[2]
[1] *Chemical Engineering Division, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *Chemistry Division, Argonne National Laboratory, Argonne, IL 60439, USA*

The proton exchange membrane fuel cell (PEMFC) continues to benefit from intense development efforts due to a number of inherent advantages including high efficiency, low noise low emissions, and low operating temperature. At the heart of a PEMFC is a membrane electrode assembly (MEA), which consists of an anode, a cathode and a membrane electrolyte. At present, the electrode catalyst materials used at anode and cathode are primarily platinum supported over amorphous carbon. Since platinum is a precious metal with limited supply, reducing its usage will result in significant reduction in PEMFC cost for the commercialization. At Argonne, we have developed a novel method of preparing MEA for PEMFC that will reduce precious metal usage and simplify the design and fabrication of the fuel cell stack. The new approach is built on the unique properties of aligned carbon nanotubes (ACNTs) as the electric-conducting support for electrode catalyst. In addition, the transition metal (TM) precursor used in ACNT synthesis can form the active site for the oxygen reduction reaction for PEMFC.

In this presentation, we will describe our recent progress in characterizing the nature of the transition metal as the active site in ACNT using an *in situ* x-ray absorption spectroscopic method. A modified electro-chemical cell was used with the carbon nanotubes supported by a graphite layer as the working electrode. The liquid electrolyte was saturated by oxygen bubble, while the cell potential is adjusted to various values with a potentiostat. The oxidation state and coordination structure of the transition metal embedded inside the nanotubes were monitored by the EXAFS spectra collected during the electro-chemical reaction. We clearly observed the change of electronic and coordinational structures of the active site during the oxygen reduction reaction. Details of the study will be discussed.

This work was funded by U.S. Department of Energy. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38. The scanning electron microscopy was performed in the Electron Microscopy Center at Argonne.

055
Collaborative Development of Beamline Software

Tim M. Mooney
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Selected features of the collaboratively developed, EPICS-based, beamline software package called synApps will be shown. New features of synApps version 5 (EPICS version 3.14), and new software currently under development, will be described. Contributions to synApps from developers not affiliated with the APS will be highlighted.

056

Blu-Ice Implementation at GM/CA-CAT

Oleg Makarov, Alex Urakhchin, Satish Devarapalli, and Sergey Stepanov
GM/CA-CAT, Biosciences Division, Argonne National Laboratory

Dynamical developments in protein crystallography (PX) force PX groups to work at multiple synchrotron facilities and do experiments in less than a day. This creates a strong demand for standardized graphical user interface (GUI) for data acquisition software at different beamlines. Therefore, GM/CA-CAT made a commitment to adapt the SSRL Blu-Ice, which is praised for its convenience and popular within the community. This, however, created a challenge because Blu-Ice is a complete control system, while most of the APS beamlines, including GM/CA, run on EPICS. In our effort to bridge the two worlds, only the GUI part of Blu-Ice was preserved; the underlying infrastructure was replaced by EPICS. Each instance of Blu-Ice has become an EPICS client like an MEDM with additional controls to compose a PX experiment. Blu-Ice itself has a functionality to do simple beamline control operations, while it delegates more complicated tasks to EPICS servers for frame processing, edge scanning and more features to come.

Blu-Ice has been operational on the GM/CA 23-ID inboard beamline for 9 months, and the 23-ID outboard beamline is currently being commissioned. The works in progress are interfacing Blu-Ice to the ALS-style sample automounter and automated sample centering.

057

Dynamical Strain Imaging in Multiferroic Thin Films

Rebecca Sichel[1], Alexei Grigoriev[1], Dal-Hyun Do[1], Rasmi Das[1], Chang-Beom Eom[1], Paul Evans[1], and Zhong-hou Cai
[1] *University of Wisconsin-Madison*
[2] *Argonne National Laboratory*

Bismuth ferrite is a multiferroic material that has many exciting applications in sensing and data storage. Therefore, the switching dynamics are of concern to potential applications as well as providing an interesting insight into the relationship between the ferroelectric polarization and structural properties. Using x-ray microdiffraction at XOR 2-ID-D, we focused on a single bismuth ferrite capacitor and measured the change in Bragg reflection angle as a function of applied electric field, thus determining the piezoelectric coefficients in different directions (pseudocubic d33 and d31). We also observed splittings in the (002) and (103) peaks, indicating different rhombohedral structural variants. Different structural domains were seen by meeting the conditions for a particular variant's peak and scanning the sample's surface. These domains were shown to be repeatably switchable under electric fields larger than E_c.

058

GM/CA-CAT: Three Beamlines for Protein Crystallography at the APS

R. F. Fischetti, M. Becker, R. Benn, S. Corcoran, S. Devarapalli, O. Makarov, S. Rossi, R. Sanishvili, W. W. Smith, S. Stepanov, A. R. Urakhchin, S. Xu, D. Yoder, W. Diete, M. Schwoerer-Boehing, R.Signorato, and J. L. Smith
Biosciences Division, Argonne National Laboratory
ACCEL GmbH, Germany
Life Sciences Institute, University of Michigan

The National Institute of General Medical Sciences and National Cancer Institute have established the GM/CA-CAT at Argonne National Laboratory to build and operate a national user facility for crystallographic structure determination of biological macromolecules at the APS. We are completing construction of a facility at sector 23 consisting of three beamlines: two on independently tunable canted-undulator sources encompassing an energy range from 3.5 keV to 35 keV and one on a tunable bending magnet. The undulator lines are equipped with focusing mirrors and are capable of producing a focused beam 25 μm in the vertical by 60 μm in the horizontal. Several novel features implemented to achieve quality diffraction

from a variety of sample types, sizes and qualities and include "bimorph" mirrors with positional feedback, air-bearing goniometry, miniature translation stages, and high-resolution on-axis sample viewing. Development of a crystal automounter for all three beamlines is well under way. Blu-Ice has been converted into a client of the EPICS distributed control environment to provide the user interface to the experiment. Fast, continuous scans have been implemented for all beamline components at the hardware level based on novel motion controllers utilizing fiber links. User experiments are now being carried out on the initial undulator line. Commissioning of the second undulator beamline is in progress.

GM/CA-CAT has been funded in whole or in part with Federal funds from the National Cancer Institute (Y1-CO-1020) and the National Institute of General Medical Science (Y1-GM-1104).

059
Hardware-Synchronized On-the-Fly Scans at GM/CA-CAT

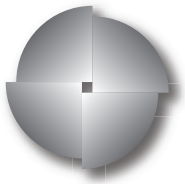
Sergey Stepanov[1], O. Makarov[1], E. Kondrashkina[2], B. Deriy[3], D. Yoder[1], S. Devarapalli[1], S. Corcoran[1], and R. Fischetti[1]
[1] GM/CA-CAT at Advanced Photon Source, Biosciences Division, Argonne National Laboratory
[2] Rigaku Innovative Technologies, Auburn Hills, MI
[3] Accelerator Systems Division, Advanced Photon Source, Argonne National Laboratory

Increasing efficiency of the APS beamlines is an important task because most beamlines are overbooked. Since a considerable share of beam time is consumed by various alignment and data-acquisition scans, the efficiently can be raised tenfold by using on-the-fly scanning algorithms. As an additional benefit for biological studies, fast scans help reduce radiation damage to samples. At the GM/CA-CAT, we have developed hardware and software for on-the-fly scans using advanced features of the Turbo-PMAC motion controllers. The software reprograms the PMAC controller before each scan, so that, for a given motion the pulse and direction, signals are fed into the Struck multichannel scaler via a converter producing up and down pulses. As a result, the scaler accumulates arrays of x-ray intensities vs. axis position. Importantly, scan axes can combine motions of several motors. With our system we have done scans with 1ms time resolution and the hardware limit is ~5 μs. The software exists as GUI implementing multidimensional scan and Perl scripting library. It also supports fast scans with fluorescence detector in protein crystallography. In that case the detector signal is discriminated by a triple-channel analyzer to select the region of interest and then is fed into the scaler.

060
GM/CA-CAT Computing Infrastructure for Fast Protein Crystallography

Satish Devarapalli and S. Stepanov
GM/CA-CAT, Biosciences Division, Argonne National Laboratory

With the great demand for high-throughput data collection in protein crystallography, where experiments typically last a day or less and produce hundreds of gigabytes of data, it is vitally important to have an efficient computing infrastructure for fast data storage, retrieval, and processing. At GMCA—CAT, we have developed cluster-type computing environments for each of our three beamlines to meet the computing needs of user experiments. The computing infrastructure at each beamline consists of 8TB storage area network (SAN) with 2Gb fiber connectivity and a mix of Linux and Windows servers. All experimental data are stored on the SAN and available to all computers through deployment of Global File System. A centralized user account management for all of Linux and Windows systems is setup using OpenLDAP and providing users with a common username and password to log onto any computer and see the same home directory. A GUI account management utility has been developed that simplifies account setup and data backup procedures to the level where any beamline staff member can do it easily. We also implemented a backup system to copy data to users' disks in background mode while the experiment is running, thus providing significant time savings.



061

Fabrication and Characterization of an Ultrasensitive Tin Oxide Nanoparticle Sensor

Ganhua Lu[1], K. L. Huebner[2], L. E. Ocola[3], S. Streiffer[3], M. Gajdardziska-Josifovska[4], and J. Chen[1]
[1] *Department of Mechanical Engineering and Laboratory for Surface Studies, University of Wisconsin-Milwaukee, Milwaukee, WI 53211*
[2] *Department of Mechanical Engineering, University of Wisconsin-Milwaukee, Milwaukee, WI 53211*
[3] *Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439*
[4] *Department of Physics and Laboratory for Surface Studies, University of Wisconsin-Milwaukee, Milwaukee, WI 53211*

Miniaturized gas sensors or electronic noses based on nanostructured materials to rapidly detect and differentiate trace amount of chemical agents are attractive for environmental monitoring, medical diagnosis, and food processing. In this poster, we report on the fabrication and characterization of a very responsive tin oxide nanoparticle gas sensor. First, a convenient and low-cost mini-arc plasma source is used to synthesize tin oxide nanoparticles. The nanoparticle size distribution is measured online using a scanning electrical mobility spectrometer (SEMS). The product nanoparticles are analyzed *ex situ* by high-resolution transmission electron microscopy (HRTEM) for morphology, crystal structure, and defects. Non-agglomerated rutile tin oxide nanoparticles as small as a few nm have been produced. These high-quality nanoparticles are then assembled onto an e-beam lithographically patterned interdigitated electrode using electrostatic force to fabricate the gas sensor, whose profile is examined using field emission scanning electron microscopy (FESEM). DC Resistance and AC impedance measurements are performed to characterize the gas sensor performance. The nanoparticle sensor exhibits an ultrafast response and high sensitivity when exposed to low concentration reducing gases.

062

Nuclear Resonance Vibrational Spectroscopy (NRVS) of Iron-Sulfur Cluster Metalloproteins and Model Compounds

Yuming Xiao[1,3], M. Smith[1], H. Wang[2], S. George[2], Y. Guo[1], W. Sturhahn[3], J. Zhao[3], E. E. Alp[3], and S. P. Cramer[1,2]
[1] *Department of Applied Science, University of California, Davis, CA 95616*
[2] *Lawrence Berkeley National Laboratory, Berkeley, CA 94720*
[3] *Advanced Photon Source, Argonne National laboratory, Argonne, IL 60439*

Iron-sulfur cluster-containing metalloproteins play key roles in many important bio-chemical 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 resonance vibrational spectroscopy (NRVS) is a relatively 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-iron, we have used NRVS to study simple iron-sulfur cluster containing enzymes and relevant model compounds. Detailed vibrational spectra, obtained at the SRI-CAT sector 3ID-D station of the Advanced Photon Source, have been recorded for rubredoxin, ferredoxins, $[NEt_4][FeCl_4]$, $[PPh_4][FeCl_4]$, $[NEt_4]_2[(FeCl_2)_2\text{-}S)_2]$, $[NBu_4]_2[Fe_4(SPh)_4]$, nitrogenases and isolated FeMo-cofactor amongst others. Density of states spectra were extracted from the raw spectra using PHOENIX [3] and compared with molecular mechanics calculations.

[1] "Nitrogenase MoFe-Protein at 1.16 Å Resolution: A Central Ligand in the FeMo-Cofactor," Einsle, O.; Tezcan, F. A.; Andrade, S. L. A.; Schmid, B.; Yoshida, M.; Howard, J. B.; Rees, D. C. *Science,* 2002, **297**, 1696-1700

[2] "Phonon Density of States Measured by Inelastic Nuclear Resonant Scattering," Sturhahn, W.; Toellner, T. S.; Alp, E. E.; Zhang, X.; Ando, M.; Yoda, Y.; Kikuta, S.; Seto, M.; Kimball, C. W.; Dabrowski, B. *Phys. Rev. Lett.,* 1995, **74**, 3832-3835

[3] "CONUSS and PHOENIX: Evaluation of Nuclear Resonant Scattering Data," Sturhahn, W., *Hyperfine Interactions,* 2000, **125**, 149-172.

063

Interfacial Cu of CdTe-Based PV Device

Xiangxin Liu[1], V. Plotnikov[1], J. Terry[2], and A. D. Compaan[1]
[1] *The University of Toledo, Ohio*
[2] *Illinois Institute of Technology and University of Notre Dame*

High-performance CdS/CdTe thin-film solar cells are usually completed with a low-resistance Cu back contact. Our EXAFS studies suggest that most of the Cu atoms are not electrically activated but form copper oxide and copper telluride, and about 90% of the Cu atoms locate at the interface between the CdTe absorber layer and the Au back contact. The effect on cell performance of these copper compounds at the interface has been studied by a recontacting process, in which we physically separate (peel off) the Au contact from CdTe film followed by a secondary deposition of various new metal contact. Cell performance study before and after recontacting suggests almost no effect of the interfacial copper but tremendous impact of the diffused copper over the interfacial layer. Around 90% of the first deposited Cu atoms were found to be peeled off with Au contact in a mixture formation of copper oxide and copper telluride.

064

Radiation Damage Tolerance in $A_2B_2O_7$ Compounds

Gregory R. Lumpkin[1], Katherine L. Smith[2], Mark G. Blackford[2], Susana Rios[1], Karl Whittle[1], and Nestor J. Zaluzec[3]
[1] *C^3I, University of Cambridge, UK*
[2] *ANSTO, Australia*
[3] *Materials Science Division, Argonne National Laboratory*

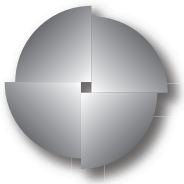
Ceramic waste forms (CWF) provide attractive alternatives to the direct disposal of spent fuel or the immobilisation of high-level radioactive waste in borosilicate glass waste forms, due to their compliance with the principle nuclear safeguards agreements and relatively high aqueous durability, respectively. Over the design lifetime of CWFs, the actinide waste species they incorporate will undergo alpha decay, releasing alpha particles and alpha recoil nuclei. These particles interact with host lattices. In some cases, this will lead to crystalline-amorphous transformations, volume expansion, cracking, and reduced chemical durability due to increased surface area and decreased thermodynamic stability. Consequently, the radiation damage responses of various potential waste form phases (including $A_2Ti_2O_7$ compounds) are of considerable interest. Some aspects of alpha decay damage can be simulated by irradiation with heavy ions. As heavy-ion irradiation does not produce radioactive products, many such radiation damage studies have been undertaken. Our poster will review recent empirical modelling of the response of ($A_2B_2O_7$) pyrochlore and defect fluorite compounds to in situ irradiation with 1 MeV Kr^{2+} ions and discuss new results on the radiation damage response of $Gd_2Ti_2O_7$ (pyrochlore) and two monoclinic layered perovskite-type phases ($Nd_2Ti_2O_7$ and $La_2Ti_2O_7$) in relation to change(s) in symmetry.

065

Magnetic Transitions in $CuFeO_2$ Induced by Magnetic Field and Low Temperatures and Measured Using NFS

S. Kharlamova, E. E. Alp, D. Brown, W. Sturhahn, and Y. Ren.
Argonne National Laboratory, Argonne, IL 60439, USA

The mechanism behind successive transitions through various modulated spin structures has received continuous interest in the field of magnetism. The nuclear forward scattering (NSF) technique is a highly selective and fast method to observe magnetic changes, through internal hyperfine field, quadruple splitting and isomer shift measurements [1]. The magnetic properties of $CuFeO_2$ attracted renewed attention both from experimental and theoretical [2-7] points of view due to the frustration of spin-spin interactions. Here we



present the NFS measurements of $CuFeO_2$, which were performed at station 3-ID-D at XSD the Advanced Photon Source. Spectra were recorded at temperatures of 300 - 4K. Second we measured the spectra of some critical magnetic points [2] applying magnetic fields (up to 7 T) using a cryostat with superconducting magnets.

[1] W. Sturhahn. *Phys.:Condens. Matter.* **16**. S497 (2004).

[2] S. Mitsuda, et al. *J. Phys. Soc. Japan* **60**. 1885 (1991).

[3] M. Mekata, et al. *J. Phys. Soc. Japan* **62**. 4474 (1993).

[4] T. Takagi, et al. *J. Phys. Soc. Japan* **64**. 4609 (1995)

[5] M. Mekata, et al. *J. Phys. Soc. Japan* **62**. 4474 (1993).

[6] N. Terada et al. *J. Phys. Soc. Japan* **73.6**. 1442 (2004).

[7] M. Mekata, et al. *J. MMM*. **104–107**, 823 (1992).

066

PNC/XOR Time-Resolved XAFS Apparatus

Dale L. Brewe,[1] E. A. Stern,[2] S. M. Heald[3], and P. Fons[4]

[1] Advanced Photon Source

[2] University of Washington

[3] Pacific Northwest National Laboratory

[4] Japanese National Institute for Advanced Industrial Science & Technology

We have developed an apparatus for time-resolved XAFS utilizing a high rep-rate laser system for laser pump/x-ray probe measurements on the PNC/XOR ID beamline at sector 20. Initial experiments include studies of laser heating in thin films of germanium and the DVD-RAM material GST ($Ge_2Sb_2Te_5$). We have used the results of the germanium studies to characterize the performance of the apparatus and have demonstrated the ability to obtain excellent time-resolved data quality, with timing performance exhibiting very little jitter compared to the limiting resolution dictated by the bunch width. Results of the germanium studies and preliminary results of the GST study will be presented, and planned improvements to the apparatus will be discussed.

067

Solute Crystallization in Freeze-Dried Formulations—Improved Sensitivity and Quantification Using Synchrotron Radiation

Dushyant B. Varshney[1], S. Kumar[2], E. Y. Shalaev[3], P. Sundaramurthi[1], S.-W. Kang[2], L. A. Gatlin[3], and R. Suryanarayanan[1]

[1] University of Minnesota, College of Pharmacy, Department of Pharmaceutics, Minneapolis, MN 55455, USA

[2] Kent State University, Department of Physics, Kent, Ohio 44242, USA

[3] Pfizer Groton Laboratories, Groton, Connecticut 06340, USA

Freeze-dried pharmaceuticals are typically multicomponent systems containing the active pharmaceutical ingredient and excipients (e.g., buffers). In such formulations the physical form of each component influences the product stability and performance. Previously, we utilized a laboratory x-ray diffractometer to study the freeze-drying cycle. Although the method provided insights into the phase behavior of several systems, the low sensitivity limited the use particularly when the analyte concentration was low. The poor sensitivity was attributed to the low flux of the x-ray source, and the use of a point detector required data collection over long time-periods. These limitations were overcome with the use of collimated synchrotron radiation and a high-resolution 2D detector. Additionally, synchrotron-based x-ray diffractometry (SXRD) enabled rapid data collection (< 1 second), and simultaneous quantification of analytes in multicomponent systems. In this poster, we demonstrate the utility of SXRD for: (1) detecting solute crystallization in frozen sodium phosphate buffer solutions, (2) investigating the effect of pH and buffer concentrations on the crystallized

glycine in frozen solutions and lyophiles, and (3) monitoring phase transitions *in situ* during the entire freeze-drying cycle. The substantially increased sensitivity of SXRD enabled us to monitor freeze-drying cycles of model systems of practical importance.

Current address: Eli Lilly and Company, Lilly Corporate Center, Indianapolis, IN 46285, USA

068

Combined X-ray Reflectivity and GI-SAXS Studies of Nucleation and Growth of ALD Hafnium Oxide High-k Dielectric Films

Andrew Allen,[1] M. Green,[1] X. Li[2], and J. Wang[2]
[1] *NIST, Gaithersburg, MD*
[2] *APS XOR, Argonne, IL*

Atomic layer deposition (ALD) is an important film-growth technique that enables accurate growth of ultra-thin layers for high-k gate dielectrics. Results will be presented of x-ray reflectivity and grazing incidence small-angle x-ray scattering (GISAXS) studies of the nucleation and growth of ALD hafnium oxide films. While reflectivity data reveal and determine the thickness of parallel-sided film formations, the GI-SAXS is related to surface roughness and internal interfaces within the films, resulting from film coalescence of the nuclei. Films grown on H-terminated Si are rough and nonplanar, exhibit greater scattering, and have greater internal surface area than films grown on chemically oxidized Si. These films have 5 times the internal surface area of films grown on chemically oxidized Si, and may be significantly porous. The characteristic scattering features are the film nuclei, which coalesce and become inherited features of the films. The nuclei size is about 2 nm, consistent with TEM observations. Films grown on chemically oxidized Si reach coalescence at about 25 cycles, or 1.3 nm thickness, consistent with electrical data. Implications arising from the different film morphologies will be discussed.

069

USAXS Studies of Growth of Nanocrystalline Ceria in Real Time

Andrew Allen,[1] V. Hackley,[1] P. Jemian,[2] J. Ilavsky,[2] J. Raitano[3], and S.-W. Chan[3]
[1] *NIST, Gaithersburg, MD*
[2] *APS, Argonne, IL*
[3] *Columbia University, New York, NY*

Nanocrystalline ceria is important for a broad array of applications, ranging from catalysts to solid oxide fuel-cell electrolytes, gas sensors, optical coatings, and electronic materials. These applications often require well-crystallized material with a narrow particle size distribution. A promising soft-chemical route for producing near-monodisperse nanoscale ceria particles is based on the homogeneous precipitation method. Typically, this process can be performed at room temperature under generally mild conditions. A real-time *in situ* USAXS study is presented of homogeneous n-ceria precipitation and growth, using an isothermal capillary flow-cell developed at NIST for applications to solution-mediated nanomaterials. Previous TEM studies have shown that the particles are single crystals with narrow size distributions. The growth process was believed to occur in two steps: an initial nucleation event followed by a slow and continuous growth at the particle surface without further nucleation. The USAXS results suggest a more complex growth mechanism, with two size populations that persist throughout the post-nucleation phase. One population remains constant in size, while the other continues to coarsen over time. USAXS measurements yield the nanoparticle size distributions, and the mean size, surface area and number distribution of each population, as a function of reaction time and temperature.

070

Effects of X-ray Optics on Coherence

Mengning Liang and I. K. Robinson
University of Illinois at Urbana-Champaign

Coherent x-ray diffraction (CXD) provides a non-invasive method of imaging the internal structure of nanoscale materials to atomic precision. A single coherent diffraction pattern is the real part of a two-dimensional slice through the three-dimensional complex Fourier transform of the object. The lost phase information may be recovered using iterative phasing algorithms and an image of the projection of the original object may be obtained. The success of the inversion relies greatly on the quality of the image and thus on the coherence properties of the beam. To be considered coherent x-ray diffraction, the temporal and spatial coherence lengths of the x-ray beam must be larger than the physical dimensions of the particle. The coherence is dependent on machine parameters but is also dependent on the optics used to focus the beam to provide sufficient flux onto the sample. According to classical optics, the measure of the coherence of a source is the visibility of the interference fringes produced when light from the source diffracts from an object. Using this figure of merit, we study the coherence effects of x-ray optics using coherent x-ray diffraction from gold nanoparticles.

071

Performance of the New 3.0-cm Undulator at GM/CA-CAT

R. F. Fischetti,[1] D. Yoder,[1] S. Xu[1], and J. L. Smith[2]
[1] GM/CA-CAT and Biosciences Division, Argonne National Laboratory
[2] GM/CA-CAT and University of Michigan

GM/CA-CAT has worked with the APS to develop a new undulator with a period of 3.0 cm. This new undulator has been optimized for macromolecular crystallography experiments utilizing MAD phasing techniques about the Se and Br K-edges. Data comparing the standard 3.3-cm (undulator A) and the new 3.0-cm devices will be presented.

072

Design and Characterization of Compact Beam Cleaner for High-Energy XAS

Benjamin Reinhart and C. Segre
Illinois Institute of Technology, Physics Division, BCPS Dept.

We have designed a compact beam cleaner for use at high energies on an undulator beamline. The design features separate rotary stages for each crystal, a compact bending mechanism and magnetic kinematic mounts for rapid crystal changes. A PIP detector is integrated into the system for rapid alignment. We report here the results of our initial characterization of silicon crystals of 1 mm and 570 μm thickness at varying bending radius at the Ba K-edge. We will present reflectivity data along with spatial acceptance and rocking curve measurements. The results of these initial experiments will determine the final choices for a double-crystal system to be used for Ba x-ray absorption spectroscopy measurements at the MRCAT undulator beamline.

This work is supported by the National Science Foundation under contract DMR0420776.

073

Spectroscopic Studies of Pb Corrosion of Reactor Materials

Daniel Olive, S. Liu, and C. Segre
Illinois Institute of Technology, Chicago, IL 60616, USA

The U.S. Department of Energy has identified lead-cooled fast neutron reactors as a promising technology for small, modular, non-refuelable reactors. These prefabricated reactors are designed to be placed in remote regions for power generation and then transported back to the factory for disposal. However, the corrosive nature of high-temperature lead mandates the careful selection of materials to be used in the primary cooling loop. To determine the kinds of corrosion likely to be found in these new reactors, this experiment attempts to determine the change in molecular structure that occurs at the lead-structural material interface. The materials (316L stainless steel, molybdenum, and spinel) all having been exposed to lead and heated in an inert atmosphere were studied using synchrotron radiation from the Advanced Photon Source at Argonne National Laboratory in both *in situ* and *ex situ* EXAFS experiments.

074

Focal Characterization of "Bimorph" Mirrors

D. Yoder[1], R. F. Fischetti[1], S. Xu[1], R. Benn[1], S. Corcoran[1], and R. Signorato[2]
[1] GM/CA-CAT and Biosciences Division, Argonne National Laboratory
[2] ACCEL Instruments, GmbH

GM/CA-CAT, in collaboration with ACCEL Instruments, GmbH, has implemented the first large "bimorph" mirrors. Biomorph mirrors are constructed from fused silica plates sandwiched around piezo-electric ceramic segments. Each segment may have multiple electrodes. The mirror can be shaped *in situ* by applying voltages to the various electrodes to provide curvature for focusing and minimization of slope error. The mirrors at GM/CA-CAT are arranged in a Kirkpatrick-Baez geometry and the horizontal and vertical focusing mirrors have 14 and 16 electrodes, respectively. This large number of degrees of freedom poses a challenge for optimizing the mirror shape. Algorithms have been developed and will be presented for focusing the x-ray beam with these mirrors. An important advantage of bimorph mirrors is that one can tweak the shape to minimize the slope error and achieve a Gaussian profile not only at the focal point but also upstream of the focus at the sample position, for example. Data will be presented showing the beam profile at the focal position and at off-focus positions.

075

Compact Systems Utilizing YAG Crystals to Image the Monochromatic Beam

S. Xu, R. F. Fischetti, D. Yoder, R. Benn, S. Corcoran, R. Sanishvili, and W. W. Smith
GM/CA-CAT and Biosciences Division, Argonne National Laboratory

Several compact imaging system utilizing YAG crystals (chromium-doped yttrium aluminum garnet, Cr^{4+}:YAG) has been developed as diagnostic tools for monochromatic x-rays. Each of these imaging systems consists of a flat YAG crystal, right-angle prism or mirror, video camera, and monitor. YAG crystals of various diameters have been installed in the UHV vacuum system of the two ID-beamlines just downstream of the major optical components (monochromators, horizontal deflecting mirrors and K-B focusing mirrors). A CCD camera mounted outside the vacuum system views the fluorescence form the YAG crystal through a view port. Other systems have been developed to aid in focusing the beam and for aligning the goniometer rotation axis to the x-ray beam. These YAG imaging systems have greatly facilitated beamline commissioning and sample alignment of small crystals of macromolecules to the x-ray beam. An overview of the optical and mechanical design and performance of these devices will be presented.

076

Inelastic X-ray Scattering Study of the Lattice Dynamics of the Exotic Spin-Peierls Transition in TiOCl

Eric Abel[1], Y. Lee[1], and E. Isaacs[2]
[1] *MIT*
[2] *ANL*

TiOCl has recently received much attention because of an exotic transition into a dimerized state. The transition is spin-Peierls–like, but has some non-conventional behavior, such as an incommensurate state that appears at temperatures just above the dimerization temperature. The dimerization occurs when the incommensurate order becomes commensurate with a wavelength of twice the lattice parameter, resulting in a new zone center at 1/2 the reciprocal lattice vector in the chain direction. Using inelastic x-ray scattering at 3-ID-C at the APS, we have investigated the lattice dynamics leading up to the lattice dimerization. In a uniform 1D spin-chain, energy can be gained if neighboring electrons form spin-singlet pairs. A spin-Peierls transition occurs when this energy gain is greater than the energy required to distort the lattice. The onset of a spin-Peierls transition is indicated by the softening of a zone-boundary phonon to zero. The first inorganic material to exhibit a spin-Peierls transition was the compound $CuGeO_3$. However, there are reasons to question whether the transition in this material is truly spin-Peierls, among which is the absence of a phonon softening, but rather a hardening through the phase transition. In our study we observe what appears to be the softening of the zone boundary longitudinal accoustic mode, indicating that perhaps TiOCl is the most ideal inorganic spin-Peierls material yet observed.

077

Engineering Metal-Impurity Nanodefects for Low-Cost Solar Cells

Tonio Buonassisi[1], A. A. Istratov, M. A. Marcus, B. Lai, Z. Cai, S. M. Heald, and E. R. Weber
[1] *University of California, Berkeley and Lawrence Berkeley National Labortory*

As the demand for high-quality solar-cell feedstock exceeds supply and drives prices upwards, cheaper but dirtier alternative feedstock materials are being developed. Successful use of these alternative feedstocks requires that one rigorously control the deleterious effects of the more abundant metallic impurities. In this study, we demonstrate how metal nanodefect engineering can be used to reduce the electrical activity of metallic impurities, resulting in dramatic enhancements of performance even in heavily contaminated solar-cell material. Highly sensitive synchrotron-based measurements directly confirm that the spatial and size distributions of metal nanodefects regulate the minority-carrier diffusion length, a key parameter for determining the actual performance of solar-cell devices. By engineering the distributions of metal-impurity nanodefects in a controlled fashion, the minority-carrier diffusion length can be increased by up to a factor of four, indicating that the use of lower-quality feedstocks with proper controls may be a viable alternative to producing cost-effective solar cells.

078

High-Throughput Protein Crystallography at IMCA-CAT

A. Mulichak, K. P. Battaile, J. L. Muir, and L. J. Keefe
Sector 17, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

The Industrial Macromolecular Crystallography Association Collaborative Access Team (IMCA-CAT) operates a data collection facility for protein crystallography at sector 17 of the Advanced Photon Source. Equipped with a Rigaku ACTOR robot, the 17-ID insertion device beamline has offered automated sample handling for routine use since 2004. Automated mounting, centering, and retrieval of protein samples enable high-throughput sample screening and unattended data collection, significantly reducing the time neces-

sary for crystal screening and the need for direct operator interaction. The ACTOR system has also greatly facilitated the use of a mail-in data collection option for IMCA members. Upgrades of robotics hardware and software, including a second system with higher storage capacity being installed at the 17-BM bending magnet beamline, continue to expand the role of automation at IMCA-CAT.

While targeting the needs of drug discovery research for IMCA member pharmaceutical companies, the automation capabilities at IMCA-CAT are also ideally suited for structural genomics and other research efforts requiring high-throughput experiments. Such efforts include ongoing collaborations of IMCA-CAT staff with structural genomics centers.

079

Precision Mechanical Design for a Hard X-ray Nanoprobe Instrument with Active Vibration Control in Nanometer Scale

D. Shu, J. Maser, M. Holt, B. Lai, S. Vogt, C. Preissner, A. Smolyanitskiy, R. Winarski, and G. B. Stephenson
Argonne National Laboratory, Argonne, IL 60439, USA

We are developing a new hard x-ray nanoprobe instrument that is one of the centerpieces of the characterization facilities of the Center for Nanoscale Materials (CNM) being constructed at Argonne National Laboratory (ANL). This new probe will cover an energy range of 3-30 keV with 30-nm special resolution. Imaging and spectroscopy at this resolution level require staging of x-ray optics and specimens with a mechanical repeatability of better than 10 nm [1]. Fast feedback for differential vibration control between the zone-plate x-ray optics and the sample holder has been implemented in its design using a digital-signal-processor (DSP)-based real-time closed-loop feedback technique. A specially designed, custom-built laser Doppler displacement meter (LDDM) system provides two-dimensional differential displacement measurement with subnanometer resolution between the zone-plate x-ray optics and the sample holder [2]. Preliminary precision optomechanical design of the hard x-ray nanoprobe is presented in this poster.

[1] J. Maser, G. B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider, *SRI 2003 Conf. Proc.*, **705**, AIP (2004) 470-473.

[2] D. Shu, J. Maser, B. Lai, S. Vogt, M. Holt, C. Preissner, A. Smolyanitskiy, B. Tieman, R. Winarski, and G. B. Stephenson, To be published in the *Proceedings of XRM2005,* Japan.

This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No.W-31-109-ENG-38.

080

XAFS Studies of Fe-Doped $PbTiO_3$ Nanoparticles

Tomohiro Shibata[1], S. Chattopadhyay[1], and V. R. Palkar[2]
[1] BCPS, Illinois Institute of Technology, Chicago IL 60616, USA
[2] Tata Institute of Fundamental Research, Homi Bhabha Rd, Mumbai 400005 India

$PbTiO_3$ is a very well known ferroelectric material with a perovskite structure and the ferroelectric-to-paraelectric transition temperature at 495°C. Properties of these materials can be tailored by doping the cation sites with various elements. Studies have revealed that the doping the Ti site with Fe converts the ferroelectric material into a magetoelectric material, thus making it suitable for memory storage devices. Nanoparticles of $PbTi_{1-x}$ Fe_xO_3 with different amount of the doped Fe have been prepared by coprecipitate techniques. In order to explain the coexistence of magnetism and ferroelectricity in this system, we have studied Ti K-edge, Fe K-edge, and Pb L_3-edge XAFS for these samples. The change in the local structure around Ti Fe and Pb atoms with different in particle size and dopant concentration will be presented.

081

Quantum Effects in Water

C. J. Benmore[1,2] and Q. Mei[1]
[1] *IPNS, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *APS, Argonne National Laboratory, Argonne, IL 60439, USA*

High-energy x-ray diffraction has been used to measure the temperature variation of H/D isotopic quantum effects on the structure of liquid water. The magnitude of the effect increases by a factor of 3.5 as the temperature is reduced from 318K to 268K. The results are compared to a long-standing mean field theory and several recent quantum molecular dynamics simulations.

082

Relationships between Intermediate- and Short-Range Order in Glasses

C. J. Benmore
IPNS and APS, Argonne National Laboratory, Argonne, IL 60439, USA

Pulsed neutron and high-energy x-ray diffraction have been employed to investigate the relationship between local structure and network formation in glasses. Two examples are presented. (1) In amorphous ice, the role of interstitial molecules between the first and second shells greatly affects the extent of intermediate-range order. An analogy is made with the structures of liquid Si and Ge. (2) A comparison of several binary selenide glasses as a function of composition has revealed a strong correlation between the first sharp diffraction peak (associated with intermediate-range order) and the local coordination number.

083

Design of an Ultrahigh Vacuum Artificial Channel-Cut Monochromator for Coherent Scattering Applications

Suresh Narayanan, D. Shu, M. Sprung, and A. Sandy
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

We describe here the design of a compact ultrahigh vacuum (UHV) artificial channel-cut monochromator. The precision and stability of the design facilitates the alignment of two independent crystals to the same performance as does a channel-cut crystal and hence is called an "artificial channel-cut monochromator." The advantage of this design is that the two independent crystals can be super polished to state-of-the-art for preserving the beam brilliance, whereas the same is not feasible with a channel-cut crystal. The monochromator is designed for a small gap between the two crystals (~3 mm), which helps in maintaining a nearly constant spatial offset while changing energy. The monochromator will be used at beamline 8-ID of the Advanced Photon Source for coherent small-angle x-ray scattering studies. The design incorporates a novel in-vacuum sine-bar-drive mechanism for the combined pitch motion of the two crystals and a flexure-based high-stiffness weak-link mechanism for the fine tuning of the pitch and roll of the second crystal relative to the first crystal. The sine-bar is driven by an UHV linear motion stage from Nanomotion Inc. that operates under closed-loop encoder feedback to a precision of better than 10 nm. This concept eliminates the use of bellows to transmit the motion and thereby any vacuum force to overcome. The fine pitch of the second crystal is driven by a combination of picomotor operating under open-loop and a piezo actuator operating under closed-loop conditions.

084

Observation of Surface Layering in a Molecular Liquid

Haiding Mo[1], G. Evmenenko[1], S. Kewalramani[1], K. Kim[1], P. Dutta[1], and S. N. Ehrlich[2]
[1] *Department of Physics and Astronomy, Northwestern University*
[2] *NSLS, Brookhaven National Laboratory*

Oscillatory density profiles (layers) have previously been observed at the free surfaces of liquid metals but not in other isotropic liquids. We have used x-ray reflectivity to study a molecular liquid, tetrakis(2-ethylhexoxy)silane. When cooled to $T/T_c = 0.25$ (well above the freezing point for this liquid), density oscillations appear at the surface. Lateral order within the layers is liquidlike. Our results confirm theoretical predictions that a surface-layered state will appear even in dielectric liquids at sufficiently low temperatures, if not preempted by freezing.

085

XRTA Real-Time Studies on FePt Nanostructures

J. Skuza[1], R. A. Lukaszew[1], and E. Dufresne[2]
[1] *Physics and Astronomy Dept., University of Toledo*
[2] *Advanced Photon Source, Argonne National Laboratory*

Highly ordered $L1_0$ FePt thin films and nanostructures are important for magneto-recording applications because this ordered phase exhibits very large magnetic anisotropy. $L1_0$ FePt nanomagnets can overcome the super-paramagnetic limit that prevents further bit-size reduction when using conventional magnetic materials. One possibility to achieve nanostructures with high degree of chemical order in epitaxial but somewhat chemically disordered FePt films is to perform annealing treatments. One variation of such treatments is rapid thermal annealing (RTA) to limit diffusion and thus preserve the size of the ordered nanostructures. Here we describe an innovative application of x-ray undulator radiation to simultaneously perform RTA and probe structural changes that occur during annealing. In our studies, we have used XRTA to enhance chemical order in epitaxial (001) FePt thin films. We observed the enhancement of the fcc-fct transition with 30-ms temporal resolution in Bragg geometry. The results demonstrate that undulator radiation offers unique possibilities for materials processing, particularly the ability to use the same beam for structure modification and probing.

NSF, Research Corporation and ACS funded this research.

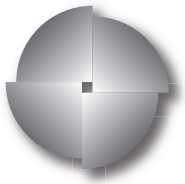
086

The Small-Angle Scattering Instruments at Argonne National Laboratory

Pete R. Jemian
ANL Small-Angle Scattering Group, Argonne National Laboratory, Argonne, IL 60439

Small-angle scattering is the premier technique for the size characterization of nanoscale objects. Twelve different instruments for measuring small-angle scattering (SAXS and SANS) are available from the Scientific User Facilities at Argonne National Laboratory, including the Advanced Photon Source and the Intense Pulsed Neutron Source. The combined capabilities of these instruments span a broad range of reciprocal space and wavelength allowing for investigations from many disciplines of science including materials science, chemistry, environmental science, biology, medicine, and physics.

The Small-Angle Scattering special interest group at Argonne National Laboratory has been created to promote awareness of the small-angle scattering facilities at the APS, IPNS, and elsewhere on the Argonne campus and to foster communications between the various research groups. Through this group, we believe we can build a strong user community for small-angle scattering at the APS, IPNS, and throughout Argonne.



087

Picosecond X-ray Diffraction Measurements of Coherent Phonons at a Buried Interface

S. H. Lee[1], D. M. Fritz[1], A. L. Cavalieri[1], Y. Sheu[1], M. C. Swan[1], J. Walstrand[1], D. A. Reis[1], R. S. Hegde[2], M. Reason[2], R. S. Goldman[2], D. Walko[3], and E. Landhal[3]
[1] *FOCUS Center and Department of Physics, University of Michigan*
[2] *Department of Materials Scienece and Engineering, University of Michigan*
[3] *Advanced Photon Source, Argonne National Laboratory*

Thermal transport in nanoscale materials is a fundamental problem in condensed matter physics and is of great importance for development of practical devices. Of particular importance at small length scales are the interfaces between materials. Phonons transfer heat between materials and their scattering is particularly sensitive to the details of the interface. X-ray techniques are able to yield quantitative information about the atomic positions, and time-resolved x-ray diffraction (TRXD) has proven a versatile probe of coherent acoustic phonons on picosecond time-scales [1]. Here, we present novel experiments on the transport of a coherent acoustic wave-packet across the nearly ideal AlGaAs/GaAs interface. A femtosecond laser impulsively excites the underlying substrate such that the coherent acoustic phonons take the form of two counter-propagating unipolar strain pulses. Picosecond TRXD is used to observe the laser-induced strain separately for the film and substrate. On the time-scale of these experiments, the strain in the film is purely elastic because the carriers are confined in the substrate and thermal diffusion is slow compared to the sound propagation time across the film. We observe the transmission and reflection of the acoustic pulse in the two extreme limits of a near perfect acoustic match between the film and the substrate and a near infinite acoustic mismatch between the film and the air. At low laser fluence, the x-ray data are in good agreement with numerical simulations of the strain generation, assuming instantaneous stress due to the volume deformation potential, and propagation assuming an acoustic mismatch model for the two interfaces. We find that excitation with higher pump-laser fluence leads to sudden changes in temporal and spatial profile of the strain. Here free carrier absorption (FCA) leads to saturation in the amplitudes of the strain pulses. At these levels Auger recombination following FCA introduces significant lattice heating in the GaAs substrate layer within a sub-nanosecond time scale. These experiments demonstrate that time-resolved x-ray diffraction can provide quantitative information on nanoscale thermal transport, as well as revealing much more detailed information than previously reported on the generation of the impulsive strain.

[1] S. H. Lee et al., *Phys. Rev. Lett*. **95**, 246104 (2005).

088

High-Frequency Domain-Wall Motion and Magnetization Rotation of Patterned Permalloy Films under External Magnetic Field Excitation

Syed Azeemuddin[1], P. Wang[1], A. Hoffmann[2], S.-H. Chung[2], R. Divan[3] A. Hoffmann[3], S.-H. Chung[3], and M. J. Donahue[4]
[1] *Department of Electrical and Computer Engineering, Southern Illinois University Carbondale, IL 62901*
[2] *Material Science Division, Argonne National Laboratory, Argonne, IL 60439*
[3] *Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439*
[4] *Mathematical & Computational Sciences Division, National Institute of Standards and Technology, Gaithersburg, MD 20899*

The incorporation of ferromagnetic materials into integrated microwave devices is a promising approach for the development of on-chip high-performance circuit components. Therefore, high-frequency domain-wall motion and magnetization rotation, which yield permeability, are of primary interest. However, so far physical separation of high-frequency domain-wall motion and magnetization rotation that are under high-frequency magnetic field excitation has not been attempted. Nor have there been attempts at the correspond-

ing characterizations. In this work, patterned permalloy films are integrated with on-chip transmission lines. Domain-wall motion and magnetization rotation are separated through aspect ratio and dimension control. The measured results show that high-frequency field-driven domain-wall motion is fast, different from current-driven domain-wall motion. It is also shown that coupling effects are not important when the distance between two adjacent permalloy films is ~1 μm despite their large lateral dimensions. The experimental results agree with simulation results. High-frequency field-driven domain-wall motion can follow external magnetic field up to 5 GHz even though the associated loss began to increase at ~ 2 GHz. Magnetization rotation, which is believed to be a faster process, shows lower operating frequency range. Obvious ferromagnetic resonance peaks are not observed. Magnetization at both ends of the "single domain" structures may have played a role in the observed characteristics.

089

Upgrade of IMCA-CAT Bending Magnet Beamline 17-BM for Macromolecular Crystallography at the Advanced Photon Source

I. Koshelev[1], R. Huang[1], T. Graber[2], M. Meron[2], J. L. Muir[1], W. Lavender[3], K. Battaile[1], A. M. Mulichak[1], and L. J. Keefe[1]
[1] IMCA-CAT, The Center for Advanced Radiation Sources, University of Chicago, Chicago, Illinois 60637
[2] ChemMatCARS, The Center for Advanced Radiation Sources, University of Chicago, Chicago, Illinois 60637
[3] Biological, Chemical, and Physical Sciences Department,Illinois Institute of Technology,Chicago, Illinois 60616

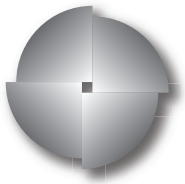
Macromolecular crystallography provides essential information for pharmaceutical industry research in drug design and development. It is critical to know the phases of the x-rays scattered by a protein crystal to extort the *de novo*, three-dimensional atomic structure of a protein from crystallographic data. A common way to obtain experimental phases is to conduct a multi-wavelength anomalous dispersion (MAD) experiment. Due to its source, high-beam-divergence bending magnet radiation provides significant challenges for collecting good quality MAD phasing data at a beamline with large acceptance angles. Dedicated to macromolecular crystallography, the IMCA-CAT bending magnet beamline was upgraded to provide the energy resolution necessary for successful MAD data collection on protein crystals. A collimating mirror was installed upstream of a double-crystal monochromator, helping to achieve the energy resolution suitable for MAD data collection without sacrificing the flux delivered onto the sample. The beam is focused sagittally and meridionally, to a beam size of 160 μm (V) x 230 μm (H), a flux density of 4 E^{+12} photons/sec/mm^2 at 12.398 keV, and energy resolution of $\delta E/E = 1.45\ E^{-4}$ at 10keV. In its operation range of 7.5-17.5 keV, the beamline delivers beam to the sample, stable to 10-20 μm with an energy reproducibility of 0.2 eV.

090

X-ray PEEM Study of Vortex Dynamics in Ferromagnetic Nanodots

K. Yu. Guslienko[1], X. F. Han[2], D. J. Keavney[2], R. Divan[2], and S. D. Bader[1]
[1] Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
[2] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

We report direct imaging by means of x-ray photoemission electron microscopy of the dynamics of magnetic vortices confined in micron-sized circular permalloy dots that are 30-nm thick. The vortex core positions oscillate on a 10-ns timescale in a self-induced magnetostatic potential well after the in-plane magnetic field is turned off. The observed vortex core position oscillations are interpreted as the translational mode of the magnetic vortex motion about the equilibrium position induced by a gyroforce and a dynamic magnetostatic restoring force. The measured oscillation frequencies as a function of the aspect ratio (thickness/radius) of the dots are in agreement with theoretical calculations presented for the same geometry.



091

Ultrafast Lattice Dynamics of FeRh Films

D. A. Walko[1], E. C. Landahl[1], J. Wang[1], J.-U. Thiele[2], and E. E. Fullerton[2]
[1] *Argonne National Laboratory*
[2] *Hitachi Global Storage Technologies*

FeRh undergoes magnetic and structural phase transitions at ~100°C where a transition from antiferromagnetic to ferromagnetic ordering occurs upon heating. Commensurate with this magnetic transition is a ~1% expansion in the lattice parameter. Recent optical measurements have shown that the magnetic transition can be on picosecond time scales. We have used ultrafast x-ray diffraction techniques at the Advanced Photon Source to probe the speed of the corresponding structural transition. An epitaxial FeRh thin film on a MgO(001) substrate was driven through the phase transition by ultrafast laser excitation, and the response of the lattice was directly observed via time-resolved x-ray diffraction. The temporal evolution of the FeRh lattice is reported as a function of laser fluence.

092

Magnetic Soliton Pair Dynamics

K. S. Buchanan[1], P. Roy[1], M. Grimsditch[2], F. Y. Fradin[2], K. Yu. Guslienko[2], J. Pearson[2], R. Divan[2], V. Novosad[2], and S. D. Bader[2]
[1] *Uppsala University, Sweden*
[2] *Materials Science Division and Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439*

We have explored the novel dynamical behavior of coupled pairs of magnetic vortices trapped within small ferromagnetic ellipses. The magnetic ellipses were patterned via electron-beam lithography directly on top of a microwave guide used to detect the magnetic resonant frequencies associated with the collective motion of the spin system. While the spiral motion associated with a single magnetic vortex had been studied previously, we added a new level of the complexity to the system by introducing a second vortex into the confined structure. This enabled us to observe for the first time new excitations that arise due to the magnetostatic interactions between trapped vortices [1]. Four different modes we modeled that correspond to different combinations of in-phase and out-of-phase vortex core motion along these axes, three of which can be excited by the spatially uniform magnetic fields that we applied experimentally. One observed mode involves core polarizations that are parallel to each other, while the other two observable modes correspond to excitations of vortex pairs with antiparallel polarizations. The key feature we revealed is that the relative core polarizations (parallel vs. anti-parallel) determine the dynamical response. This is especially surprising since the relative core polarizations are unimportant in determining the static properties of the system, such as, the energy of the equilibrium, ground-state configuration.

[1] K.S Buchanan, P. E. Roy, M. Grimsditch, F. Y. Fradin, K. Yu. Guslienko, S. D Bader, and V. Novosad, *Nature Physics* **1** (3): 172-176 (2005).

093

Dynamic Origin of Stripe Domains

M. Yan[1], G. Leaf[1], H. Kaper[1], V. Novosad[2], P. Vavassori[3], R. E. Camley[4], and M. Grimsditch[2]
[1] *Mathematics and Computer Science Division, Argonne National Laboratory, Argonne, IL 60439*
[2] *Materials Science Division, Argonne National Laboratory, Argonne, IL 60439*
[3] *Dipartamento di Fisica, Universita di Ferrara, Via Paradiso 12, I-44100 Ferrara, Italy*
[4] *Physics Department, University of Colorado, Colorado Springs, CO 80918*

A theoretical approach based on micromagnetic simulations is developed to calculate magnetic normal modes in confined magnetic structures. We apply this method to study the relation between stripe domains at remanence and magnetic normal modes in a single crystal Co bar. We find different stripe patterns depending on field history and in each case the domain structure can be related to a soft mode that triggers

a phase transition. The stripe domain structure when the external field is along the long axis of the bar is shown to be generated by a standing wave mode, which has the same spatial structure as the stripes. At all fields this mode has the lowest frequency of all the standing wave modes. This mode goes soft at a second order phase transition where the stripe domains emerge. For other directions of the field, the symmetry of soft modes is found to be consistent with the change in symmetry of the ground state and that the phase transition can be first order.

094

Hard X-ray Nanoprobe

R.P. Winarski, J. Maser, M. Holt, G. B. Stephenson, D. Shu, C. Benson, G. Wiemerslage, and D. Carbaugh
Argonne National Laboratory

The Center for Nanoscale Material's Hard X-ray Nanoprobe beamline at sector 26 of the Advanced Photon Source will explore nanoscale objects at spatial resolutions of 30 nanometers or smaller, using x-ray fluorescence spectroscopy, transmission imaging, diffraction, and scattering. X-ray fluorescence measurements will provide element-specific imaging of individual nanoparticles inside of samples. Transmission imaging will allow three-dimensional mappings of thick specimens and devices. X-ray diffraction and scattering capabilities will examine strain states and ordering in nanoscale systems. The beamline will provide x-rays with photon energies between 3 keV and 30 keV using two collinear insertion devices to maximize the coherent x-ray flux for nanoprobe experiments. The beamline is designed for two modes of operation: a scanning probe mode, where the spatially coherent fraction of the x-ray beam is focused by high-resolution x-ray optics onto a small area of a sample, and a full field transmission mode, where the full, partially coherent x-ray beam is used to illuminate a sample for transmission imaging at high resolution.

095

Measurement of Atomic Displacements in Binary Alloys with Size Mismatch

Y. S. Puzyrev and G. E. Ice
Oak Ridge National Laboratory

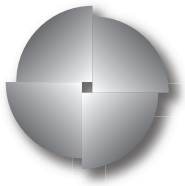
We obtain the diffuse scattering intensity for a copper-gold alloy, which has a pronounced size effect. The recovery of static displacements using the Ice-Sparks procedure is obscured by large thermal diffuse scattering. It cannot be removed experimentally due to large difference in atomic number. Molecular dynamics methods provide both static and thermal displacements along with diffuse scattering intensity, which can be directly compared with experimental results

096

Measurement of Atomic Displacements in Binary Alloys with Size Mismatch

Y.S. Puzyrev and G.E. Ice
Oak Ridge National Laboratory

We obtain the diffuse scattering intensity for copper-gold alloy, which has a pronounced size effect. The recovery of static displacements using the Ice-Sparks procedure is obscured by large thermal diffuse scattering. It cannot be removed experimentally because of the large difference in atomic number. Molecular dynamics methods provide both static and thermal displacements along with diffuse scattering intensity, which can be directly compared with experimental results.



097

Probing the Mechanism of a C-C Bond Hydrolase by Crystallographic Studies

Shiva Bhowmik[1], Geoff P. Horsman[2], Jiyuan Ke[3], Shaodong Dai[4], Lindsay D. Eltis[2], and Jeffrey T. Bolin[1]
[1] *Department of Biological Sciences, Purdue University, West Lafayette, IN, USA*
[2] *Department of Biochemistry and Molecular Biology, University of British Columbia, Vancouver, BC, Canada*
[3] *Center On Aging Foundation, University of Kentucky, Lexington, KY, USA*
[4] *Integrated Department of Immunology, National Jewish Medical and Research Center, Denver, CO, USA*

2-Hydroxy-6-oxo-6-phenylhexa-2,4-dienoate (HOPDA) hydrolase (BphD) is a key enzyme in the bph pathway. This pathway may be employed for aerobic transformation of polychlorinated biphenyls (PCBs), a common environmental pollutant. However, the failure of the pathway to efficiently process some PCBs and PCB-metabolites limits strategies for bioremediation.

$BphD_{LB400}$ (BphD from *Burkholderia* strain LB400), is a serine hydrolase, and catalysis involves the participation of a triad S112-H265-D237, typical of α/β hydrolases. Crystal structures of $BphD_{LB400}$, its S112A mutant with and without substrate, and with a recalcitrant PCB-metabolite (3-Cl-HOPDA), have been determined at 1.8Å resolution, providing significant insights into the mechanism and inhibition.

Consistence with the kinetic data, crystal structures of BphD S112A:HOPDA complex trapped the substrate as a keto isomer and revealed the involvement of H265 in tautomerization, a key step in the mechanism. Moreover, superimposing this structure onto wild-type BphD structure showed no room for water between S112 and C6 of HOPDA, suggesting a serine-nucleophile mechanism. Identification of the nucleophile has been a focus point of biochemical studies.

The crystal structure of BphD S112A:3-Cl-HOPDA showed the PCB bound in enol form. A plausible explanation is the presence of the 3-Cl substituent limiting the accessibility of the C2–OH of 3-Cl-HOPDA to H265, preventing its tautomerization and subsequent steps of the mechanism.

098

Growth Transient Scaling during Pulsed Laser Deposition

J.Z. Tischler[1], Gyula Eres[1], Christopher M. Rouleau[1], and B.C. Larson[1], and P. Zschack[2]
[1] *Materials Science & Technology Division, Oak Ridge National Laboratory*
[2] *UNI-CAT & Materials Research Laboratory, UIUC*

Time-resolved surface diffraction was used to study the growth kinetics during pulsed laser deposition of $SrTiO_3$. Time-dependent scattering at the (0 0 1/2) position along the crystal truncation rod appears nearly self-similar for dwell times between laser pulses ranging over a factor of 250 (0.2s to 50s). AFM measurements done before and after growth show that the growth always proceeds with no measurable increase of the surface roughness, indicating nearly ideal layer-by-layer growth. These results imply that the transverse length scale of surface structures in layer-by-layer growth is determined by the annealing time between laser pulses as well as the amount of material deposited per pulse. Analysis of the time-dependent intensities shows that extremely fast nonequilibrium interlayer transport, which occurs concurrently with the arrival of the laser plume, dominates the deposition process. A much smaller fraction of material, which is governed by the dwell time between successive laser shots, is transfered by slow, thermally driven interlayer transport processes.

Research sponsored by the Division of Material Sciences and Engineering, Office of Basic Energy Sciences, U.S. DOE, contract DE-AC05-00)R22725 with Oak Ridge National Laboratory, operated by UT-Battelle, LLC. The APS supported by the DOE under Award No. DEFG02-91ER45439. UNICAT supported by ORNL, UIUC, NIST, and UOP.

099

Patterned Superconducting Micro-Bolometers for Array Detectors

V. Yefremenko[1], V. Novosad[1], R. Divan[2], J. Pearson[1], and S. D. Bader[1]
[1]Materials Science Division and [2]Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439 USA

One of the most sensitive classes of detectors in the far-infrared spectral range are superconducting bolometers. A problem associated with the creation of large, low-temperature arrays is the conjugation of the detectors with the multiplexer, because conventional read-out integrated circuit electronics have limited functionality at low temperatures and cannot be integrated with superconducting materials without affecting their properties. A new optically multiplexed superconducting microbolometer array will be presented. This approach allows single detectors to be connected in series, and, as a result, an image can be formed by registering the signal with only two electrical terminals.

Epitaxial Y-Ba-Cu-O films (200 nm thickness) were used for fabricating arrays (1×10 and 32×32 pixels) to visualize an infrared point source. Spatial distribution of the array sensitivity was investigated. Operating detectors at low temperatures provides the notable advantages of lower noise and better sensitivity. The proximity effect in superconductors was used to optimize the working temperature of the transition edge sensors (TES). A bilayer film of Mo/Au (30 nm/50 nm thickness) was found to provide the operation of detectors in the temperature interval 0.4–0.6K. A TES microthermometer (50×50 μm^2) was fabricated. The electron beam lithography and wet chemical etching were carried out at Argonne's Center for Nanoscale Materials and applied via pattern transfer processes for both Y-Ba-Cu-O and Mo/Au detectors.

100

A Small Survey of Vibration Measurement and Analysis Techniques

Curt Preissner and Deming Shu
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA

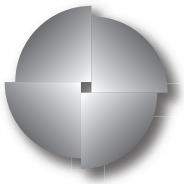
Low-level vibration measurement and analysis are becoming more important in the operation and servicing of x-ray beamlines. State-of-the-art x-ray optics are routinely producing effective beam spot sizes of a few tens of nanometers, placing extreme requirements on the beamline support structures. As the performance of x-ray optics has increased, demand for vibration measurements and data analyses has also increased. This poster reviews some APS engineers' experience in vibration measurement and diagnoses of x-ray beamline equipment, particularly with low-level vibrations. Areas to be addressed include low-level measurements, transducer selection, transducer mounting, signal conditioning, and data acquisition. Specific measurement case studies will include a monochromator and an instrument support structure.

101

X-ray Scattering Studies of Silica Nanoparticles Distributed on Liposome Vesicle Surface

Mrinmay K. Mukhopadhyay[1], Laurence Lurio[2], Lyle Marchande[2], Liangfang Zhang[3], and Steve Granick[3]
[1] Department of Physics, University of California San Diego, La Jolla, CA 92093 USA
[2] Department of Physics, Northern Illinois University, DeKalb, IL 60115 USA
[3] Materials Research Laboratory and Department of Chemical and Biomolecular Engineering, University of Illinois, Urbana, IL 61801 USA

Small-angle x-ray scattering measurements have been performed on a solution of spherical liposome nanoparticles (~100 nm radii) stabilized using silica nanoparticles (~5 nm radii) distributed on the surface. The measurements were done at the sector 8 beamline of the Advanced Photon Source. The experimental results indicate the presence of distributed silica nanoparticles on the liposome vesicle surface. We have simulated the structure factor for the various distributions of the silica nanoparticles on the surface of liposome vesicles and a comparison of the distribution with simulations will be presented. X-ray photon



correlation spectroscopy measurements were also performed to study the both the diffusion of the liposomes through solution and the migration of the nanoparticles along the liposome surface. The understanding of the distribution of nanoparticles on the surface of the sphere and their migration dynamics on the surface will provide important insight on the properties of particles embedded in vesicle walls and may serve as a model system to understand cell-wall properties in living organisms.

102

In Situ SAXS Observations on Deformations of Unoriented Styrene-Butadiene-Styrene Triblock Copolymer Grafted with Polyhedral Oligomeric Silsesquioxanes

Daniel B. Drazkowski and Andre Le
Department of Chemical Engineering & Materials Science, Michigan State University

A series of copolymers of polystyrene-polybutadiene-polystyrene (SBS) grafted with polyhedral oligomeric silsesquioxane (POSS) molecules were synthesized to examine the effects of nanostructure chemistry on the deformation of unoriented, nanostructured block copolymers. Two electronically different chemical substituents, isobutyl (iBt) and phenyl (ph), were grafted up to 20 wt% by a hydrosilation method. At 30°C, it was observed that the mechanical response of the two POSS-copolymer systems was similar; however, the morphological deformation dynamics differed considerably, as shown by SAXS. The Bt-POSS showed similar deformation dynamics to the pure SBS because of its compatibility with the polybutadiene phase to which it is grafted. The ph-POSS, because of its greater affinity toward polystyrene, effectively restricts the morphological deformation and orientation. At higher temperatures, the Bt-POSS system begins to diverge from the pure SBS by slightly retarding morphological orientation, and there is a greater overall difference from the POSS copolymer systems and pure SBS.

103

Light-Induced Time-Resolved X-ray Absorption as a Spectroscopic Probe of Chemical Transformations

Erik Wasinger[1], Xiaoyi Zhang[2], Klaus Attenkofer[2], Guy Jennings[2], and Lin X. Chen[3]
[1]Chemistry Division, [2]Advanced Photon Source, [3]Chemistry Division, Argonne National Laboratory, Argonne, IL 60439 USA

Determining the geometric and structural changes throughout the time course of a reaction plays a vital role in understanding chemical mechanisms. Toward this end, light-induced time-resolved x-ray absorption spectroscopy (LITR-XAS) has been shown to be capable of examining the structural changes accompanying electronic excitation on a picosecond time scale. Following the success of this technique in studying photochemical complexes, it is now being applied to systems relevant to homogeneous and biological catalysis. Recent work on exemplary model systems such as the Zn(bis-dipirine) and Fe(pacman) systems is discussed.

104

X-ray Microprobe of Atoms in Strong Optical Fields

L. Young, D. A. Arms, E. M. Dufresne, R. W. Dunford, D. L. Ederer, C. Höhr, E. P. Kanter, B. Krässig, E. C. Landahl, E. R. Peterson, N. Rohringer, J. Rudati, R. Santra, and S. H. Southworth
Argonne National Laboratory, Argonne, IL 60439 USA

We have developed a microfocused, tunable, polarized, time-resolved x-ray probe of atoms and molecules in the presence of an intense optical laser field. The experiments are performed at the APS sector 7 undulator beamline in combination with a Ti:sapphire ultrafast laser system that is phase-locked to the rf clock of the storage ring. The laser and x-ray pulses are focused and overlapped in time and space through a gas-phase target within a vacuum chamber, and the emitted x-ray fluorescence, ions, and electrons are detected.

The first experiments involve single- and double-ionization of one or two 4*p* electrons of atomic krypton by the strong field of the laser (1.3 mJ, 50 fs, 100 μm focal spot, 1 kHz, 4 x 10^{14} W/cm^2 peak intensity). The K-edge excitation spectra of Kr^+ and Kr^{2+} near 14.3 keV are recorded using tunable x-ray pulses (10^6 x-rays, 1.5 eV band width, 100 ps, 272 kHz) focused to 10 μm using Kirkpatrick-Baez mirrors. Strong 1*s* to 4*p* resonances are observed that are absent in the K-edge spectrum of neutral Kr because its 4*p* shell is fully occupied. By tuning the x-ray energy to the 1*s* to 4*p* resonance we can monitor the ion density as a function of time delay between laser and x-ray pulses and of spatial separation between the two beams. We also measure a factor of two increase in the fluorescence yield at the 4*p* resonance as the angle between the linear-polarization directions of the laser and x-ray beams is varied from perpendicular to parallel. This effect is attributed to alignment (unequally populated magnetic sublevels) of the Kr^+ $4p_{3/2}$ hole state produced by laser ionization.

105

Measurement of the APS Bunch Length Using an Atomic Transition

E. C. Landahl, D. A. Arms, E. M. Dufresne, R. W. Dunford, D. L. Ederer, C. Höhr, E. P. Kanter, B. Krässig, E. R. Peterson, N. Rohringer, J. Rudati, R. Santra, S. H. Southworth, B. X. Yang, and L. Young
Argonne National Laboratory, Argonne, IL 60439 USA

The duration of synchrotron x-ray pulses at the Advanced Photon Source has been measured over an order of magnitude in single-bunch charge using time-dependent modifications to the near threshold absorption spectrum of krypton gas caused by an amplified ultrafast laser pulse. At a laser intensity of ~2 x 10^{14} W/cm^2, krypton single-ionization is saturated, and a prominent resonance (the 1*s* to 4*p* transition) appears in the x-ray absorption spectrum. This process is prompt, so that time resolution is limited only by laser duration and synchronization. The ions remain within the laser focus for several nanoseconds and a complete cross-correlation is obtained by sweeping the laser-produced ionization front across the 100 ps x-ray bunch. Results are compared with single-shot optical streak camera measurements of visible radiation produced by the same electron bunch in the storage ring. This technique will be applied to achieving overlap between laser and x-ray pulses in future gas-phase experiments as well as for characterizing temporally complex x-ray pulses produced at fourth-generation and upgraded third-generation light sources.

106

Effect of Valence on Antimony Uptake by *Leishmania*

Ann LeFurgey[1], Stefan Vogt[2], Barry Lai[2], and Peter Ingram[3]
[1] *Veterans Affairs and Duke University Medical Centers, Durham, NC 27705 USA*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA*
[3] *Duke University Medical Center, Durham, NC 27710 USA*

Although new treatments have been developed for visceral leishmaniasis, antimony-containing drugs remain the most effective treatment for the cutaneous lesions caused by the parasitic protozoan *Leishmania*. Antimony in these drugs has a valence of 5+, whereas its toxicity to the parasite is caused by Sb in a valence state of 3+, requiring cellular conversion. We tested the effects of valence on the quantitative uptake of antimony by *Leishmania* amastigotes in vitro. Cells were exposed to Sb^{3+}, Sb^{5+} or both Sb^{3+} and Sb^{5+}, and individual cell Sb content was determined by x-ray probe x-ray microanalysis (XPXMA) using APS beamline 2-ID-D. Average Sb cell content was highest in cells exposed to Sb^{5+} alone; cell content was significantly less with either Sb^{3+} or Sb^{3+} combined with Sb^{5+}. Uptake was heterogeneous from cell to cell

following Sb^{5+} exposure, with approximately 60% of cells containing >0.05 μg Sb/cm^2 and the remaining 40% of cells containing <0.01 μg Sb/cm^2. This bimodal distribution in content was not observed with Sb^{3+}, in which Sb content exhibited a normal distribution among cells, or with Sb^{5+}, which exhibited a multimodal distribution. These results are biologically relevant in that they demonstrate varied individual cell sequestration and thus susceptibility to antimony of differing valence states.

VAMC studies were supported by a VA Merit Award to AL. Work at Argonne was supported by U.S. Department of Energy, Office of Basic Energy Sciences, Contract No. W-31-109-ENG-38.

107

Structure and Dynamics of Attractive Colloidal Glasses

Xinhui Lu[1], Simon Mochrie[1], Suresh Narayanan[2], Alec Sandy[2], and Michael Sprung[2]
[1] *Physics Department, Yale University, New Haven, CT 06511 USA*
[2] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA*

Even after decades of study, understanding the glass transition remains a grand challenge for condensed matter science. Beyond molecular glass-formers, it is also possible for dense colloidal suspensions to show a glass transition, The glass transition of hard sphere colloids has been extensively studied over the last 15 years. More recently, however, the character of the glass transition in systems with attraction have become the subject of theoretical and experimental interest. One way to realize colloids with attractive interactions is to create a suspension in a near-critical binary fluid mixture. We will use XPCS (x-ray photon correlation spectroscopy), to determine the static structure and dynamics near the glass transition of such a system.

108

BioCARS: A Facility for Macromolecular Crystallography at the Advanced Photon Source

R. W. Henning, V. Srajer, R. Pahl, T. Graber, S. Anderson, S. Ruan, G. Macha, J. VonOsinski, M. Bolbat, H. Brewer, N. Lei, H. Tong, and K. Moffat
Consortium for Advanced Radiation Sources, The University of Chicago, Chicago, IL

The BioCARS facility is a part of the multi-disciplinary and multi-institutional Consortium for Advanced Radiation Sources (CARS) managed by the University of Chicago. CARS has designed, constructed, and operates the experimental facilities at the Advanced Photon Source (APS) as national synchrotron resources available to the scientific community. BioCARS consists of two beamlines: an insertion device beamline, 14-ID, with an undulator and one experimental station (14-ID-B), and a bending magnet beamline, 14-BM, with two stations that are used simultaneously (14-BM-C and 14-BM-D). BioCARS provides state-of-the-art facilities and scientific and technical support for studies of macromolecular assemblies that form crystals with large unit cells, MAD phasing, high-resolution crystallography, Laue crystallography, and time-resolved crystallography. BioCARS is also the only facility at the APS that has been approved for safe work on biosafety level 3 samples such as pathogenic human viruses. Beamtime is allocated through peer review of proposals. To apply for beamtime or for more information about the BioCARS facility, visit http://biocars.org.

109 – Withdrawn

110

Novel Growth and Coarsening Behavior of Nanocrystals Arising from Quantum Size Effects: Pb on Si(111)7x7 Surface

C. A. Jeffrey[1], P. F. Miceli[1], K. Roberts[1], S. Hayden[1], M. Gramlich[1], E. H. Conrad[2], R. Feng[2], M. Hupalo[3], M. C. Tringides[3], C. Kim[4], and P. J. Ryan[5]
[1] *Dept. of Physics and Astronomy, University of Missouri-Columbia*
[2] *School of Physics, Georgia Institute of Technology*
[3] *Ames Lab, Iowa State University*
[4] *Dept. of Physics and Research Inst. of Basic Sciences, Kyunghee University, Korea*
[5] *MU-CAT, Advanced Photon Source, Argonne National Laboratory*

The energetics of very thin metallic films can be affected by the quantum confinement of electrons and, thus, can lead to dramatic changes in surface morphology. Such quantum size effects are known to influence Pb deposited on the Si(111)7x7 surface where Pb nanocrystals form with "magic" island heights over a limited coverage and temperature range. Therefore, a question of broad fundamental importance is this: just how do atomic-scale mechanisms lead to this peculiar growth behavior?

We have explored this question by looking at the coarsening of the nanocrystals using in situ x-ray scattering. We find very unusual behavior. Ostwald ripening is not observed, an unexpectedly strong dependence on the deposition flux rate is found, and we measure decay times that are orders of magnitude faster than predicted. These can have unanticipated consequences. For example, lower island densities are achieved by using higher deposition fluxes. These results have important implications for understanding the controlled growth and self-organization of nanostructures.

The MU-CAT 6-ID-C beamline is supported through the Ames Lab by the US-DOE. Research funding is supported by NSF, PRF (PFM, CAJ), Ames Lab (MCT, MH), Canim Scientific (EHC), NSERC-Canada (CAJ), Ministry of Science and Technology–Korea (CK).

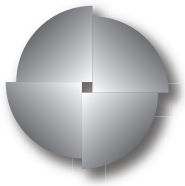
111

The Formation of a Sharp Metal-Semiconductor Interface for the Growth of Quantum Size Effect Islands

R. Feng[1], E. H. Conrad[1], C. A. Jeffrey[2], P. F. Miceli[2], K. Roberts[2], S. Hayden[2], M. Gramlich[2], M. Hupalo[3], M. C. Tringides[3], C. Kim[4], and P. J. Ryan[5]
[1] *School of Physics, Georgia Institute of Technology*
[2] *Dept. of Physics and Astronomy, University of Missouri-Columbia*
[3] *Ames Lab, Iowa State University*
[4] *Dept. of Physics and Research Inst. of Basic Sciences, Kyunghee University, Korea*
[5] *MU-CAT, Advanced Photon Source, Argonne National Laboratory*

In order to form quantum size effect (QSE) metal islands on semiconductors, a smooth island-substrate interface is necessary to set up the electron standing waves that lead to the new quantum confined states. How this occurs for the Pb-Si(111)7x7 system is a mystery because of the large lattice mismatch and the highly corrugated 7x7 reconstruction. To understand how QSE islands develop in this system, we have performed in situ surface x-ray scattering experiments on the initial formation of Pb islands grown on Si(111)7x7. We show that a smooth Pb-semiconductor interface develops through a series of structural arrangements. Once a vertically disordered Pb monolayer is completed, a second layer of Pb atoms begins to nucleate fcc Pb clusters. These clusters undergo a displacive transition that lifts them to sites above the Si adatoms. This displacement allows the Pb islands to "float" above the Si substrate so that the first island layer is smooth, thus setting up the proper boundary condition for QSE.

The MU-CAT 6-ID-C beamline is supported through the Ames Lab by the US-DOE. Research funding is supported by NSF, PRF (PFM, CAJ), Ames Lab (MCT, MH), Canim Scientific (EHC), NSERC-Canada (CAJ), Ministry of Science and Technology–Korea (CK).



112

X-ray Standing Wave Atomic Imaging of Platinum Nano-Crystals on $SrTiO_3$ (001)

Steve Christensen[1], Alexander Kazimirov[1,2], Tien-Lin Lee[1], David Marasco[1], Stephen Streiffer[3], Mark Hersam[1], and Michael Bedzyk[1,3]
[1] *Institute of Environmental Catalysis, Northwestern University*
[2] *CHESS, Cornell University*
[3] *Materials Science Division, Argonne National Laboratory*

Sub-monolayer coverages of Pt on (2x1) reconstructed $SrTiO_3$ (001) were characterized using x-ray standing waves (XSW), grazing-incidence small-angle x-ray scattering (GISAXS), and atomic force microscopy (AFM). The AFM and GISAXS studies revealed the formation of Pt nano-clusters. The summation of the XSW measured Fourier components for the Pt distribution revealed that the Pt atoms occupied lattice positions coherent to the underlying $SrTiO_3$ cubic lattice. Changes in the coverage and annealing conditions caused changes to the observed Pt atomic distribution. This system has relevance to photocatalysis and electronic device applications. The XSW measurements were conducted at APS 12-ID-D and the GISAXS at 12-ID-C.

113

Nonresonant Inelastic X-ray Scattering and Energy-Resolved Wannier Function Investigation of Local Excitations in NiO and CoO

B. C. Larson[1], Wei Ku[2], J. Z. Tischler[1], Chi-Cheng Lee[3], Oscar Restrepo[4], A. G. Eguiluz[1,4], P. Zschack[5], and K. D. Finkelstein[6]
[1] *Oak Ridge National Laboratory*
[2] *Brookhaven National Laboratory and SUNY, Stony Brook, NY*
[3] *Tamkang University, Taiwan*
[4] *University of Tennessee, Knoxville*
[5] *XOR/APS, Argonne National Laboratory*
[6] *CHESS, Cornell University, Ithaca, NY*

Nonresonant inelastic x-ray scattering (NIXS) and energy-resolved Wannier function analysis have been used to probe the strongly correlated electronic structure of NiO and CoO. NIXS measurements of the dynamical structure factor $s(\mathbf{q}, \omega)$ as a function of momentum transfer q and frequency omega have shown that dipole-forbidden, *d-d* excitations appear within the optical gap for large wavevectors ($q > 2/Å$), become the dominant structure in the loss spectra for $q > 3/Å$, and reach a maximum at $q \sim 7/Å$. In contrast to the loss-spectra observed in resonant-probe studies of NiO and CoO, nonresonant spectra show only two excitations that are highly anisotropic—strongest in the [111] direction and weakest (or missing) in the [001] direction. Energy-resolved Wannier function analyses of vertex matrix elements within LDA+*U* demonstrate that the anisotropy provides a sensitive measure of electronic symmetry-breaking in these atomic-like *d-d* excitations as a result of point-group symmetry selection rules.

Research sponsored by the DOE, Office of Science, DMS at ORNL managed by UT-Battelle, LLC, under contract DE-AC05-00OR22725. Work was performed on the XOR/UNI beamline at the APS, which is sponsored by the DOE.

114

The Effect of Fe(II)-Fe(II) Coordination on the Reduction of U(VI) Determined by Fe- and U-edge XAFS

Maxim I. Boyanov[1], Edward J. O'Loughlin[1], Shelly D. Kelly[1], Bruce D. Ravel[1], Eric E. Roden[2], Jeremy B. Fein[3], and Kenneth M. Kemner[1]
[1] *Argonne National Laboratory*
[2] *University of Wisconsin-Madison*
[3] *University of Notre Dame*

Fe(II) is a known reductant of groundwater contaminants such as Cr(VI) and pesticides. Efforts are currently underway to understand U(VI) reduction by Fe(II) to use bacterially produced Fe(II) for bioremediation. The U(VI)-Fe(II) redox reaction is kinetically inhibited in homogeneous solution. However, increased reduction rates are observed when a surface is present. To understand the role of the surface we have performed Fe K-edge and U L-edge x-ray absorption fine-structure (XAFS) experiments on an aqueous system containing Fe(II), U(VI), and a carboxyl-functionalized colloid. Under conditions for which Fe EXAFS data indicated only monomeric Fe(II) present in the system, U XAFS data indicated only U(VI) species that were inner-sphere complexed to the surface carboxyl groups. Under conditions for which Fe EXAFS data indicated Fe-Fe correlation, the U EXAFS data indicated complete reduction of U(VI) to U(IV) and the formation of uraninite nanoparticles. The U-Fe electron transfer was further confirmed by Fe XAFS, which indicated the oxidation of the Fe species. These results and the structure of the reduced U(IV) product will be discussed in the context of a proposed U(VI)-Fe(II) redox mechanism.

The XAFS experiments were performed at the MR-CAT beamline, sector 10-ID of the Advanced Photon Source, supported by DOE under contract DE-FG02-94-ER45525 and the member institutions. Work supported by the U.S. Department of Energy, Office of Science, Office of Biological and Environmental Research, Environmental Remediation Sciences Division, under contract W-31-109-Eng-38, and the Environmental Molecular Science Institute at University of Notre Dame (funded by NSF grant EAR-0221966).

115

X-ray Micro(spectro)scopy of Metal Transformations Inside and Near Single Bacterial Cells

Maxim I. Boyanov[1], Barry Lai[2], Susan Glasauer[3], Matthew J. Marshall[4], S. Langley[5], Alice C. Dohnalkova[4], James K. Fredrickson[4], T. J Beveridge[3], and Kenneth M. Kemner[1]
[1] *Bioscienes Division, Argonne National Laboratory*
[2] *Advanced Photon Source, Argonne National Laboratory*
[3] *University of Guelph, Guelph, Ontario N1G 2W1, Canada*
[4] *Pacific Northwest National Laboratory, Richland, Washington 99354, USA*
[5] *University of Ottawa, Ottawa, ON K1N 6N5, Canada*

The interactions of bacteria with the dissolved metals in their environment take place over the length scale of their size, usually one to several micrometers. The interplay between adsorption, reduction/oxidation, cross-membrane transfer, precipitation, and mineral dissolution can create several different species of an element in the vicinity of a single cell, making the system less amenable to characterization by bulk methods. Using the current capabilities of the XOR x-ray microscopy beamline (sector 2-ID-D at the Advanced Photon Source) this inhomogeniety can be addressed on the 150 nm lengthscale, and insight on the functioning of cells and their biogeochemical interactions can be obtained. We have combined the high spatial resolution of TEM imaging with the high elemental sensitivity of the x-ray microprobe to study products of the respiration of *Shewanella putrefaciens* CN32 and *Shewanella oneidensis* MR-1 on Fe(III) and U(VI), respectively.

Growth of *Shewanella putrefaciens* CN32 on solid-phase Fe(III) leads to the formation of abundant 40-nm-sized intracellular granules. Elemental maps of the cells showed the presence of typical cellular elements (P, Ca, Cl, S), together with high Fe concentrations at the locations of high electron density on the TEM micrographs. Fe K-edge XANES measurements on selected areas of the cells showed that the internal Fe precipi-

tates were more oxidized than Fe associated with other parts of the cell, but more reduced than magnetite. The valence state of extracellular precipitates in the vicinity of the cell was consistent with magnetite.

Growth of *Shewanella oneidensis* MR-1 in the presence of U(VI) leads to the precipitation of uniform uraninite nanoparticles along thin (<100 nm in diameter) fiber-like structures. The x-ray elemental analysis revealed co-localization of the uraninite particles with Fe and P, pointing to the bacterial origin of these structures. Heme staining with colloidal gold corroborated the finding and demonstrated for the first time extracellular localization of a decaheme cytochrome in direct association with UO_2 nanoparticles.

Work supported by the U.S. Department of Energy, Office of Science, Office of Biological and Environmental Research, Environmental Remediation Sciences Division, under contract W-31-109-Eng-38. Support for M.B. provided by the Environmental Molecular Science Institute at University of Notre Dame (funded by NSF grant EAR-0221966).

116

Studies of Heme Proteins by Time-Resolved Crystallography: Allosteric Action and Structural Relaxation

Vukica Šrajer[1], Reinhard Pahl[1], James Knapp[2], William Royer[2], and Marius Schmidt[3]
[1] *The University of Chicago, Chicago (IL), USA*
[2] *University of Massachusetts Medical School, Worchester (MA), USA*
[3] *Technische Universität München, Germany*

Time-resolved macromolecular crystallography is reaching a mature phase with demonstrated ability to detect small structural changes on nanosecond and sub-nanosecond time scale [1-6] and with important advances in the analysis of time-resolved crystallographic data, such as the use of the singular value decomposition (SVD) method to determine the structures of intermediates and elucidate the reaction mechanism [3-4]. We present results of nanosecond time-resolved crystallographic studies of heme proteins: allosteric action in real time in cooperative dimeric hemoglobin and structural relaxation processes in myoglobin [1]. These pump-probe experiments were carried out at the BioCARS beamline 14-ID at the Advanced Photon Source (USA).

[1] Schmidt et al. *PNAS* 102 (33) 11704-11709 (2005).

[2] Ihee et al. *PNAS* 102, 7145-7150 (2005).

[3] Rajagopal et al. *Structure* 13, 55-63 (2005).

[4] Schmidt et al. *PNAS* 101, 4799-4804 (2004).

[5] Schotte et al. *Science* 300, 1944 (2003).

[6] Šrajer et al. *Biochemistry* 40, 13802 (2001).

117

Nano- and Micro-Size Confinement of 2D Magnetic Stripe Domain System

Changyeon Won[1], Ralu Divan[2], Samuel Bader[1,2], and David Keavney[3]
[1] *Material Science Division,* [2] *Center for Nanoscale Materials, and* [3] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA*

When an ultra-thin Fe film (< 1 nm) is grown on a Cu substrate, Fe has face-centered-cubic (FCC) structure, although it has body-centered structure in the bulk phase. This FCC Fe is known to have out-of-plane magnetic anisotropy. Because of the competition between anisotropy energy and dipole energy, the film shows alternate magnetic domains, which are called stripe domains, and the width is a function of film thickness in the nanoscale range. As the film thickness increases, but still remaining within the few monolayer range, the magnetization direction of the film changes to in-plane because of the influence of the bulk shape anisotropy.

To see the effect of confinement on the stripe domain phase, nanostructure patterning with Fe thin film on Cu substrate is necessary. However, since metallic substrates usually are treated in ultrahigh vacuum with ion sputtering and high-temperature annealing to get a clean and flat surface, with Fe then epitaxially grown in the same system, a traditional lift-off lithography method cannot be applied. To overcome this difficulty, we did the deposition through a stencil mask made by nanopatterning with an e-beam writer of suspended low-stress silicon nitride membranes and subsequent etching with a reactive ion etching system.

Details about stencil mask fabrication and first results for 2D magnetic systems are presented.

Part of this work was carried out at the Center for Nanoscale Material and the Advanced Photon Source, Argonne National Laboratory, supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.

118

Electron Hopping in Pi-Stacked Covalent and Self-Assembled Perylene Diimides Observed by ENDOR Spectroscopy

Richard F. Kelley, Michael J. Tauber, Jovan M. Giaimo, Boris Rybtchinski, and Michael R. Wasielewski
Department of Chemistry and Center for Nanofabrication and Molecular Self-Assembly, Northwestern University, Evanston, IL 60208 USA

We have carried out room-temperature, solution-phase electron paramagnetic resonance (EPR) and electron-nuclear double resonance (ENDOR) studies on a series of radical anions based upon perylene-3,4:9,10-bis(dicarboximide) (PDI). The following systems were studied: two PDI monomers; a covalent, cofacial dimer; and two covalent trefoil-PDI3 molecules, one of which self-assembles into pi-stacked dimers as determined by small-angle x-ray scattering (SAXS). Full sharing of the unpaired electron in the covalent and self-assembled dimers is revealed by a halving of the hyperfine coupling constants in these species, relative to those of the monomers. These results and the electronic absorption spectra show that electron hopping on a timescale greater than 10^7 Hz occurs between a reduced and neutral chromophoric pair.

119

Near-Field Coherent Diffraction Imaging (NFCDI) of Electro-Deposited Lead Microparticles

Martin D. de Jonge, Xianghui Xiao, Yong S. Chu, and Qun Shen
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA

Recent developments in coherent diffraction imaging have demonstrated full-field reconstructions of several-micron samples with resolution down to 10 nm. However, there is significant debate about the optimum geometry for the measurement. Other developments have modified the technique in an attempt to resolve ambiguities arising from the reconstruction algorithm resulting, for example, from the twin-image problem. We have recently implemented a technique for overcoming the twin-image problem, in which the coherent scattering pattern is recorded in the near-field, as suggested by Xiao and Shen.

We report recent measurements of lead microparticles electro-deposited onto a silicon-nitride membrane. The measurements were made using a highly coherent beam of 8-keV x-rays at 32-ID of the APS. A pinhole was used to define an illumination region on the specimen. Further pinholes were used to remove high-order Airy rings from the illuminating beam. Images were recorded in the near-field, with a Fresnel number of around 1. Preliminary analysis suggests that we may be able to reconstruct the structure of these particles to 100 nm – 200 nm resolution.

Use of the Advanced Photon Source 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.

120

X-ray Laue Microdiffraction Studies of Individual ZnO Nanostructures

J. D. Budai[1], W. Yang[2], W. Liu[2], J. Z. Tischler[1], B.C. Larson[1], Z. Pan[1], Y. W. Heo[3], and D. N. Norton[3]
[1] *Oak Ridge National Laboratory, Oak Ridge, TN*
[2] *UNI-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL*
[3] *University of Florida, Gainesville, FL*

Various approaches for characterizing individual nanostructures using synchrotron microbeams are currently being developed. At sector 34 of the APS, we have used polychromatic Laue microdiffraction and microfluorescence to probe the local crystal structure and perfection of various forms of ZnO meso- and nano-structures. With Kirkpatrick-Baez mirrors for achromatic focusing, we use a scanning x-ray technique for mapping the spatially resolved lattice structure, composition, and strain. Off-axis undulator radiation is routinely focused to ~0.5 micron and beams as small as 90 nm have been demonstrated. Laue diffraction patterns are collected using a CCD area detector, and computer analysis yields spatial maps of the crystal phase, grain orientations (texture), and strain tensor. An important feature of the white-beam approach is the ability to collect a full diffraction pattern without rotating the sample. We have mapped the structure of individual ZnO structures using samples fabricated in different shapes: rods, belts, and tapered styluses. TEM samples serve as ideal x-ray microdiffraction samples because of the low background signal. Even when mounted on a relatively thick crystal substrate, full diffraction patterns can be measured from ZnO rods as narrow as 200 nm diameter. We find that all of the ZnO structures have a faceted, hexagonal crystal structure, with the c-axis often along the rod axis. Larger diameter rods are essentially perfect single crystals, whereas thinner rods have a high degree of flexibility, and consequently show large local mosaic bending. For tapered stylus samples, the regions where the diameter decreases remain a single crystal.

Support by DOE Office of Science, Division of Materials Sciences under contract with ORNL, managed by UT-Battelle, LLC; UNI-CAT/XOR supported by ORNL, UIUC-MRL, NIST, and UOP LLC/DOE-BES; APS supported by DOE.

121

COBRA Studies of Ultrathin Ferroelectric Oxide Films

C. Cionca[1], D.-P. Kumah[1], R. Clarke[1], C-B. Eom[2], D. A. Walko[3], and Y. Yacoby[4]
[1] *Applied Physics, University of Michigan, Ann Arbor*
[2] *Materials Science and Engineering, University of Wisconsin-Madison*
[3] *Advanced Photon Source, Argonne National Laboratory*
[4] *Racah Institute of Physics, Hebrew University, Jerusalem, Israel*

The study attempts to bring an understanding to what structural distortions are responsible for the ferroelectric domain formation in ultrathin perovskite ferroelectric films. We used coherent Bragg rod analysis (COBRA) and x-ray synchrotron radiation to investigate the structure of epitaxial thin films of $PbTiO_3$ deposited by sputtering on $SrTiO_3$ substrates. The measurements were performed under different electrical boundary conditions, including the presence of conducting electrodes. The data analysis reveals details of the atomic displacements in different layers and shows subtle variations as a function of distance from the interface. Diffuse scattering maps were used to extract information about on the ferroelectric domain landscape, and they were correlated with the distortions revealed by COBRA.

This work was conducted at the MHATT-CAT insertion device beamline at the Advanced Photon Source and was supported in part by the U.S. Department of Energy, Grants No. DE-FG02-03ER46023

122

Benefits of Aberration Correction for Elemental Mapping

Bernd Kabius[1], N. Zaluzec[1], W. Fan[1], O. Auciello[1], C. Hetherington[2], and A. Kirkland[2]
[1] *Materials Science Division, Argonne National Laboratory*
[2] *University of Oxford, UK*

Energy-filtering transmission electron microscopy (EFTEM) has been frequently applied to material science problems because of its capabilities for elemental mapping of large sample areas. The spatial resolution of these elemental maps is limited by lens aberrations. It has been demonstrated that Cs correction improves the analytical resolution in EFTEM mode by a factor of about two and allows fast elemental mapping of large fields of view with resolution a spatial resolution of 0.3 nm.

123

Assignment of Chiral Indices of Boron Nitride Nanotubes by Electron Diffraction

R. Arenal[1], M. Kociak [2], A. Loiseau[3], and D.J. Miller[1]
[1] *EMC group, MSD Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *LPS, University Paris-Sud and CNRS, 91405 Orsay, France*
[3] *LEM, UMR 104 CNRS-ONERA, 92322 Chatillon, France*

Boron nitride nanotubes (BNNTs) are an emerging nanomaterial with properties that complement the well-known carbon nanotubes. BNNTs are insulators with a constant band gap > 5.5 eV, independent of their helicity and diameters [1]. Detailed knowledge of the atomic structure of these nanotubes (chiral angle and diameter) is fundamental to understand their growth mechanism. The most reliable technique for obtaining this information from the nanotubes is electron diffraction (ED). In this communication, we show for the first time the ED patterns of a single- and double-walled BNNT, providing us rich information concerning their atomic structure. The intensities of ED patterns from individuals BNNTs, as well as from bundles of these tubes have been recorded using a nanometer-sized coherent electron beam in the nano-area ED geometry [2]. To identify the structure of the nanotubes the experimental measurement of the helicities will be compared with the simulated diffraction images [3]. This is the first determination of the chiral indices of single- and double-walled BNNTs.

[1] R. Arenal, et al., *Phys. Rev. Lett.* 95, 127601 (2005).

[2] R. Arenal, M. Kociak, A. Loiseau, D.J. Miller, submitted (2006).

[3] Ph. Lambin and A. A. Lucas, *Phys. Rev. B* 56, 3571 (1997).

124

Structural Materials for Fusion Reactors

M. A. Kirk[1], Z. Yao[2], and M. L. Jenkins[2]
[1] *Materials Science Division, Argonne National Laboratory*
[2] *University of Oxford*

A long-term solution to problems of energy production, greenhouse gas generation, and pollution control may rest with controlled nuclear fusion reactors. Candidate structural materials for such reactors include low-activation ferritic steels. Understanding and eliminating deleterious irradiation effects in these materials is the goal of continuing experiments of this collaboration using the Argone facility.

In recent experiments on ferritic alloys, we have found a significant difference with alloy composition in the microstructural response to irradiation, which corresponds to a bulk mechanical property change at a similar composition. In a collaboration between the Department of Materials at the University of Oxford and the Materials Science Division at Argonne National Laboratory, experiments that employ the unique transmission electron microscope and *in situ* ion irradiation user facility at Argonne were performed on a series of

Fe-Cr alloys. Enhanced nanometer-sized defect formation with Cr concentrations up to 11% have been found and correlated with a decrease in mechanical hardening and embrittlement in similar alloys

125

Non-Local and Double Scattering Models in High Resolution Electron Spectroscopy and Imaging

N. J. Zaluzec[1], L. J. Allen[2], S. D. Findlay[2], C. Witte[2], and M. P. Oxley[3]
[1] Materials Science Division, Argonne National Laboratory
[2] School of Physics, The University of Melbourne
[3] Condensed Matter Sciences Division, Oak Ridge National Laboratory

There are a number of factors affecting the formation of images based on core loss spectroscopy in high spatial resolution electron microscopy. We demonstrate unambiguously the need to use a full nonlocal description of the effective core-loss interaction for experimental results obtained from high angular resolution electron channelling energy-loss spectroscopy. The implications of this model are investigated for atomic resolution scanning transmission electron microscopy.

126

Orientation Dependence of the Near Edge Fine Structure in Electron Energy Loss Spectra from Graphite

A. Liu and N. J. Zaluzec
Materials Science Division, Argonne National Laboratory

Electron energy loss spectroscopy (EELS) in the transmission electron microscope (TEM) is the technique of choice for the chemical fingerprinting of amorphous carbon compounds. In this process, crystalline graphite is generally used as the sp2 standard. However, the energy loss near edge structure (ELNES) from graphite is highly orientation dependent, making its comparison with amorphous materials fraught. Many works prescribe a set of experimental conditions that minimize the orientation dependence, i.e., the "magic angle." Not only does this throw away valuable resolution, but there are many discrepancies both between theories and between experiments. New measurements using high angular resolution electron channeling electron spectroscopy (HARECES) from the graphite K edge are mined to compare to theoretical predictions, suggesting a lack in present formulations.

127

Development of Alternative Glass Formulations for Vitrification of Excess Plutonium—SEM/XRD Analyses

Charles L. Crawford, J. C. Marra, D. K. Peeler, N. E. Bibler, and C. M. Jantzen
Savannah River National Laboratory (SRNL)

The Department of Energy Office of Environmental Management (DOE/EM) plans to conduct the Plutonium Disposition Project at the Savannah River Site (SRS) to disposition excess weapons-usable plutonium. A plutonium glass waste form is the leading candidate for immobilization of the plutonium for subsequent disposition in a geologic repository. A reference lanthanide borosilicate (LABS) glass was developed and durability tested to provide data to support the Yucca Mountain License Application process. In addition to the reference LABS glass, alternative frit formulations have also been recently developed. This poster will present SEM/EDS and XRD data gathered at SRNL in these recent studies on surrogate glasses and actual plutonium-containing glasses. SEM/EDS applications were used to investigate various plutonium and hafnium oxide crystals observed in the glasses melted at 1500°C and in heat-treated glasses. XRD data were collected on these same powdered glass samples to confirm crystalline phase identification.

128

HARECES Measurements on Hexagonal Boron Nitride

R. Arenal[1], M. Kociak[2], and N. J. Zaluzec[1]
[1] *EMC group, Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA*
[2] *LPS, University Paris-Sud and CNRS, 91240 Orsay, France*

Hexagonal boron nitride (h-BN) is an insulating, anisotropic material structurally analogous to semimetallic graphite. Recently, the interest in h-BN has increased due to the synthesis of boron nitride nanotubes (BNNTs), which can be seen as a h-BN sheet rolled up into a cylinder. An improved knowledge of the physical properties of h-BN is necessary to understand the related properties in BNNTs. In this way, we are using a high angular resolution electron channeling electron spectroscopy (HARECES) technique to study electronic structure of h-BN. By studying both the core-loss and low-loss regions of an EEL spectrum, we are able to derive complementary information of the electronic structure and dielectric response of this material. We investigated both core-loss and low-loss regions of the EEL spectra recorded at different scattering vectors on a single crystal of h-BN, and we interpret the different excitations by comparison with theoretical models. Our results significantly improve on the older work on this material, and we demonstrate that HARECES is the most powerful technique to perform momentum-resolved EELS.

129

Recent Results from the Single-Crystal Diffractometer (SCD) Instrument at IPNS

T. F. Koetzle, M. E. Miller, P. M. B. Piccoli, and A. J. Schultz
Intense Pulsed Neutron Source Division, Argonne National Laboratory, Argonne, IL 60439 USA

The SCD instrument at IPNS has been upgraded with the installation of two new position-sensitive Anger camera scintillation detectors, leading to higher throughput on the instrument. This poster will highlight results of some recent experiments that have been carried out under the SCD user program. In particular, we will present results on a hydrogen-bonded material, tetraacetylethane, where we have undertaken a parametric study exploring how the short, intramolecular O-H-O hydrogen bond responds to changes in temperature. In a second project, we have studied covalent bond activation in polypyrazolylborate complexes. Polypyrazolyl-borates (also called scorpionates) are sigma complexes, defined as systems in which the two electrons in an X-H sigma bond form a dative bond with a transition metal.

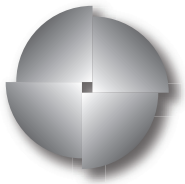
Work at ANL supported by U.S. DOE, Basic Energy Sciences - Materials Sciences, under contract W-31-109-ENG-38. We particularly wish to thank our collaborators J. Eckert (Los Alamos National Laboratory and UC Santa Barbara; tetraacetylethane) and S. Trofimenko (U Delaware; scorpionates).

130

Nano-Electro-Mechanical Systems Made of Diamond

N. Moldovan[1], R. Agrawal[1], K-H. Kim[1], H.D. Espinosa[1], R. Divan[2], D.C. Mancini[2], O. Auciello[3], and J.A. Carlisle[3]
[1] *Department of Mechanical Engineering, Northwestern University, Evanston, IL 60208-3111, USA*
[2] *Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA*
[3] *Materials Science Divisions, Argonne National Laboratory, Argonne, IL 60439, USA*

The low grain size (3-5 nm) and low roughness of ultra-nanocrystalline diamond (UNCD) made possible its 3-D patterning down to far sub-micrometer levels. Two applications are presented: UNCD probes for atomic force microscopy, showing tip radii as small as 25 nm, and nanocantilevers of width from 1000 nm to 100 nm and length in the 10^0–10^2 micrometer range. The UNCD probes were obtained by depositing the diamond in silicon molding pits achieved with anisotropic chemical etching and oxidation sharpening. UNCD nano-cantilevers were made by e-beam lithography, aluminum lift off, reactive ion etching of diamond, and anisotropic chemical etching of the underlying silicon. UNCD probes were characterized geometrically and functionally by high-resolution electron microscopy and various AFM techniques,



including AFM potentiometry. The UNCD nano-cantilevers were part of a comparative study with similarly fabricated silicon nitride structures, showing comparable nanofabrication capabilities and superior mechanical characteristics. The study shows that UNCD can be used in NEMS devices to benefit from its low wear rates, low friction coefficients, high Young's modulus, and tunable electrical conductivity.

131

Fabrication of Single, Magnetic Nanoparticle Devices

Jerald J. Kavich[1,2] and R. H. Kodama[1]
[1] Dept. of Physics, University of Illinois at Chicago, Chicago, IL 60607
[2] Advanced Photon Source, Argonne National Laboratory, Argonne IL 60439

We present our progress in fabricating and characterizing devices for measurement of electrical resistance of single magnetic nanoparticles of 20 nm diameter. Such devices are a promising testing ground for a host of topics in basic and applied nanomagnetism (e.g., spin dynamics, ballistic magnetoresistance, magnetic single-electron transistors, magnetic memories). The process involves deposition of monodisperse Ni nanoparticles by a cluster-beam method, using a high-pressure sputtering source (developed at UIC). This is followed by reactive sputter deposition of a thick film of amorphous alumina. A FIB (at Argonne's EMC) is used to mill small holes in the dielectric film covering a single particle. Finally, a W or Pt contact pad is deposited, making contact with the exposed surface of a nanoparticle. The high resolution of both electron and ion beams of this instrument allow identifying the location of particles to precisely locate the hole. AFM images taken on a series of holes using different milling times show that there is sufficient control over the depth to make contact without milling away much of the particle. Resistance measurements on the first set of devices were consistent with theoretical expectations for single, metallic constrictions < 10 nm in diameter.

132

An Overview of the Electron Microscopy Center

The Electron Microscopy Center Staff
Electron Microscopy Center, Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

This poster presents an overview of the mission and capabilities of the Electron Microscopy Center (EMC) for Materials Research at Argonne National Laboratory.

The EMC is a DOE-supported national user facility that provides scientists with forefront resources for electron beam characterization of materials. The EMC emphasizes three major areas: materials research, technique and instrumentation development, and operation as a national research facility. Users have access to resources and expertise to analyze structural and compositional information over critical length scales complementary to those probed by neutrons and photons.

The EMC currently maintains a suite of eight instruments that includes some of the world's unique electron microscopes. These instruments enable research in the following areas, among many others: *in situ* observation of ion irradiation, high- and low-temperature processes, and processes related to mechanical deformation; magnetic domain and moment imaging, high-resolution imaging (0.165 nm point resolution), field imaging using electron holography, chemical quantitation and distribution maps, EXELFS (similar to EXAFS), etc. Some examples will be provided.

133

In Situ TEM Ion Irradiation Study of Fe-Sulfide Compounds Found in Comets

Roy Christoffersen[1] and L. P. Keller[2]
[1] *SAIC, 2200 Space Park Drive, Houston, TX 77058*
[2] *Astromaterials Directorate, NASA Johnson Space Center, Houston, TX 77058*

NASA's STARDUST mission successfully returned samples from the Comet Wild-2 in January 2006. The mission is part of the agency's initiative to understand the processes that formed the primordial solid grains that comprised the early solar nebula. One of these key processes was the interaction between nebular grains and the energetic ion component of space plasmas. We have utilized the capabilities of the IVEM-Tandem facility to make the first *in situ* TEM ion radiation study of ion radiation processing in the mineral pyrrhotite, $Fe_{1-x}S$, an Fe-sulfide compound that is found in particles from comets. In these particles pyrrhotite is found in nanophase aggregates that may preserve relict interstellar materials highly processed by energetic place plasmas. In these aggregates, pyrrhotite is interpreted as a "radiation resistant" component within a matrix of radiation-amorphized silicate. Our results showed that pyrrhotite remains crystalline up to the highest practical experimental doses from ions with high deposition rates of nuclear collision energy. In contrast, a comparison sample of the Fe-Mg silicate mineral olivine became totally amorphous at a factor 4 lower level of deposited nuclear collision energy. The results constitute a confirmation of the current working model for these nanophase aggregates and have potential application for modeling the space radiation exposure histories of other solid grains in samples from the early solar system.

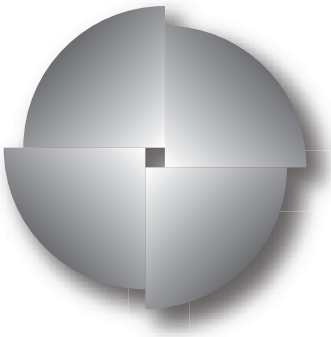
134

Analysis of the Element Composition and Microstructure of Ancient Eurasian Gold Work

David Peterson[1] and Lori Khatchadourian[2]
[1] *University of Chicago*
[2] *University of Michigan*

In January 2006, graduate student researchers with the Making of Ancient Eurasia project (MAE) began training in the use of the Hitachi S-4700 FESEM under the supervision of Rachel Koritala at the Electron Microscopy Center at Argonne National Laboratory. MAE is an integrated interdisciplinary research program in which researchers from the Department of Anthropology at the University of Chicago and from Argonne National Laboratory are collaborating in the simultaneous investigation of ceramic and metal technologies in the Eurasian steppes, the Caucasus, and central China in the fifth to first millennia BC.

Among the very first objects examined in the initial analyses have been samples of copper and bronze pendants covered in gold foil from Samara in the East European steppes of Russia, dating from the Late Bronze Age period of the mid-2nd millennium BC. A previous wavelength dispersed spectrometry (WDS) analysis of these objects showed a segregation of high-gold and high-silver zones in the foil that covers one of the pendants. This was unusual since gold and silver are completely soluble in one another, suggesting that it may have been a product of fabrication through the use of diffusion bonding. Close examination with FESEM and EDS now indicates that it is more likely to be a product of corrosion and depletion of silver on one side of the foil.



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Poster Index

Abbreviations

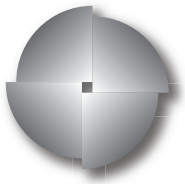
Cond. Matt. = Condensed Matter Physics

Elect./Mag. Mat & Devices = Electronic and Magnetic Material and Devices (CNM theme)

Env./Geo. = Environmental & Geology

NA = No abstract number *(submitted after publication deadline)*

Tech. = Techniques

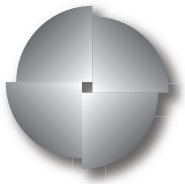


Center for Nanoscale Materials

Poster	Abstract	Submitter	Title	CNM Topic
C01	61	Chen, J.	Fabrication and Characterization of an Ultrasensitive Tin Oxide Nanoparticle Sensor	Nanopatterning
C02	117	Won	Nano- and Micro-Size Confinement of 2D Magnetic Stripe Domain System	Nanopatterning
C03	93	Yan, M.	Dynamic Origin of Stripe Domains	Theory and Simulation
C04	130	Moldovan	Nano-Electro-Mechanical Systems Made of Diamond	Nanophotonics
C05	10	Rose	Interacting Magnetic Nanostructures	Elec./Mag. Mat. & Devices
C06	16	Schmitt	Synthesis and Properties of Single-Crystalline FeSi and CoSi Nanowires	Elec./Mag. Mat. & Devices
C07	20	Bierman	Synthesis and Physical Properties of Ferromagnetic Semiconducting EuO Nanorods	Elec./Mag. Mat. & Devices
C08	88	Syed	High-Frequency Domain-Wall Motion and Magnetization Rotation of Patterned Permalloy Films under External Magnetic Field Excitation	Elec./Mag. Mat. & Devices
C09	90	Gusliyenko	X-ray PEEM Study of Vortex Dynamics in Ferromagnetic Nanodots	Elec./Mag. Mat. & Devices
C10	92	Buchanan	Magnetic Soliton Pair Dynamics	Elec./Mag. Mat. & Devices
C11	99	Yefremenko	Patterned Superconducting Micro-Bolometers for Array Detectors	Elec./Mag. Mat. & Devices
C12		omitted		
C13	65	Kharlamova	Magnetic Transitions in $CuFeO_2$ Induced by Magnetic Field and Low Temperatures and Measured Using NFS	Elec./Mag. Mat. & Devices
C14	77	Buonassisi	Engineering Metal-Impurity Nanodefects for Low-Cost Solar Cells	Elec./Mag. Mat. & Devices
C15	110	Miceli	Novel Growth and Coarsening Behavior of Nanocrystals Arising from Quantum Size Effects: Pb on Si(111)7x7 Surface	Elec./Mag. Mat. & Devices
C16	111	Miceli	The Formation of a Sharp Metal-Semiconductor Interface for the Growth of Quantum Size Effect Islands	Elec./Mag. Mat. & Devices
C17	118	Kelley	Electron Hopping in Pi-Stacked Covalent and Self-Assembled Perylene Diimides Observed by ENDOR Spectroscopy	Elec./Mag. Mat. & Devices
C18	NA	Shpyrko	Quantum Tunneling of Magnetic Domain Walls Studied with Coherent X-ray Speckle	Elec./Mag. Mat. & Devices
C19	123	Arenal	Assignment of Chiral Indices of Boron Nitride Nanotubes by Electron Diffraction	Elec./Mag. Mat. & Devices
C20	7	Liu, C.	Nanometer Focusing of Hard X-rays with Linear Zone-Plate Multilayer Laue Lenses	X-ray Imaging and Scattering

Center for Nanoscale Materials

Poster	Abstract	Submitter	Title	CNM Topic
C21	33	Bellare	USAXS Characterization of the Micro- and Nanostructure of Polyethylene Used in Orthopaedic Implants	X-ray Imaging and Scattering
C22	94	Winarski	Hard X-ray Nanoprobe	X-ray Imaging and Scattering
C23	101	Mukhopadhyay	X-ray Scattering Studies of Silica Nanoparticles Distributed on Liposome Vesicle Surface	X-ray Imaging and Scattering
C24	120	Budai	X-ray Laue Microdiffraction Studies of Individual ZnO Nanostructures	X-ray Imaging and Scattering
C25	54	Liu, D-J.	In Situ XAS Characterization of Aligned Carbon Nanotubes for PEM Fuel Cell Application	X-ray Imaging and Scattering
C26	102	Drazkowski	In Situ SAXS Observations on Deformations of Unoriented Styrene-Butadiene-Styrene Triblock Copolymer Grafted with Polyhedral Oligomeric Silsesquioxanes	X-ray Imaging and Scattering
C27	4	Vajda	X-ray Scattering Studies of Gold and Platinum Nanoparticles Formed by Atomic Cluster Deposition	X-ray Imaging and Scattering
C28	13	Casa	Inelastic X-ray Study of Plasmons in Oriented Single and Multi-Wall Carbon Nanotubes	X-ray Imaging and Scattering
C29	23	Hagman	Marker Motion Measurements of Nanoparticles in Polymeric Matrices	X-ray Imaging and Scattering
C30	35	Schultz	Monolayers, Wrinkling and Reversibility in Langmuir Films of Gold Nanoparticles	X-ray Imaging and Scattering
C31	52	Grigoriev	Dynamics of Strain and Polarization in Ferroelectrics	X-ray Imaging and Scattering
C32	70	Liang	Effects of X-ray Optics on Coherence	X-ray Imaging and Scattering
C33	76	Abel	Inelastic X-ray Scattering Study of the Lattice Dynamics of the Exotic Spin-Peierls Transition in TiOCl	X-ray Imaging and Scattering
C34	79	Shu	Precision Mechanical Design for a Hard X-ray Nanoprobe Instrument with Active Vibration Control in Nanometer Scale	X-ray Imaging and Scattering
C35	85	Lukaszew	XRTA Real-Time Studies on FePt Nanostructures	X-ray Imaging and Scattering
C36	106	LeFurgey	Effect of Valence on Antimony Uptake by Leishmania	X-ray Imaging and Scattering
C37	119	de Jonge	Near-Field Coherent Diffraction Imaging (NFCDI) of Electro-Deposited Lead Microparticles	X-ray Imaging and Scattering
C38	56	Makarov	Blu-Ice Implementation at GM/CA-CAT	X-ray Imaging and Scattering
C39	86	Jemian	The Small-Angle Scattering Instruments at Argonne National Laboratory	X-ray Imaging and Scattering
C40	NA	Rau	Imaging Nano-Structured Materials with a Hard X-Ray Microscope	X-ray Imaging and Scattering



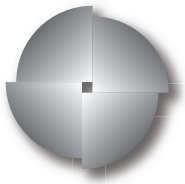
Electron Microscopy Center/Intense Pulsed Neutron Source

Poster	Abstract	Submitter	Title
E01	37	Kolesnik	Compositional Studies of Transition Metal Substituted ZnO
E02	64	Smith, K.	Radiation Damage Tolerance in $A_2B_2O_7$ Compounds
E03	122	Kabius	Benefits of Aberration Correction for Elemental Mapping
E04	123	Arenal	Assignment of Chiral Indices of Boron Nitride Nanotubes by Electron Diffraction
E05	124	Kirk	Structural Materials for Fusion Reactors
E06	125	Zalusec	Non-Local and Double Scattering Models in High Resolution Electron Spectroscopy and Imaging
E07	126	Zalusec	Orientation Dependence of the Near Edge Fine Structure in Electron Energy Loss Spectra from Graphite
E08	127	Crawford	Development of Alternative Glass Formulations for Vitrification of Excess Plutonium - SEM/XRD Analyses
E09	128	Arenal	HARECES Measurements on Hexagonal Boron Nitride
E10	131	Kodama	Fabrication of Single, Magnetic Nanoparticle Devices
E11	132	Cook	An Overview of the Electron Microscopy Center
E12	133	Christoffersen	*In Situ* TEM Ion Irradiation Study of Fe-Sulfide Compounds Found In Comets
E13	134	Peterson	Analysis of the Element Composition and Microstructure of Ancient Eurasian Gold Work
E13	106	LeFurgey	Effect of Valence on Antimony Uptake by Leishmania
E14	NA	Okuniewski	The Effects of Helium and Hydrogen in Irradiated Single Crystal BCC Iron: An Experimental and Modeling Study
E15	61	Chen, J.	Fabrication and Characterization of an Ultrasensitive Tin Oxide Nanoparticle Sensor
N1	3		Holoubek Self-Assembled Nanostructures in Copolymer Blends
N2	82	Benmore	Relationships between Intermediate- and Short-Range Order in Glasses
N3	129	Koetzle	Recent Results from the Single-Crystal Diffractometer (SCD) Instrument at IPNS
N4	56	Makarov	Blu-Ice Implementation at GM/CA-CAT
N5	86	Jemian	The Small-Angle Scattering Instruments at Argonne National Laboratory

Advanced Photon Source

Topics: Biology, polymers, chemistry, environmental & geology, condensed matter physics, materials science, nanoscience, techniques (diffraction, imaging, SAXS, surface studies, time-resolved, XAFS), other, instrumentation

Poster	Abstract	Submitter	Title	APS Topic
A1	78	Mulichak	High-Throughput Protein Crystallography at IMCA-CAT	Biology
A2	58	Smith, W.	GM/CA-CAT: Three Beamlines for Protein Crystallography at the APS	Biology
A3	108	Henning	BioCARS: A Facility for Macromolecular Crystallography at the Advanced Photon Source	Biology
A4	2	Fischetti	Wide Angle X-ray Scattering Reveals Crowding-Induced Suppression Protein Breathing	Biology
A5	42	Leu	Nuclear Resonance Vibrational Spectroscopy: A Quantitative Picture of Iron Dynamics in Proteins and Model Compounds	Biology
A6	95	Koksal	Measurement of Atomic Displacements in Binary Alloys with Size Mismatch	Biology
A7	97	Bhowmik	Probing the Mechanism of a C-C Bond Hydrolase by Crystallographic Studies	Biology
A8	106	LeFurgey	Effect of Valence on Antimony Uptake by Leishmania	Biology
A9	116	Srajer	Studies of Heme Proteins by Time-Resolved Crystallography: Allosteric Action and Structural Relaxation	Biology
A10	23	Hagman	Marker Motion Measurements of Nanoparticles in Polymeric Matrices	Polymers
A11	44	Malkova	Dewetting and Dynamics of Polymer Bilayer Films	Polymers
A12	33	Bellare	USAXS Characterization of the Micro- and Nanostructure of Polyethylene Used in Orthopaedic Implants	Polymers
A13	4	Vajda	X-ray Scattering Studies of Gold and Platinum Nanoparticles Formed by Atomic Cluster Deposition	Chemistry
A14	6	Silvernail	Observation of Fe Vibrational Modes in Heme Using Orientated Single Crystal Nuclear Resonance Vibrational Spectroscopy	Chemistry
A15	62	Xiao	Nuclear Resonance Vibrational Spectroscopy (NRVS) of Iron-Sulfur Cluster Metalloproteins and Model Compounds	Chemistry
A16	81	Benmore	Quantum Effects in Water	Chemistry
A17	103	Chen, L.	Light-Induced Time-Resolved X-ray Absorption as a Spectroscopic Probe of Chemical Transformations	Chemistry

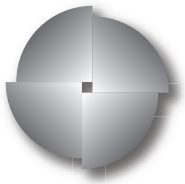


Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A18	104	Southworth	X-ray Microprobe of Atoms in Strong Optical Fields	Chemistry
A19	118	Kelley	Electron Hopping in Pi-Stacked Covalent and Self-Assembled Perylene Diimides Observed by ENDOR Spectroscopy	Chemistry
A20	8	Tyufekchieva	Spectromicroscopy Studies to Characterize Sulfur Species in Oxic and Anoxic Forest Soils	Env./Geo.
A21	21	Catalano	Resonant Anomalous X-ray Reflectivity Studies of As(V) Adsorption on Iron and Aluminum Oxide Surfaces	Env./Geo.
A22	114	Boyanov	The Effect of Fe(II)-Fe(II) Coordination on the Reduction of U(VI) Determined by Fe- and U-edge XAFS	Env./Geo.
A23	115	Boyanov	X-ray Micro(spectro)scopy of Metal Transformations Inside and Near Single Bacterial Cells	Env./Geo.
A24	NA	Prakpenka	On-line combination of Brillouin spectroscopy and x-ray diffraction at GSECARS	Env./Geo.
A25	51	Alatas	Inelastic X-ray Scattering Study of Supercooled Liquid and Solid Silicon	Cond. Matt.
A26	52	Grigoriev	Dynamics of Strain and Polarization in Ferroelectrics	Cond. Matt.
A27	57	Sichel	Dynamical Strain Imaging in Multiferroic Thin Films	Cond. Matt.
A28	1	Mei	Topological Changes in Glassy $GeSe_2$ under Pressure	Cond. Matt.
A29	11	Hong, H.	Time-Resolved Study of Ordering between Quantum Well Islands	Cond. Matt.
A30	19	Heald	XAFS Studies of Candidate Dilute Magnetic Semiconductor Materials	Cond. Matt.
A31	65	Kharlamova	Magnetic Transitions in $CuFeO_2$ Induced by Magnetic Field and Low Temperatures and Measured Using NFS	Cond. Matt.
A32	76	Abel	Inelastic X-ray Scattering Study of the Lattice Dynamics of the Exotic Spin-Peierls Transition in TiOCl	Cond. Matt.
A33	84	Mo	Observation of Surface Layering in a Molecular Liquid	Cond. Matt.
A34	107	Sandy	Structure and Dynamics of Attractive Colloidal Glasses	Cond. Matt.
A35	113	Larson	Nonresonant Inelastic X-ray Scattering and Energy-Resolved Wannier Function Investigation of Local Excitations in NiO and CoO	Cond. Matt.

Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A36	NA	Shpyrko	Quantum Tunneling of Magnetic Domain Walls Studied with Coherent X-ray Speckle	Cond. Matt.
A37	48	Baker	Application of Bond Constraint Theory to the $Ge_2Sb_2Te_5$ Optical Memory Material	Materials Science
A38	50	Yang, N.	*In Situ* Characterization of Catalysts and Adsorbents Using Synchrotron Powder Diffraction	Materials Science
A39	9	Gateshki	The Atomic-Scale Structures of $Ba_xSr_{1-x}TiO_3$ (x = 1, 0.5, 0) and GaN Nanocrystals Studied with High-Energy X-ray Diffraction	Materials Science
A40	25	Balasubramanian	Application of *In Situ* XAFS: Structural Study of Lithium-Ion Intercalation in Battery Materials	Materials Science
A41	29	Braun	Structure Analysis of Diesel Exhaust Fine Particulate Matter with Wide- and Small-Angle X-ray Scattering	Materials Science
A42	30	Braun	Ostwald-Ripening of Cobalt Precipitates in Silica Aerogels? – An Ultra-Small-Angle X-ray Scattering Study	Materials Science
A43	32	Do, D-H.	Quantitative Measurements of Piezoelectric Response in Ferroelectric Thin Films	Materials Science
A44	39	Zukoski	Ultra-Small-Angle X-ray Scattering from Dense Nanoparticle Suspensions	Materials Science
A45	43	Kodituwakku	Electronic Excitations in Organic Semiconductor Copper Phthalocyanine	Materials Science
A46	46	Stoupin	XRD and EXAFS Study of Single-Phase $PbTi_{(1-x)}Mn_{(x)}O_3$ Perovskites at High Mn Concentration	Materials Science
A47	67	Suryanarayanan	Solute Crystallization in Freeze-Dried Formulations—Improved Sensitivity and Quantification Using Synchrotron Radiation	Materials Science
A48	68	Allen	Combined X-ray Reflectivity and GI-SAXS Studies of Nucleation and Growth of ALD Hafnium Oxide High-k Dielectric Films	Materials Science
A49	69	Allen	USAXS Studies of Growth of Nanocrystalline Ceria in Real Time	Materials Science
A50	73	Olive	Spectroscopic Studies of Pb Corrosion of Reactor Materials	Materials Science
A51	80	Shibata	XAFS Studies of Fe-Doped $PbTiO_3$ Nanoparticles	Materials Science
A52	96	Puzyrev	Measurement of Atomic Displacements in Binary Alloys with Size Mismatch	Materials Science
A53	98	Tischler	Growth Transient Scaling during Pulsed Laser Deposition	Materials Science

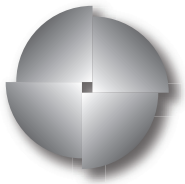


Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A54	102	Drazkowski	In Situ SAXS Observations on Deformations of Unoriented Styrene-Butadiene-Styrene Triblock Copolymer Grafted with Polyhedral Oligomeric Silsesquioxanes	Materials Science
A55	112	Christensen	X-ray Standing Wave Atomic Imaging of Platinum Nano-Crystals on $SrTiO_3$ (001)	Materials Science
A56	NA	Young	High Energy X-Ray Diffraction Measurements and Imaging of Superconducting Mg/MgB2 Composites under Compressive Loading	Materials Science
A57	90	Gusliyenko	X-ray PEEM Study of Vortex Dynamics in Ferromagnetic Nanodots	Materials Science
A58	127	Crawford	Development of Alternative Glass Formulations for Vitrification of Excess Plutonium - SEM/XRD Analyses	Materials Science
A59	54	Liu, D-J.	In Situ XAS Characterization of Aligned Carbon Nanotubes for PEM Fuel Cell Application	Nanoscience
A60	35	Schultz	Monolayers, Wrinkling and Reversibility in Langmuir Films of Gold Nanoparticles	Nanoscience
A61	77	Buonassisi	Engineering Metal-Impurity Nanodefects for Low-Cost Solar Cells	Nanoscience
A62	85	Lukaszew	XRTA Real-Time Studies on FePt Nanostructures	Nanoscience
A63	110	Miceli	Novel Growth and Coarsening Behavior of Nanocrystals Arising from Quantum Size Effects: Pb on Si(111)7x7 Surface	Nanoscience
A64	111	Miceli	The Formation of a Sharp Metal-Semiconductor Interface for the Growth of Quantum Size Effect Islands	Nanoscience
A65	88	Syed	High-Frequency Domain-Wall Motion and Magnetization Rotation of Patterned Permalloy Films under External Magnetic Field Excitation	Nanoscience
A66	94	Winarski	Hard X-ray Nanoprobe	Nanoscience
A67	101	Mukhopadhyay	X-ray Scattering Studies of Silica Nanoparticles Distributed on Liposome Vesicle Surface	Nanoscience
A68	120	Budai	X-ray Laue Microdiffraction Studies of Individual ZnO Nanostructures	Nanoscience
A69	123	Arenal	Assignment of Chiral Indices of Boron Nitride Nanotubes by Electron Diffraction	
A70	49	Chen, G.	Depth-Resolved Stress Profiling in Nanocrystalline Films with Submicron Resolution	Tech.-Diffraction
A71	13	Casa	Inelastic X-ray Study of Plasmons in Oriented Single and Multi-Wall Carbon Nanotubes	Tech.-Diffraction

Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A72	18	Fuller	Texture and Residual Strain Measurement under a Mild Scratch on Inconel 600 Using Synchrotron Micro-Laue Diffraction	Tech.-Diffraction
A73	70	Liang	Effects of X-ray Optics on Coherence	Tech.-Imaging
A74	119	de Jonge	Near-Field Coherent Diffraction Imaging (NFCDI) of Electro-Deposited Lead Microparticles	Tech.-Imaging
A75	NA	Erdmann	Sector 32-ID Beamline Upgrade at the Advanced Photon Source	Tech.-Imaging
A76	NA	Rau	Imaging Nano-Structured Materials with a Hard X-Ray Microscope	Tech.-Imaging
A77	NA	Richter	Visualizing soft tissue in the mammalian cochlea with In-line phase contrast imaging	Tech.-Imaging
A78	5	Williamson	Protein Self-Association Analyzed by Solution X-ray Scattering and Singular Value Decomposition	Tech./SAXS
A79	27	Irving	A High-Sensitivity, High-Spatial-Resolution CCD-Based Detector for Non-Crystalline Diffraction and SAXS Applications at Third-Generation Synchrotron Sources	Tech.-SAXS
A80	31	Segre	Nucleation of Glycine Crystals from Supersaturated Solutions Examined by Small-Angle X-ray Scattering	Tech.-SAXS
A81	34	Ilavsky	APS USAXS Instrument—Capabilities and Examples of Science	Tech.-SAXS
A82	86	Jemian	The Small-Angle Scattering Instruments at Argonne National Laboratory	Tech.-SAXS
A83	NA	Li	Developing A Dedicated Grazing-Incidence Small-Angle X-ray Scattering Beamline at the APS	Tech.-SAXS
A84	NA	Winans	Upgrade of 12-ID with Canted Undulators for Dedicated SAXS Beamlines	Tech.-SAXS
A85	53	Park, C.	New Capabilities of Probing Ion Adsorption at Solid-Liquid Interfaces with Resonant Anomalous X-ray Reflectivity	Tech.-Surface Studies
A86	12	Gordon	Characteristics of the MBE1 End Station at PNC/XOR	Tech.-Surface Studies
A87	121	Cionca	COBRA Studies of Ultrathin Ferroelectric Oxide Films	Tech.-Surface Studies
A88	66	Brewe	PNC/XOR Time-Resolved XAFS Apparatus	Tech.-Time-Resolved
A89	87	Reis	Picosecond X-ray Diffraction Measurements of Coherent Phonons at a Buried Interface	Tech.-Time-Resolved
A90	91	Walko	Ultrafast Lattice Dynamics of FeRh Films	Tech.-Time-Resolved

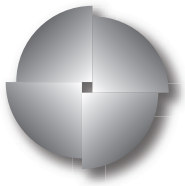


Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A91	105	Landahl	Measurement of the APS Bunch Length Using an Atomic Transition	Tech.-Time-Resolved
A92	45	Heald	Low-Energy XAFS at High Temperatures and Pressures: Ca^{2+} and Cl^{-} K-Edge Spectra of Supercritical Aqueous Solution	Tech.-XAFS
A93	26	Chattopadhyay	Local Structure of Nanosized Dilute Magnetic Semiconductors $Zn_{1-x}Mn_xO$ Studied Using Extended X-ray Absorption Fine Structure	Tech.-XAFS
A94	63	Liu, X.	Interfacial Cu of CdTe-Based PV Device	Tech.-XAFS
A95	14	Collins	Design and Testing of the Support/Mover System for the Undulators of the Linac Coherent Light Source	Other
A96	24	Jaski	Design and Application of CVD Diamond Windows for X-rays at the Advanced Photon Source	Other
A97	28	Lee, S-H.	Design of a Horizontal Bounce Double-Crystal Monochromator at the Advanced Photon Source	Other
A98	47	Wang, J.	NE-CAT 8-BM Upgrade	Instrumentation
A99	55	Mooney	Collaborative Development of Beamline Software	Instrumentation
A100	56	Makarov	Blu-Ice Implementation at GM/CA-CAT	Instrumentation
A101	59	Stepanov	Hardware-Synchronized On-the-Fly Scans at GM/CA-CAT	Instrumentation
A102	60	Devarapalli	GM/CA-CAT Computing Infrastructure for Fast Protein Crystallography	Instrumentation
A103	15	Kline	The EPICS Brick Development, Application, and Current Status	Instrumentation
A104	17	Benson	White and Pink Beam Slits for the Hard X-ray Nanoprobe Beamline at the Advanced Photon Source	Instrumentation
A105	22	Madden	Lens-Based Large Area CCD Detector for X-ray Crystallography	Instrumentation
A106	36	Sullivan	New Controls for 8-ID	Instrumentation
A107	38	Weizeorick	Detector Development in the XSD Beam Line Technical Support Group	Instrumentation
A108	40	Tieman	CCD Image Server: An EPICS-Based Server Program for Area Detector Acquisition	Instrumentation
A109	41	Tieman	One Interface to Control Them All—The Integrated Tomography System at 2BM	Instrumentation
A110	71	Fischetti	Performance of the New 3.0-cm Undulator at GM/CA-CAT	Instrumentation
A111	72	Segre	Design and Characterization of Compact Beam Cleaner for High-Energy XAS	Instrumentation

Advanced Photon Source

Poster	Abstract	Submitter	Title	APS Topic
A112	74	Yoder	Focal Characterization of "Bimorph" Mirrors	Instrumentation
A113	75	Xu, S.	Compact Systems Utilizing YAG Crystals to Image the Monochromatic Beam	Instrumentation
A114	79	Shu	Precision Mechanical Design for a Hard X-ray Nanoprobe Instrument with Active Vibration Control in Nanometer Scale	Instrumentation
A115	83	Narayanan	Design of an Ultrahigh Vacuum Artificial Channel-Cut Monochromator for Coherent Scattering Applications	Instrumentation
A116	89	Koshelev	Upgrade of IMCA-CAT Bending Magnet Beamline 17-BM for Macromolecular Crystallography at the Advanced Photon Source	Instrumentation
A117	100	Preissner	A Small Survey of Vibration Measurement and Analysis Techniques	Instrumentation
A118	NA	Brajuskovic	Use of CVD diamond in the design of synchrotron devices designed to withstand extreme heat loads	Instrumentation
A119	7	Liu, C.	Nanometer Focusing of Hard X-rays with Linear Zone-Plate Multilayer Laue Lenses	Instrumentation
A120	NA	Yang, N.	Effect of Different Preparation Procedures on the Sulfidibility of Ni-W Hydrocracking Catalysts Using *in situ* EXAFS and HRTEM	Chemistry

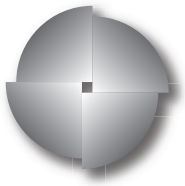


Index by Submitter

Abstract	APS	CNM	EMC/IPNS	Submitter	Title
76	A32	C33		Abel	Inelastic X-ray Scattering Study of the Lattice Dynamics of the Exotic Spin-Peierls Transition in TiOCl
51	A25			Alatas	Inelastic X-ray Scattering Study of Supercooled Liquid and Solid Silicon
68	A48			Allen	Combined X-ray Reflectivity and GI-SAXS Studies of Nucleation and Growth of ALD Hafnium Oxide High-k Dielectric Films
69	A49			Allen	USAXS Studies of Growth of Nanocrystalline Ceria in Real Time
123	A69	C19	E04	Arenal	Assignment of Chiral Indices of Boron Nitride Nanotubes by Electron Diffraction
128			E09	Arenal	HARECES Measurements on Hexagonal Boron Nitride
48	A37			Baker	Application of Bond Constraint Theory to the $Ge_2Sb_2Te_5$ Optical Memory Material
25	A40			Balasubramanian	Application of *In Situ* XAFS: Structural Study of Lithium-Ion Intercalation in Battery Materials
33	A12	C21		Bellare	USAXS Characterization of the Micro- and Nanostructure of Polyethylene Used in Orthopaedic Implants
81	A16			Benmore	Quantum Effects in Water
82			N2	Benmore	Relationships between Intermediate- and Short-Range Order in Glasses
17	A104			Benson	White and Pink Beam Slits for the Hard X-ray Nanoprobe Beamline at the Advanced Photon Source
97	A7			Bhowmik	Probing the Mechanism of a C-C Bond Hydrolase by Crystallographic Studies
20		C07		Bierman	Synthesis and Physical Properties of Ferromagnetic Semiconducting EuO Nanorods
114	A22			Boyanov	The Effect of Fe(II)-Fe(II) Coordination on the Reduction of U(VI) Determined by Fe- and U-edge XAFS
115	A23			Boyanov	X-ray Micro(spectro)scopy of Metal Transformations Inside and Near Single Bacterial Cells
NA	A118			Brajuskovic	Use of CVD diamond in the design of synchrotron devices designed to withstand extreme heat loads
29	A41			Braun	Structure Analysis of Diesel Exhaust Fine Particulate Matter with Wide- and Small-Angle X-ray Scattering
30	A42			Braun	Ostwald-Ripening of Cobalt Precipitates in Silica Aerogels? – An Ultra-Small-Angle X-ray Scattering Study
66	A88			Brewe	PNC/XOR Time-Resolved XAFS Apparatus
92		C10		Buchanan	Magnetic Soliton Pair Dynamics
120	A68	C24		Budai	X-ray Laue Microdiffraction Studies of Individual ZnO Nanostructures
77	A61	C14		Buonassisi	Engineering Metal-Impurity Nanodefects for Low-Cost Solar Cells
13	A71	C28		Casa	Inelastic X-ray Study of Plasmons in Oriented Single and Multi-Wall Carbon Nanotubes
21	A21			Catalano	Resonant Anomalous X-ray Reflectivity Studies of As(V) Adsorption on Iron and Aluminum Oxide Surfaces
26	A93			Chattopadhyay	Local Structure of Nanosized Dilute Magnetic Semiconductors $Zn_{1-x}Mn_xO$ Studied Using Extended X-ray Absorption Fine Structure
49	A70			Chen, G.	Depth-Resolved Stress Profiling in Nanocrystalline Films with Submicron Resolution
61		C01	E15	Chen, J.	Fabrication and Characterization of an Ultrasensitive Tin Oxide Nanoparticle Sensor
103	A17			Chen, L.	Light-Induced Time-Resolved X-ray Absorption as a Spectroscopic Probe of Chemical Transformations
112	A55			Christensen	X-ray Standing Wave Atomic Imaging of Platinum Nano-Crystals on $SrTiO_3$ (001)
133			E12	Christoffersen	*In Situ* TEM Ion Irradiation Study of Fe-Sulfide Compounds Found In Comets
121	A87			Cionca	COBRA Studies of Ultrathin Ferroelectric Oxide Films
14	A95			Collins	Design and Testing of the Support/Mover System for the Undulators of the Linac Coherent Light Source
132			E11	Cook	An Overview of the Electron Microscopy Center

Poster Index

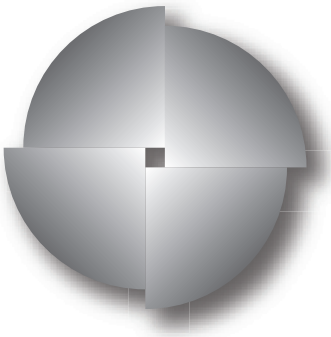
Abstract	APS	CNM	EMC/IPNS	Submitter	Title
127	A58		E08	Crawford	Development of Alternative Glass Formulations for Vitrification of Excess Plutonium - SEM/XRD Analyses
119	A74	C37		de Jonge	Near-Field Coherent Diffraction Imaging (NFCDI) of Electro-Deposited Lead Microparticles
60	A102			Devarapalli	GM/CA-CAT Computing Infrastructure for Fast Protein Crystallography
32	A43			Do, D-H.	Quantitative Measurements of Piezoelectric Response in Ferroelectric Thin Films
102	A54	C26		Drazkowski	*In Situ* SAXS Observations on Deformations of Unoriented Styrene-Butadiene-Styrene Triblock Copolymer Grafted with Polyhedral Oligomeric Silsesquioxanes
NA	A75			Erdmann	Sector 32-ID Beamline Upgrade at the Advanced Photon Source
2	A4			Fischetti	Wide Angle X-ray Scattering Reveals Crowding-Induced Suppression Protein Breathing
71	A110			Fischetti	Performance of the New 3.0-cm Undulator at GM/CA-CAT
18	A72			Fuller	Texture and Residual Strain Measurement under a Mild Scratch on Inconel 600 Using Synchrotron Micro-Laue Diffraction
9	A39			Gateshki	The Atomic-Scale Structures of $Ba_xSr_{1-x}TiO_3$ (x = 1, 0.5, 0) and GaN Nanocrystals Studied with High-Energy X-ray Diffraction
12	A86			Gordon	Characteristics of the MBE1 End Station at PNC/XOR
52	A26	C31		Grigoriev	Dynamics of Strain and Polarization in Ferroelectrics
90	A57	C09		Gusliyenko	X-ray PEEM Study of Vortex Dynamics in Ferromagnetic Nanodots
23	A10	C29		Hagman	Marker Motion Measurements of Nanoparticles in Polymeric Matrices
19	A30			Heald	XAFS Studies of Candidate Dilute Magnetic Semiconductor Materials
45	A92			Heald	Low-Energy XAFS at High Temperatures and Pressures: Ca^{2+} and Cl^- K-Edge Spectra of Supercritical Aqueous Solution
108	A3			Henning	BioCARS: A Facility for Macromolecular Crystallography at the Advanced Photon Source
3			N1	Holoubek	Self-Assembled Nanostructures in Copolymer Blends
11	A29			Hong, H.	Time-Resolved Study of Ordering between Quantum Well Islands
34	A81			Ilavsky	APS USAXS Instrument—Capabilities and Examples of Science
27	A79			Irving	A High-Sensitivity, High-Spatial-Resolution CCD-Based Detector for Non-Crystalline Diffraction and SAXS Applications at Third-Generation Synchrotron Sources
24	A96			Jaski	Design and Application of CVD Diamond Windows for X-rays at the Advanced Photon Source
86	A82	C39	N5	Jemian	The Small-Angle Scattering Instruments at Argonne National Laboratory
122			E03	Kabius	Benefits of Aberration Correction for Elemental Mapping
118	A19	C17		Kelley	Electron Hopping in Pi-Stacked Covalent and Self-Assembled Perylene Diimides Observed by ENDOR Spectroscopy
65	A31	C13		Kharlamova	Magnetic Transitions in $CuFeO_2$ Induced by Magnetic Field and Low Temperatures and Measured Using NFS
124			E05	Kirk	Structural Materials for Fusion Reactors
15	A103			Kline	The EPICS Brick Development, Application, and Current Status
131			E10	Kodama	Fabrication of Single, Magnetic Nanoparticle Devices
43	A45			Kodituwakku	Electronic Excitations in Organic Semiconductor Copper Phthalocyanine
129			N3	Koetzle	Recent Results from the Single-Crystal Diffractometer (SCD) Instrument at IPNS
95	A6			Koksal	Measurement of Atomic Displacements in Binary Alloys with Size Mismatch
37			E01	Kolesnik	Compositional Studies of Transition Metal Substituted ZnO
89	A116			Koshelev	Upgrade of IMCA-CAT Bending Magnet Beamline 17-BM for Macromolecular Crystallography at the Advanced Photon Source
105	A91			Landahl	Measurement of the APS Bunch Length Using an Atomic Transition
113	A35			Larson	Nonresonant Inelastic X-ray Scattering and Energy-Resolved Wannier Function Investigation of Local Excitations in NiO and CoO
28	A97			Lee, S-H.	Design of a Horizontal Bounce Double-Crystal Monochromator at the Advanced Photon Source



Abstract	APS	CNM	EMC/IPNS	Submitter	Title
106	A8	C36	E13	LeFurgey	Effect of Valence on Antimony Uptake by Leishmania
42	A5			Leu	Nuclear Resonance Vibrational Spectroscopy: A Quantitative Picture of Iron Dynamics in Proteins and Model Compounds
NA	A83			Li	Developing A Dedicated Grazing-Incidence Small-Angle X-ray Scattering Beamline at the APS
70	A73	C32		Liang	Effects of X-ray Optics on Coherence
7	A119	C20		Liu, C.	Nanometer Focusing of Hard X-rays with Linear Zone-Plate Multilayer Laue Lenses
54	A59	C25		Liu, D-J.	In Situ XAS Characterization of Aligned Carbon Nanotubes for PEM Fuel Cell Application
63	A94			Liu, X.	Interfacial Cu of CdTe-Based PV Device
85	A62	C35		Lukaszew	XRTA Real-Time Studies on FePt Nanostructures
22	A105			Madden	Lens-Based Large Area CCD Detector for X-ray Crystallography
56	A100	C38	N4	Makarov	Blu-Ice Implementation at GM/CA-CAT
44	A11			Malkova	Dewetting and Dynamics of Polymer Bilayer Films
1	A28			Mei	Topological Changes in Glassy $GeSe_2$ under Pressure
110	A63	C15		Miceli	Novel Growth and Coarsening Behavior of Nanocrystals Arising from Quantum Size Effects: Pb on Si(111)7x7 Surface
111	A64	C16		Miceli	The Formation of a Sharp Metal-Semiconductor Interface for the Growth of Quantum Size Effect Islands
84	A33			Mo	Observation of Surface Layering in a Molecular Liquid
130		C04		Moldovan	Nano-Electro-Mechanical Systems Made of Diamond
55	A99			Mooney	Collaborative Development of Beamline Software
101	A67	C23		Mukhopadhyay	X-ray Scattering Studies of Silica Nanoparticles Distributed on Liposome Vesicle Surface
78	A1			Mulichak	High-Throughput Protein Crystallography at IMCA-CAT
83	A115			Narayanan	Design of an Ultrahigh Vacuum Artificial Channel-Cut Monochromator for Coherent Scattering Applications
NA			E14	Okuniewski	The Effects of Helium and Hydrogen in Irradiated Single Crystal BCC Iron: An Experimental and Modeling Study
73	A50			Olive	Spectroscopic Studies of Pb Corrosion of Reactor Materials
53	A85			Park, C.	New Capabilities of Probing Ion Adsorption at Solid-Liquid Interfaces with Resonant Anomalous X-ray Reflectivity
134			E13	Peterson	Analysis of the Element Composition and Microstructure of Ancient Eurasian Gold Work
NA	A24			Prakpenka	On-line combination of Brillouin spectroscopy and x-ray diffraction at GSECARS
100	A117			Preissner	A Small Survey of Vibration Measurement and Analysis Techniques
96	A52			Puzyrev	Measurement of Atomic Displacements in Binary Alloys with Size Mismatch
NA	A76	C40		Rau	Imaging Nano-Structured Materials with a Hard X-Ray Microscope
87	A89			Reis	Picosecond X-ray Diffraction Measurements of Coherent Phonons at a Buried Interface
NA	A77			Richter	Visualizing soft tissue in the mammalian cochlea with In-line phase contrast imaging
10		C05		Rose	Interacting Magnetic Nanostructures
107	A34			Sandy	Structure and Dynamics of Attractive Colloidal Glasses
16		C06		Schmitt	Synthesis and Properties of Single-Crystalline FeSi and CoSi Nanowires
35	A60	C30		Schultz	Monolayers, Wrinkling and Reversibility in Langmuir Films of Gold Nanoparticles
31	A80			Segre	Nucleation of Glycine Crystals from Supersaturated Solutions Examined by Small-Angle X-ray Scattering
72	A111			Segre	Design and Characterization of Compact Beam Cleaner for High-Energy XAS
80	A51			Shibata	XAFS Studies of Fe-Doped $PbTiO_3$ Nanoparticles
NA	A36	C18		Shpyrko	Quantum Tunneling of Magnetic Domain Walls Studied with Coherent X-ray Speckle
79	A114	C34		Shu	Precision Mechanical Design for a Hard X-ray Nanoprobe Instrument with Active Vibration Control in Nanometer Scale

Poster Index

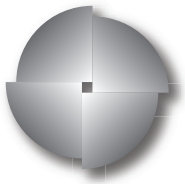
Abstract	APS	CNM	EMC/IPNS	Submitter	Title
57	A27			Sichel	Dynamical Strain Imaging in Multiferroic Thin Films
6	A14			Silvernail	Observation of Fe Vibrational Modes in Heme Using Orientated Single Crystal Nuclear Resonance Vibrational Spectroscopy
64			E02	Smith, K.	Radiation Damage Tolerance in $A_2B_2O_7$ Compounds
58	A2			Smith, W.	GM/CA-CAT: Three Beamlines for Protein Crystallography at the APS
104	A18			Southworth	X-ray Microprobe of Atoms in Strong Optical Fields
116	A9			Srajer	Studies of Heme Proteins by Time-Resolved Crystallography: Allosteric Action and Structural Relaxation
59	A101			Stepanov	Hardware-Synchronized On-the-Fly Scans at GM/CA-CAT
46	A46			Stoupin	XRD and EXAFS Study of Single-Phase $PbTi_{(1-x)}Mn_{(x)}O_3$ Perovskites at High Mn Concentration
36	A106			Sullivan	New Controls for 8-ID
67	A47			Suryanarayanan	Solute Crystallization in Freeze-Dried Formulations—Improved Sensitivity and Quantification Using Synchrotron Radiation
88	A65	C08		Syed	High-Frequency Domain-Wall Motion and Magnetization Rotation of Patterned Permalloy Films under External Magnetic Field Excitation
40	A108			Tieman	CCD Image Server: An EPICS-Based Server Program for Area Detector Acquisition
41	A109			Tieman	One Interface to Control Them All—The Integrated Tomography System at 2BM
98	A53			Tischler	Growth Transient Scaling during Pulsed Laser Deposition
8	A20			Tyufekchieva	Spectromicroscopy Studies to Characterize Sulfur Species in Oxic and Anoxic Forest Soils
4	A13	C27		Vajda	X-ray Scattering Studies of Gold and Platinum Nanoparticles Formed by Atomic Cluster Deposition
91	A90			Walko	Ultrafast Lattice Dynamics of FeRh Films
47	A98			Wang, J.	NE-CAT 8-BM Upgrade
38	A107			Weizeorick	Detector Development in the XSD Beam Line Technical Support Group
5	A78			Williamson	Protein Self-Association Analyzed by Solution X-ray Scattering and Singular Value Decomposition
NA	A84			Winans	Upgrade of 12-ID with Canted Undulators for Dedicated SAXS Beamlines
94	A66	C22		Winarski	Hard X-ray Nanoprobe
117		C02		Won	Nano- and Micro-Size Confinement of 2D Magnetic Stripe Domain System
62	A15			Xiao	Nuclear Resonance Vibrational Spectroscopy (NRVS) of Iron-Sulfur Cluster Metalloproteins and Model Compounds
75	A113			Xu, S.	Compact Systems Utilizing YAG Crystals to Image the Monochromatic Beam
93		C03		Yan, M.	Dynamic Origin of Stripe Domains
50	A38			Yang, N.	*In Situ* Characterization of Catalysts and Adsorbents Using Synchrotron Powder Diffraction
NA	A120			Yang, N.	Effect of Different Preparation Procedures on the Sulfidibility of Ni-W Hydrocracking Catalysts Using *in situ* EXAFS and HRTEM
99		C11		Yefremenko	Patterned Superconducting Micro-Bolometers for Array Detectors
74	A112			Yoder	Focal Characterization of "Bimorph" Mirrors
NA	A56			Young	High Energy X-Ray Diffraction Measurements and Imaging of Superconducting Mg/MgB2 Composites under Compressive Loading
125			E06	Zalusec	Non-Local and Double Scattering Models in High Resolution Electron Spectroscopy and Imaging
126			E07	Zalusec	Orientation Dependence of the Near Edge Fine Structure in Electron Energy Loss Spectra from Graphite
39	A44			Zukoski	Ultra-Small-Angle X-ray Scattering from Dense Nanoparticle Suspensions



2006 USERS MEETINGS

for DOE/BES User Facilities
at Argonne National Laboratory

Directory of Suppliers



Sponsors

Advanced Design Consulting USA, Inc. — General meeting support
MDC Vacuum Products/Insulator Seal — Tuesday poster session
Merck Research Laboratories — General meeting support
Oxford Danfysik — $500 Student Poster Prize
Veeco Instruments — $500 Student Poster Prize

Exhibitors (as of April 12, 2006)*

A&N Corporation
ACCEL Instruments GmbH
Advanced Design Consulting USA, Inc.*
Advent Associates, Ltd.
Aerotech, Inc.*
Alan Burrill Technical Sales*
Blake Industries, Inc.*
Brushwellman Electrofusion Products*
CAEN Technologies, Inc.*
CeramTec North America*
Fairchild Imaging, Inc.
Goodfellow Corporation*
Indelco Plastics
JEOL USA, Inc.*
Johnsen Ultravac Inc.*
Key High Vacuum Products Inc.
Kohzu America Inc.*
Mar USA, Inc.*
MDC Vacuum Products/Insulator Seal*
Micro Photonics
Midwest Vacuum, Inc.
Minarik Automation and Control*
Newport Corp. / Spectra-Physics*
Omicron NanoTechnology USA*
Oxford Danfysik*
Oxford Instruments*
Pfeiffer Vacuum*
Phytron, Inc.
Princeton Instruments/Acton*
Pro-Dex—Oregon Micro Systems*
PSIA, Inc.
Rigaku*
Struck Innovative Systeme GmbH*
Synergy Vacuum, Inc.
TecRep Corp.*
Televac*
Vacuum One*
Veeco Instruments*
VG Scienta, Inc.
Varian, Inc.
WIENER, Plein & Baus, Ltd.*
Xradia, Inc.*

**An asterisk indicates the company has provided information for this book. Detailed information about all exhibitors is provided in the registration materials and on the meeting web site.*

Building-Blocks for Vacuum Science & Technology

MDC VACUUM PRODUCTS, LLC

MDC's California manufacturing facility consists of five buildings

MDC Vacuum Products, LLC was founded in 1975 to serve a rapidly expanding vacuum industry. From its inception, MDC has established itself as an industry leader in satisfying the global demand for high

The Industry's largest in-stock Inventory

quality vacuum components while ensuring fast delivery and reasonable prices. MDC's reputation for quality, continuous process improvement, innovation and reliability have established MDC as a premier provider of vacuum instruments and components the world over. Customers include national laboratories, colleges and universities, original equipment manufacturers and domestic as well as international commercial accounts. MDC features the industry's largest selection of standard, off-the-shelf vacuum components. From the simplest of airtight seals to complex electron-beam coating instruments, MDC's product line provides a comprehensive solution to the world's scientific and industrial vacuum community's needs.
The MDC manufacturing facilities located in Hayward, California, total over 106,000 square feet.
MDC's provides Electrical & Optical, Ceramic-to-Metal, vacuum components via it's Insulator Seal division located in Sarasota, Florida. ISI has achieved a global reputation

MDC employs advanced (TIG) welding techniques

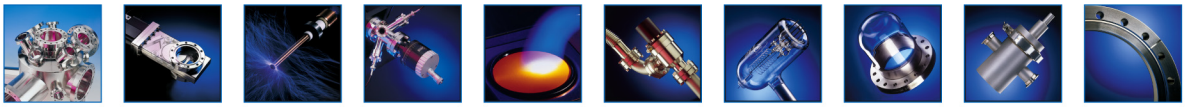
as well, for its high quality manufacturing and fast delivery of thousands of electrical and optical vacuum components. Some of the products available from Insulator Seal include a complete line of thermocouple feedthroughs, single and double-ended multipin instrumentation feedthroughs, an extensive selection of industry standard coaxial instrumentation and power feedthroughs, high frequency RF power feedthroughs, electrical breaks and viewports as well as custom engineered products for exotic or demanding applications.
MDC and ISI are committed to setting the industry standard for producing the highest quality vacuum products available and are both ISO 9001: 2000 certified.
A worldwide network of distributors and representatives cover North America, in the United States and Canada, while international representation extends to the Pacific Rim, Australia, China, India, Japan, Korea,

ISI INSULATOR SEAL
a division of MDC Vacuum Products, LLC

ISI, MDC's ceramic-to-metal division, is located in beautiful Sarasota, Florida

Malaysia, Singapore, Taiwan, Thailand and the Middle East. Representation for the European market is provided by Caburn-MDC Europe Ltd whose facilities include sales offices in the United Kingdom, France, Germany and Italy. For a comprehensive listing of all MDC and ISI sales representatives, or for complete details on any of our products, visit us online at **www.mdcvacuum.com** and **www.insulatorseal.com** and **www.caburn.co.uk**

ISI's Metalization application and assembly process



■ Flanges & Fittings ■ Valves ■ Roughing Components ■ Feedthroughs ■ Viewports ■ Motion & Manipulation ■ Chambers

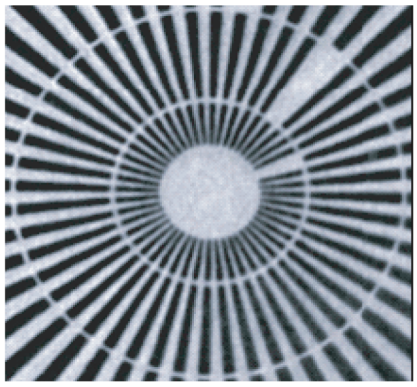
Turnkey X-ray Microscopy Solutions and Nanofocusing X-ray Optics

xradia

nanoXCT: Turn-key Transmission X-ray Microscope

20nm resolution at 8kV
2kV – 20kV x-ray energy
Zernike phase contrast mode
Synchrotron & Laboratory model
Automated 3D imaging
Energy tunable
Customization

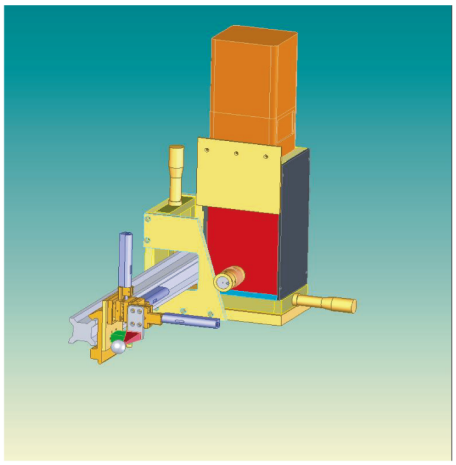
50nm lines/spaces



1 um

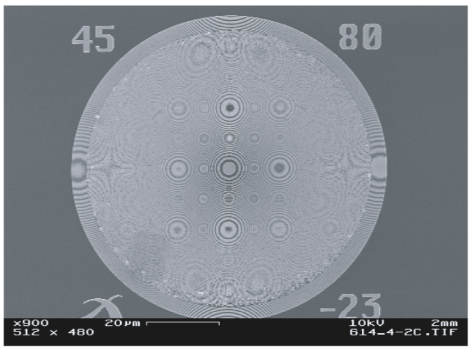
NanoRXI: Reflective Microscope for Surface Science

Non-contact surface x-ray imaging
Live monitoring of dynamic reactions
Imaging single atom-layer steps
100 nm lateral resolution
Integrates easily with existing diffractometers



Zone Plates

Highest resolution: outer zone width as fine as 25 nm
High x-ray energy range up to 50 keV
Up to 4.5mm diameter
Integrated central stop available



Kirkpatrick-Baez Mirror System

Turn-key system with sub-um resolution
Full remote control and readout position
Vacuum compatible models
100 & 200mm mirror length

Xradia, Inc. 5052 Commercial Circle, Concord CA 94520
Phone: (925) 288-1228 Fax: (925) 288-0310
www.xradia.com
sales@xradia.com

SCHEDULE AT A GLANCE

2006 Users Meeting for DOE/BES User Facilities at Argonne National Laboratory

	Monday, May 1	Tuesday, May 2	Wednesday, May 3	Thursday, May 4	Friday, May 5
	Exhibits 8:00 – 5:00 402 Gallery, 401 Atrium	**Exhibits** 8:00 – 5:00 402 Gallery, 401 Atrium	**Exhibits** 8:00 – 5:00 402 Gallery, 401 Atrium	**Exhibits** 8:00 – 12:00 402 Gallery, 401 Atrium	
Morning	**CNM Meeting – Introductory Session & Nanoscience I** 8:30 – 12:00 402 Lecture Hall *DOE Perspective* *CNM Updates* — *Chad Mirkin*	**WK1.A** Toward 1 nm X-ray Beams 9:00 – 12:15 402 Lecture Hall **WK2** Nanomaterials for Energy 8:30 – 12:30 402 Rm. E1100/E1200 **WK3** Nanophotonics 8:30 – 12:00 401 Rm. A5000 **WK4.A** Microscopy and Imaging 8:30 – 12:10 401 Rm. A1100	**Joint Meeting – Opening Session & Science Session I** 9:00 – 12:00 402 Lecture Hall *View from the Hill* *APS Update* — *Michelle Buchanan* *Dean Myles*	**WK7.A** Texture and Strain Mapping 8:30 – 12:00 401 Rm. A1100 **WK8.A** Inelastic X-ray Scattering 8:30 – 12:20 402 Rm. E1100/E1200 **WK9.A** X-ray Spectromicroscopy 8:30 – 12:10 402 Lecture Hall **WK10** Diffuse Scattering 8:30 – 12:00 401 Rm. A5000	**WK12.A** Microdiffraction in Structural Biology 9:00 – 11:45 402 Lecture Hall
Noon	Lunch 12:00 – 1:30 Tent	Lunch 12:00 – 1:30 Tent	Lunch 12:00 – 1:30 Tent	Lunch 12:00 – 1:30 Tent **APSUO Meeting** 12:15 – 1:30 401, A5000	Lunch 12:00 – 1:30 Tent
Afternoon	**CNM Meeting – Nanoscience I & II** 1:30 – 4:30 402 Lecture Hall *Seth Darling* *Gabriel Aeppli* *Matthias Bode* *Phillipe Guyot-Sionnest*	**WK1.B** Toward 1-nm X-ray Beams 1:30 – 4:30 402 Lecture Hall **WK4.B** Microscopy and Imaging 1:30 – 4:20 401 Rm. A1100 **WK5** Nanopatterning 1:30 – 5:30 402 Rm. E1100/E1200 **WK6** Quantum Nanomagnetism 1:30 – 5:30 401 Rm. A5000	**Joint Meeting – Science Session I** 1:30 – 4:30 402 Lecture Hall *Franklin Award Winner* *Mark Sutton* *Scott Chambers* *Ken Kemner* *Amy Rosensweig*	**WK7.B** Texture and Strain Mapping 1:30 – 5:00 401 Rm. A1100 **WK8.B** Inelastic X-ray Scattering 1:15 – 5:00 402 Rm. E1100/E1200 **WK9.B** X-ray Spectromicroscopy 1:30 – 6:00 402 Lecture Hall **WK11** Beamline Controls 1:30 – 5:30 401 Rm. A5000	**WK12.B** Microdiffraction in Structural Biology 1:30 – 4:15 402 Lecture Hall
Evening	**CNM Poster Session** 5:00 – 7:00 Bldg. 440* **Opening Reception** 5:00 – 7:00 Bldg. 440* **CNM Banquet** 7:30 – ?? Guest House	**EMC/IPNS Poster Session** 4:30 – 6:30 Bldg. 440* **APS Partner User Council** 6:30 – 8:30	**APS Poster Session** 4:30 – 6:30 Bldg. 440* **User Meeting Banquet** 6:45 – ?? Guest House	**Tenth Anniversary of APS Operations Celebration** Symposium 5:00 – 6:30 Argonne Guest House **Tenth Anniversary of APS Operations Celebration** Dinner 7:00 – ?? Argonne Guest House	

* The atrium of Building 440 may be entered from the parking lot off Kearney Road or from the APS experiment hall at sector 26 (follow posted signs to the atrium). **Be aware that portions of Building 440 may have work-in-progress areas. Do not enter any areas posted as such; obey all signs**.