Argonne NATIONAL LABORATORY

USERS WEEK 2009

Program and Abstracts

Argonne National Laboratory
May 4–6, 2009

SPECIAL FOCUS ON APS RENEWAL

U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by UChicago Argonne, LLC

PROGRAM AND ABSTRACTS

May 4–6, 2009

Advanced Photon Source
Argonne National Laboratory
Argonne, Illinois USA

SPECIAL FOCUS ON APS RENEWAL

User Facilities at Argonne National Laboratory

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U.S. Department of Energy, Office of Science, Office of Basic Energy Science

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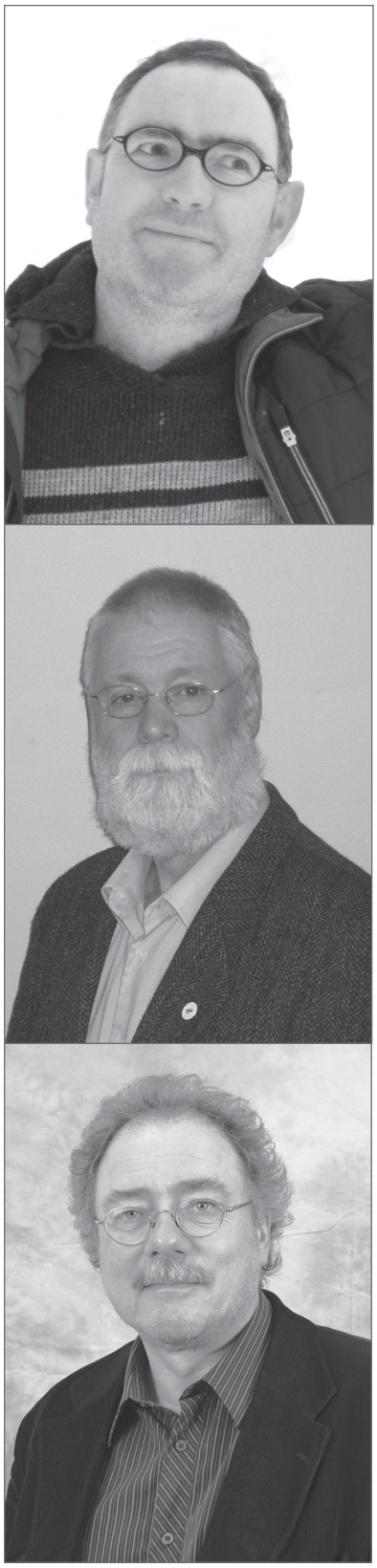

Simon G. J. Mochrie (top), Mark Sutton (middle), and Gerhard Grübel (bottom)

2009 Arthur H. Compton Award

The Department of Energy's Advanced Photon Source (APS) and the APS Users Organization announced that the 2009 Arthur H. Compton Award will be presented jointly to Simon Mochrie, Mark Sutton, and Gerhard Grübel for their pioneering efforts in x-ray photon correlation spectroscopy (XPCS), which exploits the coherent properties of synchrotron x-rays to study the slow dynamics of condensed matter at short length scales.

"XPCS seemed like a heroic experiment only a decade ago, but it is now used routinely to do great science at APS and other sources. We are grateful for the pioneering vision of our winners in making this research possible," said Murray Gibson, director of the APS.

The XPCS technique has evolved into a sophisticated tool for studying slow dynamics in inhomogeneous systems at length scales too small for other techniques. The wide range of systems studied include block copolymers, micellar systems, colloidal suspensions, liquid surfaces, molten polymer films, membranes and binary alloys. The award winners have played a significant role in driving the evolution and application of this technique.

X-ray photon correlation spectroscopy makes use of the way coherent light scatters from irregular structures. The coherent portion at the center of a synchrotron undulator beam is selected with a pinhole aperture. When this coherent beam hits the sample, the scattered light bunches into spots, called speckles. As the structure of the sample changes, the intensity of light at each speckle changes. By monitoring how the intensity fluctuates across the whole pattern of speckles—like watching flames pass through a bed of embers—it is possible to learn how the structure of the sample changes with time.

The technique was first reported for x-rays by Sutton, Mochrie, and colleagues in a 1991 letter to *Nature* [1], in which they described work at the National Synchrotron Light Source at Brookhaven National Laboratory. A similar technique, dynamical light scattering with visible laser light, was widely used at the time, but the extension to x-rays makes it possible to study opaque samples and work at much smaller length scales (well under 200 nanometers).

The technical challenges of the technique were significant. Because so little of the available beam is coherent, and because x-rays interact weakly with samples in general, these XPCS pioneers had to devise both efficient detection strategies for tiny signals and sophisticated algorithms to analyze those signals as they varied over a large area of detector space.

Simon Mochrie and Mark Sutton were key contributors in the development of beamline 8-ID at the APS, which remains the only facility for XPCS in the United States. Both were active in the management and scientific direction of the IBM-McGill-MIT-Yale Collaborative Access Team (IMMY-CAT), which built and operated sector 8 until it transitioned to APS management in 2003. Many other investigators associated with IMMY-CAT also contributed to the development of the technique and its application. Mochrie is currently Professor of Physics and Professor of Applied Physics at Yale University, New Haven, Connecticut; Sutton is Professor of Physics at McGill University in Montreal, Canada.

Gerhard Grübel brought the technique to Europe, leading the development of the ID10 (Troika) beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, and pioneered many innovative applications of XPCS. He is now a senior scientist at Hasylab at the Deutsches Elektronen-Synchrotron (DESY) in Hamburg, Germany, where he is involved in the development of coherence based techniques for free-electron laser sources.

In addition to developing the "how" of XPCS, all three winners have used it to significantly advance the "what" of their own disciplines. The following are examples selected by the winners as illustrating the impact of XPCS in their work.

Simon Mochrie studies the properties, phase behavior, and phase transitions of soft matter. His work to characterize a sponge phase of a block copolymer [2] marked a step forward in terms of both technique and science. The quality of the data showed that XPCS is useful for studying polymer dynamics; furthermore, as one of the first studies to use a fast x-ray area detector, this work has prompted further development of those detectors. In terms of the system studied, XPCS revealed a previously unobserved crossover from stretched to compressed exponential relaxations as a function of temperature. The data also provided a clear example of mysterious compressed exponential relaxations that do not appear to evolve with time, realizing a new paradigm for relaxations in highly viscous media.

Mark Sutton's group studies the time evolution of non-equilibrium systems. He and colleagues have since demonstrated a controllable way to combine a reference signal (heterodyning) with coherent small angle x-ray scattering [3]. This adaptation of the XPCS technique gives direct access to phase information that can, for instance, be used to separate the effects of advection (local flow) from dissipation (randomness).

Gerhard Grübel and his group work primarily on the bulk and surface dynamic properties of complex fluids and more recently on glassy and magnetic systems. His work on colloidal silica suspensions [4] illustrated the strength of XPCS in combination with small-angle scattering for the quantitative characterization of colloidal fluids. It motivated a series of subsequent studies challenging, in particular, our knowledge of the direct and indirect hydrodynamic interactions in concentrated soft-sphere fluids.

Along with their early collaborators Steve Dierker, Larry Lurio, Ian Robinson, Brian Stephenson, and others, the three award winners have pioneered a scientific thrust that has had long-range influence on research at synchrotron sources. The techniques they have developed will continue to drive development and science at beamlines at APS, ESRF, and elsewhere and will have significant influence on the next generation of free-electron lasers.

References

[1] M. Sutton, S. G. J. Mochrie, T. Greytak, S. E. Nagler, L. E. Berman, G. A. Held, and G. B. Stephenson, "Observation of speckle by diffraction with coherent X-rays," *Nature* **352**, 608–610 (15 August 1991).

[2] P. Falus, S. Narayanan, A. R. Sandy, S. G. J. Mochrie, "Crossover from stretched to compressed exponential relaxations in a polymer-based sponge phase," *Phys. Rev. Lett.* **97,** 066102 (2006)

[3] F. Livet, F. Bley, F. Ehrburger-Dolle, I. Morfin, E. Geissler, and M. Sutton, "X-ray intensity fluctuation spectroscopy by heterodyne detection," *J. Synchrotron Rad.* **13,** 453-458 (2006).

[4] G. Grübel, D. L. Abernathy, D. O. Riese, W.L. Vos, and G. H. Wegdam, "Dynamics of dense, charge-stabilized suspensions of colloidal silica studied by correlation spectroscopy with coherent X-rays," *J. Appl. Cryst.* **33,** 424 (2000).

COMPREHENSIVE PROGRAM

Sunday, May 3

11:00 – 3:00 Exhibitor Set-up
Bldg. 402, Gallery;
Bldg. 401, Atrium

5:00 – 7:00 Registration
Bldg. 401, Atrium

5:00 – 7:00 Exhibitor Expo and Reception
Bldg. 402, Gallery;
Bldg. 401, Atrium

Monday, May 4

Registration: 7:00 – 5:00
Bldg. 401, Atrium

Exhibits: 8:00 – 5:00
Bldg. 402, Gallery;
Bldg. 401, Atrium

Lunch: 11:45 – 1:30
In the tent

Opening Session

Bldg. 402, Lecture Hall

8:15 – 8:30 Welcome
Laurence Lurio, Northern Illinois University, APSUO Chair

8:30 – 9:30 Update on the Advanced Photon Source
Murray Gibson, Director, APS

9:30 – 10:00 DOE Perspective
Pedro Montano, Director, Scientific User Facilities Division,
Office of Basic Energy Sciences, Office of Science, U.S. Department of Energy

10:00– 10:30 Coffee Break
Gallery and Atrium

10:30 – 11:00 Compton Award Presentation and Lecture
Simon Mochrie, Yale University
Mark Sutton, McGill University
Gerhard Grübel, Hasylab/DESY

11:00 – 11:15 Update on the Center for Nanoscale Materials
Stephen Streiffer, Acting Director, CNM

11:15 – 11:45 Update on the Electron Microscopy Center
Dean Miller, Director, EMC

11:45 – 1:00 Lunch
Tent

Monday, May 4

Science Highlights

Bldg. 402, Lecture Hall

1:00	Welcome *Moderator: Linda Young, Argonne National Laboratory*
1:00 – 1:30	Strategic Directions at Argonne National Laboratory *Eric Isaacs, Director, Argonne National Laboratory*
1:30 – 1:50	Finding Gems in Low-quality Crystalline Proteins with an X-ray Mini-beam *Janet Smith, University of Michigan*
1:50 – 2:10	Invited Student Talk
2:10 – 2:40	Benefits of Aberration-corrected TEM for Materials Science Problems *Bernd Kabius, Argonne National Laboratory*
2:40 – 3:00	Chemical Switching of Polarization in Ultrathin Ferroelectric $PbTiO_3$ Films *Brian Stephenson, Argonne National Laboratory*
3:00 – 3:30	Coffee Break *Gallery and Atrium*
3:30 – 5:00	APS Renewal Status Report *Moderator: Dennis Mills*
5:00 – 7:00	APS Poster Session & Reception *Bldg. 437*
7:00 – 8:00	APS Partner User Council Meeting & Dinner *Argonne Guest House*
Evening	Special interest group meetings as scheduled by groups

Tuesday, May 5, 2009

Registration: 7:30 – 5:00
Bldg. 401, Atrium

Exhibits: 8:00 – 5:00
Bldg. 402, Gallery;
Bldg. 401, Atrium

APS Renewal Focus, Part 1: Science

Bldg. 402, Lecture Hall

8:00 – 8:10 Welcome
Moderator: David Tiede, Argonne National Laboratory

8:10 – 8:50 Dynamics of Life
Keith Moffat, University of Chicago

8:50 – 9:25 Materials for Energy Applications
Michelle Buchanan, Oak Ridge National Laboratory

9:25 – 10:00 Materials under Pressure
Russell Hemley, Carnegie Institution of Washington

10:00 – 10:30 Coffee Break
Gallery and Atrium

10:30 – 11:10 Applications of Synchrotron Radiation to the Characterization of Real-world Materials Systems
Roger Leach, DuPont

11:10 – 11:45 **User Visions, Part 1**
A Path Forward to Advanced Nuclear Fuels, Founded upon a Unique and Crucial New Facility at the APS
James Tobin, Lawrence Livermore National Laboratory

High-field Magnets for X-ray Scattering Studies of Materials
Zahir Islam, Argonne National Laboratory

Simultaneous Real-space Imaging and Diffraction Studies of Nano-scaled Structures
Hawoong Hong, Argonne National Laboratory

Biological Imaging at the APS: Imagining the Future
Lydia Finney, Argonne National Laboratory

11:45 – 1:00 Lunch
Tent

Tuesday, May 5, 2009

APS Renewal Focus, Part 2: Techniques

Bldg. 402, Lecture Hall

1:00 Welcome
Moderator: Peter Eng, University of Chicago

1:00 – 1:35 Coherent Diffraction and X-ray Imaging
Keith Nugent, University of Melbourne, Australia

1:35 – 2:10 X-ray Microscopy: Pictures of Complexity
Chris Jacobsen, Stony Brook University

2:10 – 2:45 Advanced X-ray Optics
Kazuto Yamauchi, Osaka University

2:45 – 3:15 Coffee Break
Gallery and Atrium

3:15 – 3:50 Advanced X-ray Detector Development at NSLS
Peter Siddons, Brookhaven National Laboratory

3:50 – 4:25 Ultrafast X-ray Spectroscopy of Solvated Transition-metal Complexes and Oxide Materials
Robert Schoenlein, Lawrence Berkeley National Laboratory

4:30 – 5:10 **User Visions, Part 2**
Picosecond Temporal Resolution for Plasmonic and Acoustic Properties in Nanomaterials
Yuelin Li, Argonne National Laboratory

Rapid, Ultra-fast Time-resolved Laue Data Collection from Tiny Crystals after Homogenous Reaction Initiation
Marius Schmidt, University of Wisconsin-Milwaukee, Physics Department

Ultra-fast Polarization Phase Selective (PPS) Measurements at APS
Kresimir Rupnik, Louisiana State University

Hard X-ray in situ Real-time Interfacial-structural-spectro-nanoscopy
Paul Fenter, Argonne National Laboratory

5:10 – 5:45 APS Renewal Discussion
Moderator: Paul Fuoss

6:00 – 9:00 Banquet
Argonne Guest House

Wednesday, May 6, 2009

Registration: 7:30 – 5:00
Bldg. 401, Atrium

Exhibits: 8:00 – 5:00
Bldg. 402, Gallery;
Bldg. 401, Atrium

11:45 – 1:30 Lunch
In the tent

12:00 – 1:30 APSUO Steering Committee
Bldg. 401, 5th Floor Gallery

5:15 – 5:45 Workshop Wrapup Reports
Bldg. 402, Lecture Hall

Half-day Workshops—Morning

8:40 – 12:15 WK2: Optics for the Future (morning)
Bldg. 402, Lecture Hall

Half-day Workshops—Afternoon

1:30 – 5:00 WK1: Detectors for the Future (afternoon)
Bldg. 402, Lecture Hall

1:30 – 5:00 WK3: NextGen Nanopositioning—Engineering Robust Systems for Manipulation and Analysis (afternoon)
Bldg. 440 (CNM), Rm. 105/106

Full-day Workshops

8:55 – 11:55
1:20 – 5:00 WK4: High-speed Imaging Opportunities with X-rays
Bldg. 402, Rm. E1100/E1200

9:00 – 12:00
1:30 – 5:00 WK5: Imaging Structural Hierarchy in Biological Systems
Bldg. 401, Rm. A1100

8:55 – 11:45
1:15 – 5:15 WK6: Synchrotron Radiation in Chemical Science
Bldg. 401, Rm. A5000

Wednesday, May 6, 2009

Auxiliary Workshop*

8:30 - 12:30
2:15 - 6:00 WK7: High Pressure Synchrotron Science: Future Directions
Bldg. 362 Auditorium (shuttle bus service from Guest House and APS Conference Center)

Workshop 7 continues on Thursday and Friday in the Bldg. 402 Lecture Hall. See workshop web page for details: http://www.hpsync.org/links/HiPreSS/.

**Auxiliary Workshop A, "High-performance Computing at the APS," which was originally scheduled for Sunday, has been postponed.*

GENERAL SESSION ABSTRACTS

General Session: Science Highlights

Finding Gems in Low-quality Crystalline Proteins with an X-ray Mini-beam

Janet L. Smith[1], Jennifer Gehret[1], Shenglan Xu[2], Derek W. Yoder[2], Oleg Makarov[2], Mark C. Hilgart[2], Sergey Stepanov[2], Sudhir Pothineni[2], Stephen Corcoran[2], Venugopalan Nagarajan[2], Craig Ogata[2], Ruslan Sanishvili[2], Michael Becker[2] and Robert F. Fischetti[2]
[1] Department of Biological Chemistry, Life Sciences Institute, University of Michigan, 210 Washtenaw Ave., Ann Arbor, MI 48109 USA
[2] GM/CA CAT, Biosciences Division, Argonne National Laboratory, 9700 South Cass Ave., Argonne, IL 60439 USA

Protein crystals frequently suffer from problems such as high mosaicity, split or smeared diffraction maxima, small size, multiple lattices and radiation sensitivity. Many such crystals are useless in an "ordinary" beam, but can yield high-quality diffraction data in the 5-µm to 20-µm mini-beam at GM/CA CAT. The enzyme TE-DC is a good example. Twenty-five crystalline samples of selenomethionyl (SeMet) TE-DC were screened, but the only sample that diffracted beyond ~4 Å was a double crystal. A single-crystal region within the double crystal was selected by diffraction rastering with a 20-µm mini-beam; however, Friedel data from this region were incomplete due to radiation damage. A fresh region of the sample was selected by diffraction rastering with a 10-µm mini-beam, and additional Friedel data were collected. The merged, complete SeMet Friedel data set yielded a 2.1-Å electron density map of stunning quality 3 hrs after the diffraction experiment. The stable mini-beam combined with an intuitive user interface offering automated sample mounting and screening, beam-size selection and raster probing have allowed many users to find gems in their low-quality samples.

Research supported by NIH grant R01 DK042303 to JLS, and by NIH interagency agreements NIGMS Y1-GM-1104 and NCI Y1-CO-1020 to RFF.

Benefits of Aberration-corrected TEM for Materials Science Problems

B. Kabius
Center for Electron Microscopy, Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

During the last 10 years several aberration-correction concepts for electron microscopes have succeeded in improving spatial resolution and analytical capabilities. Electron optical systems for correction of spherical aberration are now a valuable tool for materials science research, and several investigations have already exploited some of the benefits of C_s-correction for high-resolution TEM and STEM. The TEAM (Transmission Electron Aberration-corrected Microscopy) project is a collaborative DOE project that will extend the present capabilities of aberration-correction technology. The goals for aberration correction within the TEAM project are

- Correction of higher order aberrations, such as fifth-order spherical aberration, which is required for improving interpretability at sub-Angstrom resolution (TEM) and higher beam currents in smaller electron probes (STEM).
- Improving the information limit to 0.5 Å by correction of chromatic aberration (C_c) and energy monochromation.

This progress in electron beam instrumentation is expected to have a strong impact on *in situ* TEM, magnetic imaging, and analytical electron microscopy. The benefits of C_s- and C_c-correction for materials science problems requiring these methods will be discussed and first results using C_c-correction will be presented.

This work was supported by the U.S. Department of Energy, BES-Materials Sciences, under Contract No. DE-AC02-06CH11357

Chemical Switching of Polarization in Ultrathin Ferroelectric $PbTiO_3$ Films

G.B. Stephenson[1,2], R.-V. Wang[2], D.D. Fong[1], F. Jiang[1], M.J. Highland[1], T.T. Fister[1], P.H. Fuoss[1], J.A. Eastman[1], S.K. Streiffer[2], and Carol Thompson[3]
[1] Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
[2] Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439
[3] Department of Physics, Northern Illinois University, DeKalb, IL 60115

Ferroelectrics are both fascinating and useful because their atomic-scale structure responds strongly to electric field and temperature. Large interfacial effects make ultrathin films behave very differently than bulk crystals. We have been growing and studying such films at sector 12ID-D, using *in situ* surface diffraction to monitor film growth and observe the polarization structure as a function of temperature, thickness, and environmental conditions. Recently we discovered that the effects of applied chemical potential are similar to those of applied electric potential. The direction of polarization in an ultrathin epitaxial $PbTiO_3$ film can be switched by changing the oxygen partial pressure in equilibrium with its surface. This opens up a rich new area for study, in which surface chemistry has a large interaction with the ferroelectric phase transition.

Work supported under contract DE-AC02-06CH11357 between UChicago Argonne LLC and the Dept. of Energy.

APS Renewal Focus: Science

Dynamics of Life

Keith Moffat
Department of Biochemistry & Molecular Biology, Institute for Biophysical Dynamics and the Consortium for Advanced Radiation Sources
The University of Chicago

The organizers' title may be a bit over the top—but if it doesn't move, it's not "living". The "it" can refer to molecules small or large, organelles, whole cells or of course intact organisms. Synchrotron X-ray sources can in principle, and in many cases in practice, successfully probe biological, biochemical and chemical dynamics over a wide range of time and length scales, from femtoseconds to the generation time of an organism, and from ~10 pm to centimeters.

Illustrations will be drawn largely from studies at the molecular level in which, for example, a chemical process such as isomerization that lies at the heart of perception of light by a biological photoreceptor can be followed structurally in real time, as the atoms and groups of atoms move around.

The limitations of today's approaches to experimental dynamics—X-ray source, experimental design, detectors, radiation damage, data analysis—will be briefly discussed.

Materials for Energy Applications

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

The world's demand for energy is projected to double by 2050, requiring tremendous growth in energy generation capacity. Clearly, a more diverse portfolio of energy sources must be developed, especially when one takes into account the limited supply of readily available fossil fuels and increasing environmental concerns. Energy systems of the future, whether they convert sunlight into electricity, produce fuel from splitting water, or transform carbon dioxide into fuel, are centered on materials. Next-generation energy technologies will place dramatically higher demands on materials—requiring enhanced functionality and performance under extreme environments. The ability to design materials at the atomic level, taking advantage of new synthesis approaches, characterization tools, and computational modeling, has the potential of yielding unprecedented advances in new materials that will be needed in future energy technologies.

Materials under Pressure

Russell J. Hemley
Geophysical Laboratory, Carnegie Institution, Washington, D.C. 20015

Experiments based on diamond technology are now providing unprecedented insight into the nature of materials under extreme conditions. At pressures up to >300 GPa and temperatures from millikelvins to thousands of degrees, new phenomena are observed in hydrogen and other simple molecular systems; new superconducting, electronic, and magnetic materials are created; and unexpected findings in soft matter and biological systems are observed. These discoveries have been made possible by synergistic advances in synchrotron radiation methods, combined with allied developments in a variety of laser, electrical transport, magnetic, neutron scattering, and other analytical methods. Continued developments at dedicated high-pressure beamlines as well as beamlines optimized for particular techniques are essential for taking this research to the next level. New classes of experiments will require the use of nanobeams, higher energy photons, coherence, and beam time structure. There are important opportunities for dynamic compression and combined static/dynamic experiments at synchrotron and other advanced light sources in furthering our understanding of materials in extreme states.

Applications of Synchrotron Radiation to the Characterization of Real-world Materials Systems

Roger A. Leach, W. E. Guise, J. David Londono, and H. David Rosenfeld
Central Research and Development Department, Corporate Center for Analytical Sciences, DuPont Company

DuPont has been a participating member of DND-CAT at the Advanced Photon Source since the start-up of the facility in 1996. Scientific information generated at the APS has contributed to more than 100 different business projects and has mainly been obtained through the use of relatively straightforward diffraction, scattering, and spectroscopy experiments. The value of the synchrotron radiation source to us is derived from the brilliance, tunable wavelength, and collimation, which allow us to perform experiments that are generally not adequate or possible with laboratory-based x-ray equipment, including dynamic or *in situ* studies of materials.

This presentation will focus on several examples of how our experimental work at DND-CAT has been integrated into several interesting and successful research projects over the past few years, including real-time studies of chemical reactions and the deformation and processing of materials. The talk will close with several examples of how improved x-ray imaging methods, made possible with the proposed APS Renewal, would be expected to provide additional value to programs in the areas of nano-material and photo-voltaic device development.

APS Renewal Focus: User Visions, Part 1

A Path Forward to Advanced Nuclear Fuels, Founded upon a Unique and Crucial New Facility at the APS*

J. G. Tobin
Lawrence Livermore National Laboratory (tobin1@llnl.gov)

In a break with past paradigms, the U.S. Department of Energy has proposed a novel approach to the development of advanced nuclear fuels: predictive numerical simulation. [1] The advent of massive parallel computing and other improvements in computation capabilities has opened the door to the possibility of simulating much of the work that would have necessarily been determined empirically in the past. Nevertheless, these simulations and projections require the input of fundamental physical parameters that are experimentally generated or at least benchmarked, as well as computational electronic structure models that accurately describe 5f electronic systems. In particular, there are two crucial problems that must be resolved: (1) a dearth of fundamental thermodynamic

information and (2) an absence of an understanding of 5f electron correlation. To remedy the first malady, we propose a radical departure from past practices of calorimetry. Using the techniques first proposed by Martensson and Johansson [2] and then validated by Steiner et al. [3], we will use spectroscopically determined core level shifts to benchmark the computationally generated heats of solution. To resolve the second issue, we propose the pursuit of Fano spectroscopy [4] of the actinides, [5] including the minority transuranic daughter products so crucial to the reprocessing of nuclear fuels.

To truly resolve both of these sets of problems, a novel, dedicated Actinide Science Beamline is required, with full capabilities for handling highly radioactive transuranics, with exposed surfaces *in vacuo*, and high-brightness, high-resolution, circularly polarized soft x-rays in the hv = 100 to 500 eV range. The APS, where the possibility of a low-field device on a high-energy ring exists, is the best choice for such a synchrotron radiation source. Moreover, by utilizing the long beamline architecture of high-resolution beamlines, it will be possible to isolate the radioactive materials handling endstations in an adjacent building separated from the other APS beamlines.

**Based upon LLNL-PROP-404276.*

Lawrence Livermore National Laboratory is operated by Lawrence Livermore National Security, LLC, for the U.S. Department of Energy, National Nuclear Security Administration under Contract DE-AC52-07NA27344. This work was supported by the DOE Office of Basic Energy Science

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4. J.G. Tobin, S.W. Yu, T. Komesu, B.W. Chung, S.A. Morton, and G.D. Waddill, EuroPhys. Lett. 77, 17004 (2007).
5. S.W. Yu, J.G. Tobin, and P. Söderlind, J. Phys. Cond. Matter 20, 422202 (2008), Fast Track Comm.

High-field Magnets for X-ray Scattering Studies of Materials

Zahirul Islam and Jonathan C. Lang
Argonne National Laboratory

Studies of materials in extreme environment such as high magnetic fields emerged as a grand challenge science from a series of Basic Energy Science workshops, which have laid down the framework for future materials physics research directions funded by the DOE. The National Research Council report on opportunities in high field science (2005) and the CMMP2010 report have independently identified a national need for the development and use of high-field magnet instruments at national user facilities such as the Advanced Photon Source (APS) as well. We propose two complementary sets of extremely high-field magnets as a part of APS renewal: (1) 14-18 Tesla cryogen-free superconducting magnets; (2) 60 Tesla pulsed magnets. In both cases a split-pair and a solenoid design are envisioned. These new instruments will enable scattering and spectroscopic studies of numerous materials in extreme environment and usher in novel studies of meta-stable states of matter. The users of magnet laboratories and researchers at universities, national laboratories, and neutron facilities will greatly benefit from synchrotron x-ray studies of structural and magnetic effects induced by magnetic fields. Proposed instruments will ensure U.S. leadership in cutting-edge high-field science at a synchrotron.

Simultaneous Real-space Imaging and Diffraction Studies of Nano-scaled Structures

Hawoong Hong[1], Paul Zschack[1], Goran Karapetrov[2], Tai-Chang Chiang[3]
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[2] Materials Science Division, Argonne National Laboratory
[3] University of Illinois at Urbana-Champaign

The major focus of surface science has now been shifted to nano-scale structures such as quantum dots/wires and islands/clusters. These nanostructures can be self-assembled through the effects of electronic quantum confinement, lattice strain, and/or thermodynamics and kinetics. By controlling deposition and annealing conditions or through manipulating interface characteristics, the structure and properties of these nanostructures can be deliberately engineered. There are many examples of self-assembled nano-structures, such as quantum dots in semiconductor heteroepitaxy, metal nano-wires on semiconductors, islands of metal quantum wells, etc. In addition, metal catalysts on oxide supports are very relevant in connection with energy storage and require this line of research for comprehensive understanding. High-brilliance x-ray sources enable surface diffraction to provide unique capability for determining the atomistic structures in these various systems. X-rays are penetrating and have an additional advantage as tools for *in situ* and real-time analysis. However, the real space observation of morphology is also crucial in these studies. Since STM/AFM is the most dominant and versatile technique as a real space measurement tool, combining these two techniques to realize simultaneous measurements in real-space and in reciprocal-space without moving the sample position or changing the sample environment, could be game-changing. The full capability of a diffractometer on an undulator beam line, together with STM/AFM capability in a UHV chamber with MBE functions will allow *in situ* real-time studies of emergent properties in new, nano-scale structures.

Biological Imaging at the APS: Imagining the Future

Lydia Finney
Argonne National Laboratory

Our understanding of biological systems is at a point of rapid expansion, and imaging is one of the many technologies enabling this growth. As we look forward to the Renewal of our facility and beyond, many of our planned improvements will work together in a complementary way to further accelerate life science. Examples of these proposed developments, including improvements in both our ability to visualize biological specimens at nanometer scale, and to visualize them in the frozen-hydrated state, promise to bring new vision to our understanding of life. The potential of these capabilities to impact new science will be discussed.

APS Renewal Focus: Techniques

Coherent Diffraction and X-ray Imaging

Keith A Nugent
ARC Centre of Excellence for Coherent X-ray Science, The University of Melbourne, Vic., 3010, AUSTRALIA

Coherent diffractive imaging is a rapidly developing technique with a number of emerging applications (e.g., [1]). In this talk I will review progress in the methodology, exploring how it can be applied to general extended objects using the ptychographical [2] and keyhole [3] approaches. I will also discuss methods that incorporate and use the less-than-perfect spatial coherence available from third-generation synchrotron sources.

1. Barty A, Marchesini S, Chapman HN, Cui C, Howells MR, Shapiro DA, Minor AM, Spence JCH, Weierstall U, Ilavsky J et al: Three-dimensional coherent x-ray diffraction imaging of a ceramic nanofoam: Determination of structural deformation mechanisms. *Physical Review Letters* 2008, **101**(5):055501.
2. Rodenburg JM, Hurst AC, Cullis AG, Dobson BR, Pfeiffer F, Bunk O, David C, Jefimovs K, Johnson I: Hard-x-ray lensless imaging of extended objects. *Physical Review Letters* 2007, **98**(3):034801.
3. Abbey B, Nugent KA, Williams GJ, Clark JN, Peele AG, Pfeifer MA, De Jonge M, McNulty I: Keyhole coherent diffractive imaging. **Nature Physics** 2008, **4**(5):394-398.

X-ray Microscopy: Pictures of Complexity

Chris Jacobsen
Stony Brook University

Advances in x-ray optics and imaging techniques are providing higher resolution views of natural systems and manufactured materials. This presentation focuses on another advance: being able to view chemical bonding motifs and trace elements even in complex specimens, and to be able to place this information in structural context. Picturing complexity of course requires advances in imaging methods, but it also demands multimode signal detection and computational analysis to yield an image that is not just sharp, but informative!

Advanced X-ray Detector Development at NSLS

D. Peter Siddons
Brookhaven National Laboratory

Several detector development projects are underway at Brookhaven National Laboratory's NSLS. These projects are close collaborations between NSLS and BNL's Instrumentation Division, and address various synchrotron radiation techniques such as diffraction, spectroscopy and imaging. They are all based on custom microelectronics for high-density readout and custom sensors fabricated in-house at BNL. The talk will describe selected examples of these developments.

Ultrafast X-ray Spectroscopy of Solvated Transition-metal Complexes and Oxide Materials

Robert Schoenlein
Lawrence Berkeley National Laboratory

Understanding the complex interplay between atomic structure and electronic structure and properties is a fundamental challenge in condensed matter, particularly for correlated materials and molecular complexes exhibiting strong coupling between charge, spin, orbit, and lattice/vibrational degrees of freedom. Time-resolved measurements provide a new window to this problem by enabling the study of coupled interactions on time scales shorter than the underlying correlations. Ultrafast x-ray techniques are emerging as a powerful complement to ultrafast visible lasers, providing detailed information on both atomic dynamics, and valence charge dynamics. This talk will focus on recent time-resolved x-ray measurements of photo-induced phase transitions in transition-metal oxides and solvated transition-metal molecular complexes that reveal new information on the underlying physics.

APS Renewal Focus: User Visions, Part 2

Picosecond Temporal Resolution for Plasmonic and Acoustic Properties in Nanomaterials

Yuelin Li
X-ray Science Division, Argonne National Laboratory

Nanomaterials have wide application, particularly for energy science. In thermal electric applications, the acoustic properties of nanomaterials seem to be very promising for transmitting electrons but scattering phonons. It was also found that the coupling between plasmonic and acoustic oscillation plays a key role under certain circumstances. The characteristic time in those systems is dependent on the size of the nanoparticles and ranges from a few picoseconds to 100 ps. These have only been studied using optical/pump-probe experiment in which the optical probe may excite plasmonic/acoustic oscillation by itself. GISAXS provides a cleaner and more direct way of looking at the oscillation of the nanoparticles, but a higher temporal resolution is needed. Thus it is important to develop picosecond resolution techniques at the APS, either by shortening the x-ray pulse or developing detectors providing picosecond resolution.

Rapid, Ultra-fast Time-resolved Laue Data Collection from Tiny Crystals after Homogenous Reaction Initiation

Marius Schmidt
University of Wisconsin-Milwaukee, Physics Department (m-schmidt@uwm.edu, 414-229-4338)

Ultra-fast laser pump pulses to initiate reactions in protein crystals cannot be used to date, since the pulses do not penetrate deeply enough into the material, presumably due to two-photon absorption or other effects that make the crystals virtually black. Consequently, reaction initiation is very poor and time-resolved difference maps are usually empty when laser pulses a few pico-seconds long are employed. To circumvent this issue, small crystals a few micrometers in size must be used. Such crystals can be illuminated homogeneously even with ultra-fast laser pulses. Then, however, the scattering volume may become too small to collect a diffraction pattern with the existing synchrotron sources. At the moment, crystals with edge length of 100 μm are routinely used for time-resolved crystallographic experiments. The reduction of crystal edge lengths by one order of magnitude to 10μm also requires that the x-ray flux be increased by one order of magnitude to collect comparable time-resolved diffraction patterns, provided the beam can be focused to 10 × 10 μm^2. In this case however, potentially, only one diffraction pattern can be collected per crystal because of radiation damage. Multiple crystals must be brought successively and rapidly into the beam (see fig. 1). Then single-digit picosecond time-resolved experiments with homogenous reaction initiation will become feasible. We need to increase the x-ray flux in a single polychromatic pulse (in the hybrid mode) from 1011 to 1013 per pulse and decrease the duration of that pulse to single-digit picoseconds. By doing so, we will close the gap between the X-FELS and the synchrotrons available today.

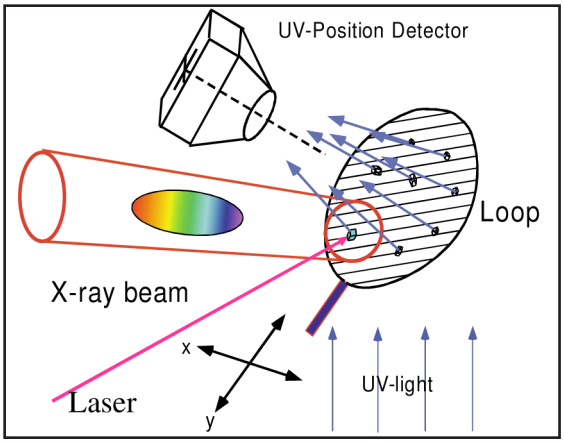


Fig. 1. Rapid scan mechanism to bring the tiny crystals in the X-ray beam. The reaction is initiated by a femtosecond or picosecond laser pulses. Either UV light or image processing can be used to determine the exact relative position of the tiny crystals.

Ultra-fast Polarization Phase Selective (PPS) Measurements at APS

Dr Kresimir Rupnik
Department of Chemistry, LSU Baton Rouge, LA 70803 (chrupn@lsu.edu)

Tailoring electromagnetic field structure *ad breve tempus* by different physical objects has been identified as one of the most productive ways to image as well as to control physical processes. Although among the most difficult methods, combined PPS studies can reveal new selective and detailed information about electronic and nuclear processes from single molecules to large biomolecular systems and materials. Methods such as magneti circular dichroism can be applied to all systems and were shown to be very useful, but they are limited by instrumental difficulties. Our nuclear vibrational resonance spectroscopy studies at APS have not yet included PPS techniques. We have recently conducted PPS studies at National High Magnetic Field Laboratory ("PPS Studies," K. Rupnik, PI) of physico-chemical processes of significant interest to biomedicine and sub-nano material architecture. These studies indicate (1) that many of the obstacles to the PPS measurements can be removed and processes at much faster time scales can be observed, and (2) there are extremely promising new results in the areas where "no enzyme has gone before." We discuss here the work in the same area that can be done at APS, a higher generation light source.

Hard X-ray *in situ* Real-time Interfacial-structural-spectro-nanoscopy

Paul Fenter
Argonne National Laboratory

Heterogeneity is a key challenge for interfacial science that is a key element controlling the reactivity of realistic interfaces and offers a rich landscape of phenomena. I will describe some examples of novel opportunities that can be achieved through a fully optimized photon-in, photon-out interfacial x-ray "nanoscope" that could be realized through the APS renewal.

WORKSHOPS AGENDAS AND ABSTRACTS

Workshop 1:
Detectors for the Future

Wednesday, May 6, 2009, afternoon
Bldg. 402, Lecture Hall

Organizers: Antonino Miceli and Patricia Fernandez (both APS)

The APS Renewal project will likely involve substantial investment in cutting-edge commercial x-ray detectors and in a detector R&D program: about 80% of the APS Renewal beamline proposals and 100% of the science case reports explicitly mention additional commercial detectors and/or the development of novel detectors as essential enablers for science. The aims of this workshop are to present and refine the current plans for detector work in the context of the APS Renewal and to explore current and future developments in x-ray sensors and detectors.

1:30 – 1:35 Welcome and Introduction

1:35 – 2:00 Detectors in the APS Renewal
Antonino Miceli and Patricia Fernandez, Argonne National Laboratory

2:00 – 2:30 X-ray Detector Developments at NSLS
Peter Siddons, Brookhaven National Laboratory

2:30 – 3:00 Radiation Detector Activities of the SLS Detectors Group
Beat Henrich, Paul Scherrer Institut

3:00 – 3:15 Coffee break

3:15 – 3:45 The Development of Large-area Picosecond-resolution Detectors
Henry Frisch, University of Chicago and Argonne National Laboratory

3:45 – 4:15 Microwave Kinetic Inductance Detectors for X-ray Science
Ben Mazin, University of California, Santa Barbara

4:15 – 4:45 Single- and Multi-element Drift Detectors for Specialized X-ray Applications
Shaul Barkan, SII NanoTechnology USA Inc.

4:45 – 5:00 Open discussion

WK1

Detectors in the APS Renewal

Antonino Miceli and Patricia Fernandez
Argonne National Laboratory

The procurement and development of advanced x-ray detectors will play a significant role in the APS Renewal. A wide majority of proposed Renewal beamline projects require state-of-the-art detectors, both from commercial suppliers and resulting from R&D efforts. This talk will summarize the detector requests related to the APS Renewal and will present a proposed strategy to address these requests.

WK1

X-ray Detector Developments at NSLS

Peter Siddons
Brookhaven National Laboratory

Several detector development projects are underway at Brookhaven National Laboratory's NSLS. These projects are close collaborations between NSLS and BNL's Instrumentation Division, and address various synchrotron radiation techniques such as diffraction, spectroscopy and imaging. They are all based on custom microelectronics for high-density readout and custom sensors fabricated in-house at BNL. The talk will describe selected examples of these developments.

WK1

Radiation Detector Activities of the SLS Detectors Group

Beat Henrich
Paul Scherrer Institut

Our research activities in novel X-ray detector systems which operate in single-photon counting mode have culminated in the foundation of DECTRIS (since 2006). Thus, this technology has become commercially available. Although this split meant a significant loss of knowledge, we continued our developments and activities towards the successor for the PILATUS chip, the Pilatus XFS. We will show the key features and the current status of this development. Further developments of the MYTHEN strip sensors will be discussed.

Knowing the constraints of single photon counting detectors, we have started to develop integrating detectors which will be inevitable for the upcoming XFEL experiments. In collaboration with DESY, the Universities of Bonn and Hamburg, we have started the AGIPD (Adaptive Gain integrating Pixel Detector) project. We will show first results and the current status of this project.

WK1

The Development of Large-area Picosecond-resolution Detectors

Henry Frisch
University of Chicago and Argonne National Laboratory

We are working on developing a photo-detector module to be used in applications requiring the coverage of areas of many square meters with time resolutions less than 10 psec and position resolutions of less than a millimeter for charged particles. The source of light for charged particle detection is Cherenkov light in a radiator/window; the amplification is provided by panels of micro-pores functionalized to act as micro-channel plates (MCPs). The good time and position resolution stem from the use of an array of parallel 50-ohm transmission lines as the collecting anodes. These anode strips feed multi-GS/sec sampling chips which digitize the pulse waveform at each end of the strip, allowing a measurement of the time from the average of the two ends, and a 2-dimensional position measurement from the difference of times on a strip, and, in the orthogonal direction, by the location of the strip. The module design is constructed so that large areas can be `tiled' by an array of modules.

WK1

Microwave Kinetic Inductance Detectors for X-ray Science

Ben Mazin
University of California, Santa Barbara

Microwave Kinetic Inductance Detectors, or MKIDs, were invented in 1999 as a way to overcome the multiplexing issues that many low temperature detectors face. After significant development, we now understand the devices well and are confident we can design and fabricate detectors with the required sensitivity for ground and space science from the millimeter to the X-ray. We have also made significant progress on readouts capable of being scaled to high pixel counts. This talk is designed to be a tutorial on basic MKID technology, with a specific focus on the issues involved in adapting MKIDs for use with a synchrotron X-ray light source.

WK1

Single- and Multi-element Drift Detectors for Specialized X-ray Applications

Shaul Barkan[1], Valeri D. Saveliev[1], Liangyuan Feng[1], Masanori Takahashi[1], Elena V. Damron[1], Carolyn R. Tull[1], and Nestor J. Zaluzec[2]
[1] SII NanoTechnology USA, Northridge, CA 91324
[2] Electron Microscopy Center, Argonne National Lab, Argonne, IL 60439

A 50 mm^2 silicon drift detector (SDD) has been successfully applied to XRF, TXRF, synchrotron and microanalysis applications. The SDD offers a large solid angle, excellent energy resolution, and high count rate performance. Several unique 4-element systems have been developed and are currently being used in high count rate synchrotron applications at the Advanced Photon Source at Argonne National Laboratory. We are pursuing several approaches in its development plan to achieve the required improved performance in the following areas.

A. One goal is to reduce entrance window thickness: a shallower entrance window results in a better peak-to-background (P/B) ratio, as well as better low energy detection. A significantly improved P/B ratio, as well as the detection of C, B and Be, has been achieved with the new shallower entrance window SDD.

B. A thicker device would enable the detector to be more efficient at higher energy. The current device's thickness is 0.35 mm with an efficiency of 0.28 and 0.08 for 20 keV and 30 keV, respectively. A recent experiment with a 1-mm-thick SDD showed an efficiency that matches the theoretical values of 0.6 and 0.4 for 20 keV and 30 keV, respectively

C. The characterization of nanoscale materials at high spatial resolution has become increasingly important to state-of-the-art research. A number of instruments ranging from Argonne's sub-angstrom electron-optical instrument to the X-ray Nanoprobe are capable of extraordinary resolution to study these materials. A special 50 mm^2 SDD with a larger solid angle for use in nanotechnology characterization was designed and assembled into an SEM at the Electron Microscopy Center at Argonne National Laboratory.

D. A detector array, larger than 4 elements, is a current project at SNTUS. Several designs are being considered: a monolithic device composed of seven elements, an array composed of single elements in a one vacuum tube, and an array composed of multiples of 4-element devices.

Workshop 2:
Optics for the Future

Wednesday, May 6, 2009, morning
Bldg. 402, Lecture Hall

Organizers: Albert Macrander, Yuri Shvyd'ko (both Advanced Photon Source)

The Optics for the Future Workshop will address hard x-ray optics developments worldwide that are likely to impact beamline designs. The topics to be covered include K-B mirror focusing, influences on beam coherence, and high-energy-resolution optics for inelastic x-ray scattering.

8:40 – 8:45 Introductory Remarks

8:45 – 9:15 A Focusing Monochromator with Extreme Energy Resolution
Alexander Chumakov, ESRF, Grenoble, France

9:15 – 9:45 Development of Ultra-high-resolution X-ray Optics for Inelastic X-ray Scattering at NSLS-II
Yong Cai, NSLS-II, Brookhaven National Laboratory, USA

9:45 – 10:15 A Novel Spectrometer for Inelastic X-ray Spectroscopy with Micro-eV Resolution
Ralf Roehlsberger, DESY, Hamburg, Germany

10:15 – 10:45 Coffee Break

10:45 – 11:15 Precise Characterization of an X-ray Nanobeam
Hidekazu Mimura, Osaka University, Japan

11:15 – 11:45 Analysis of Coherence Properties of Third-generation Synchrotron Sources and Free-electron Lasers
Ivan Vartaniants, HASYLAB/DESY, Hamburg, Germany

11:45 – 12:15 The Coherent Imaging Instrument at the Linac Coherent Light Source
Sébastien Boutet, LUSI/LCLS/SLAC, Menlo Park, CA

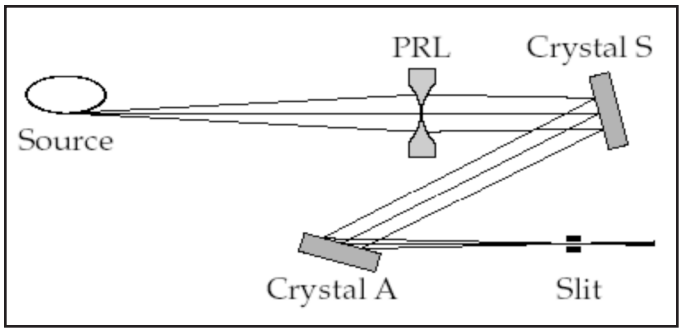
12:15 Workshop concludes

WK2

A Focusing Monochromator with Extreme Energy Resolution

V. G. Kohn[1], A. I. Chumakov[2], and R. Rüffer[2]
[1] Russian Research Center, Kurchatov Institute, 123182 Moscow, Russia
[2] ESRF, B.P.220, 38043 Grenoble

We present a concept of focusing monochromator with very high energy resolution. Different to conventional optical schemes, the device exploits not the angular but the spatial dispersion of synchrotron radiation. The monochromator can reach an energy resolution of about 0.1 meV without significant loss of the spectral density of synchrotron radiation [1].



The monochromator consists of four elements: a focusing parabolic refractive lens (PRL); a crystal in a symmetric reflection (Crystal S), which serves to keep the direction of the beam delivered by the monochromator parallel to the incident beam; a crystal in an asymmetric reflection to provide spatial dispersion of radiation components with various energies (Crystal A); and a slit to select the chosen energy band.

To obtain high energy resolution, the distance from the source to the lens should be as long as possible. The focal length of the lens should be chosen slightly smaller than the distance from the source. Then the lens focuses the beam almost to infinity, providing a very long focal distance. Under these conditions, the lens acts as a collimator, delivering the beam with a narrow angular spread. This strategy allows one to avoid losses of intensity, because the divergence of the beam is smaller than the angular acceptance of the asymmetric refection. The asymmetric high-order Bragg refection has the incidence angle larger than the exit one. This provides highest angular dispersion; i.e., waves with various energies exit the crystal at most different angles.

Despite the long focal distance, the demagnification ratio is big, because the asymmetric reflection shortens the distance to the focal point, providing a small focal spot. On the other hand, due to the high angular dispersion the radiation components with various energies are focused at sufficiently far separated points. The highest possible energy resolution is determined by the ratio of the size of the focus for a monochromatic wave to the spatial dispersion of the radiation of different energies.

The focusing monochromator operates with much smaller crystals than traditional monochromators. Therefore, this approach is less sensitive to a possible inhomogeneity of the silicon lattice constant. It solves two tasks at once: i.e., it prepares (i) high-monochromatic and (ii) focused radiation beam. Finally, the focusing monochromator allows one to change the energy bandwidth by a simple change of the slit size. Thus, researchers obtain a useful option of, e.g., a fast preview of phonon spectra with moderate resolution but high count rate before longer and more accurate high-resolution measurements.

[1] V.G. Kohn, A.I. Chumakov, and R. Rüffer, Journal of Synchrotron Radiation, in press.

WK2

Development of Ultrahigh-Resolution X-ray Optics for Inelastic X-ray Scattering at NSLS-II

Yong Cai
National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973

Inelastic x-ray scattering (IXS) is a powerful technique for studying dynamics and excitations in condensed matter systems and has been used to study excitations ranging from phonons in solids, to sound modes in liquids and polymers, to plasmons in simple metals, to complex electronic excitations in strongly correlated electron systems. One of the most important goals of the National Synchrotron Light Source II (NSLS-II) project is to achieve ultrahigh energy and momentum transfer resolution for inelastic x-ray scattering. Currently, IXS spectrometers with practical energy resolutions of > 1 meV used at all high-energy synchrotron sources rely on the use of Bragg back reflections from perfect crystals. To pursue higher resolution, one has to use high-order Bragg reflections with higher photon energies. This approach is incompatible with undulator-based synchrotron sources, which generate much less flux in the high-energy spectral range.

In this paper, we present progress at NSLS-II in developing completely new x-ray optics based on the angular dispersion effect of x-ray diffraction in extremely asymmetric Bragg back reflections [1], and the current design of an instrument that utilizes these optics for inelastic x-ray scattering experiments. These new optics provide the possibility to achieve sub-meV energy resolution with sharp tails and high efficiency at medium energy range around 9 keV, and therefore may provide a real alternative to current state-of-the-art Bragg back reflection optics for many medium-energy synchrotron sources.

[1] Yu. Shvyd'ko et al., "X-ray Bragg diffraction in asymmetric backscattering geometry," Phys. Rev. Lett. 97, 235502 (2006).

WK2

A Novel Spectrometer for Inelastic X-ray Spectroscopy with Micro-eV Resolution

Ralf Röhlsberger
DESY, Hamburg, Germany

A new kind of spectrometer is introduced for inelastic x-ray spectroscopy that allows for energy resolutions in the range of 10 μeV and an energetic tunability of several meV. It relies on nuclear resonant scattering from fast rotating samples. At rotational speeds of several 10 kHz, resonantly scattered photons experience an angular deflection of several mrad off the primary beam. The deflected photons can be Doppler tuned by transverse displacement of the rotor relative to the incident beam. This secondary beam can be used to probe vibrational dynamics with μeV resolution.

Interesting areas of research are materials with low-energy excitations such as soft matter and artificially structured materials. Recent experiments and future applications are discussed.

WK2

Precise Characterization of an X-ray Nanobeam

Hidekazu Mimura
Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan
Phone: +81-6-6879-7286, Fax: +81-6-6879-7286
E-mail: mimura@prec.eng.osaka-u.ac.jp

1. Introduction

We present a remarkably accurate method for determining the wave field of an x-ray nanobeam. The intensity profile of a beam was directly measured over a range of three orders of magnitude while its phase distribution was successfully recovered using an iterative algorithm.

2. Precise measurement of intensity profile for x-ray nanobeam

Hard x-ray beams having diameters smaller than 50 nm have been achieved using several optical systems. Precise measurement of the beam's profile near the focus is becoming quite difficult as spot sizes decrease. Thus, a method for accurately analyzing the x-ray nanobeams is strongly desired. Figure 1 shows a schematic diagram of the method employed in this study. When an object is inserted in the path of a propagating light beam, the wave field of the light behind the object can be expressed as the sum of the geometrical optics field and the diffraction field. In the case under consideration, the latter is a cylindrical wave diffracted from the edge of the object (see Figs. 1(b) and (c)). The intensity of the cylindrical wave is assumed to be proportional to the intensity of the propagating light that illuminates the edge. The object inserted into the beam (see Fig. 1) had the microbridge structure shown in Fig. 2, which was produced by electron beam deposition and focused ion beam fabrication. This microbridge is made from platinum and is approximately 2 μm wide and 50 μm long. A precisely shaped x-ray mirror with an elliptical profile was used to generate the x-ray nanobeam. The mirror was fabricated using elastic emission machining in conjunction with an interferometric surface profiler. An experiment to analyze the line-focused beam using the microbridge structure was performed at the 1-km beamline of SPring-8. Figure 3 shows a semi-logarithmic graph of the intensity distribution profile measured in the focal plane. The focal width was found to be 32 nm and this value is in good agreement with the theoretical value of 30 nm. The logarithmic plot shows that the third-order satellite peak structures are distinguishable. Figure 4 shows the figure error profile calculated using phase retrieval methods using only this intensity profile, together with the one measured using an interferometric surface profiler after mirror fabrication. The two profiles are in good agreement within a 1-nm level, corresponding to a wavefront error of 0.15 wave. The wave-field distribution along the beam propagation direction near the focus can be numerically simulated by calculating the Fresnel-Kirchhoff integral, using the recovered surface profile shown in Fig. 4.

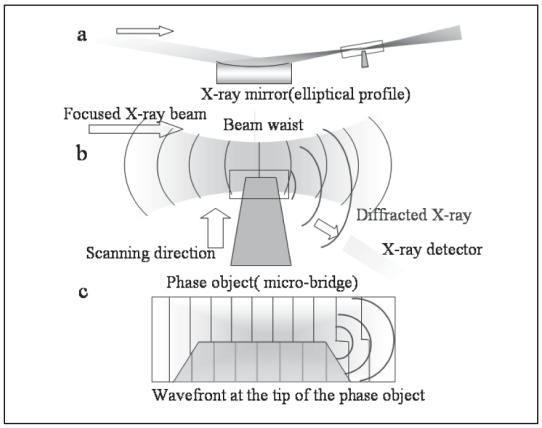


Fig. 1. Schematic diagram of x-ray intensity distribution sensing system using a microphase object (microbridge structure).

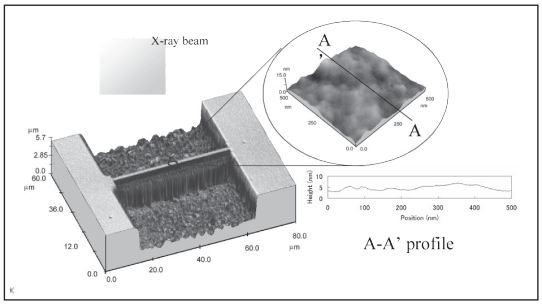


Fig. 2. Microbridge structure used for measuring the intensity distributions of a hard x-ray line focused beam.

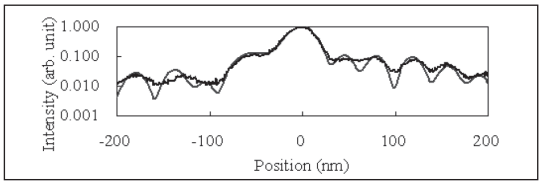


Fig. 3. Results of measuring intensity profiles in the focal plane and phase retrieval calculations.

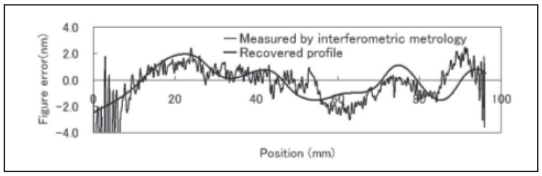


Fig. 4. Comparison of measured and reconstructed figure error profiles.

3. Discussion

To date, methods of evaluating x-ray nanobeams have been preceded by the development of focusing optics. It is anticipated that the method presented in this paper that uses a microbridge structure will be widely applicable for evaluating all x-ray focusing devices. In particular, information concerning the complex phase and intensity near the focus is indispensable for x-ray free-electron laser applications. This x-ray intensity measurement method, combined with phase retrieval calculations, promises to contribute to a breakthrough in the development of advanced x-ray focusing optics and various investigations of x-ray microscopy.

This research was partially supported by a Grant-in-Aid for Specially Promoted Research 18002009, 2009 and Global COE program, 2009 from the Ministry of Education, Culture, Sports, Science, and Technology of Japan. The use of BL29XU of the SPring-8 was supported by RIKEN.

Reference:

H. Mimura, H. Yumoto, S. Matsuyama, S. Handa, T. Kimura, Y. Sano, M. Yabashi, Y. Nishino, K. Tamasaku, T. Ishikawa, K. Yamauchi, Direct Determination of the Wave Field of an X-ray Nanobeam, *Physical Review A* **77**, 015812 (2008).

WK2

Analysis of Coherence Properties of 3-rd Generation Synchrotron Sources and Free-Electron Lasers

I. A. Vartaniants
HASYLAB at DESY, Notkestr., 85, D-22607 Hamburg, Germany

A general theoretical approach based on the results of the statistical optical theory is used for the analysis of the transverse coherence properties of the third-generation synchrotron sources and x-ray free-electron lasers (XFEL). Correlation properties of the wavefields are calculated at different distances from an equivalent Gaussian-Schell model source. For the third-generation synchrotron sources, analysis is performed for the parameters of the PETRA III storage ring that is presently under construction. In the case of XFEL sources the decomposition of the statistical fields into a sum of independently propagating transverse modes are used for the analysis of the coherence properties of these new sources. A detailed analysis was performed for the parameters of the SASE1 undulator at the European XFEL. Analysis has shown that only few modes contribute substantially to the total radiation field of that source.

WK2

The Coherent Imaging Instrument at the Linac Coherent Light Source

Sébastien Boutet
Linac Coherent Light Source, SLAC National Accelerator Laboratory

The Linac Coherent Light Source (LCLS), coming on-line in the summer of 2009, will become the first-ever hard x-ray free-electron laser (FEL). It will utilize the 13-GeV electron beam from the last kilometer of the 3-km-long linear accelerator on the site of the SLAC National Accelerator Laboratory to produce hard x-ray pulses containing 1012 photons or more in less than 100 femtoseconds. The incredible brightness of this new source will make it a unique tool with unexplored discovery potential. Among the novel scientific opportunities hard x-ray FELs offer, the possibility of obtaining snapshot images of single non-periodic objects, especially biological samples, has generated a tremendous level of excitement. The extremely short duration and high intensity of the x-ray beam promises to minimize radiation damage to biological samples during the pulse and to allow high-resolution imaging of non-crystalline objects.

In this talk, I will discuss the LCLS and the status of its commissioning as the start of operations rapidly approaches. I will introduce the coherent x-ray imaging (CXI) instrument that will be used for single-shot imaging of any nanoparticle, including biological samples beyond the radiation damage limit. I will discuss the optical design of the instrument and how high-quality optics are crucial to the success of the experiments. Finally, I will discuss the scientific capabilities of the instrument and the timeline for its availability.

Workshop 3:
NextGen Nanopositioning: Engineering Robust Systems for Manipulation and Analysis

Wednesday, May 6, 2009, afternoon
Bldg. 440 (Center for Nanoscale Materials), Rm. 105/106

Organizers: Russell Cook (EMC), Curt Preissner (APS)

This workshop will bring together engineers and instrument scientists who are interested in advancing the state of the art in nanopositioning. The goal of this workshop is to discuss the engineering developments that will facilitate the goals of next-generation x-ray optics, nano-manipulation, and electron microscopy. It is meant to provide a forum for the three major user centers at Argonne (APS, CNM, and EMC) to share ideas in nanopositioning.

Nanopositioning is a key enabling technology for x-ray scanning microprobes, x-ray nanotomography, and lensless x-ray imaging. Advances in nanopositioning engineering need to be commensurate with expected advances in x-ray optics and electron microscopy. The x-ray nanopositioning problem requires a large dynamic range with motions on the order of millimeters, and future 10-nm optics will require 1-nm resolution. Nanotomography with 30 nm resolution requires a total rotational error of 15 nm. In addition, environmental control of the sample may be required from cryogenic temperatures to hundreds of degrees. The engineering of nanopositioning systems is best accomplished with a system-oriented approach, that is, through joint consideration of the mechanics, sensing, and controls aspects of the design.

Advances in electron microscopy such as TEAM (transmission electron aberration-corrected microscopy), in which sub-Ångstrom (or 0.05 nanometer) resolution is becoming the norm, and the drive towards in situ experiments (e.g., STM/TEM, tribology, and nanoindentation) will require nanopositioning systems to push towards picometer (per hour) stability, accuracy, and repeatability in very small volumes. Further, the necessity for nanoscale studies of other analytical techniques and the trend of shrinking scales in nanomaterials manipulation will require new solutions in nanopositioning.

1:30 – 1:35 Welcome and Introduction

1:35 – 2:15 Nanopositioning Techniques Development for Synchrotron Radiation Instrumentation at the APS: The Present and the Future
Deming Shu, Advanced Photon Source, Argonne National Laboratory

2:20 – 3:00 Robust Broadband Nanopositioning: A Control Systems Perspective
Srinivasa M. Salapaka, University of Illinois at Urbana-Champaign

3:05 – 3:30 Coffee break

3:30 – 4:10 MEMS Technology for Manipulation and Control of Optical Signals
Daniel López, Center for Nanoscale Materials, Argonne National Laboratory

4:15 – 4:55 A High-stability TEM Sample Stage with Five Degrees of Freedom
Thomas Duden, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory

5:00 Adjourn

WK3

Nanopositioning Techniques Development for Synchrotron Radiation Instrumentation at the APS: The Present and the Future

Deming Shu
Advanced Photon Source/AES, Argonne National Laboratory, Argonne, IL 60439

At the new synchrotron radiation sources and beamlines, such as the renewal project at the Advanced Photon Source (APS), high-precision positioning techniques present a significant opportunity to support the state-of-the-art synchrotron radiation research. Meanwhile, the requirements for instrument positioning performance and capabilities, such as resolution, dynamic range, repeatability, speed, and synchronization of multiple axes, are exceeding the limit of commercial availability. In this presentation, I will discuss the nanopositioning techniques developed for the APS/CNM hard x-ray nanoprobe and APS/XOR high-resolution monochromators. Future nanopositioning techniques to be developed for the APS renewal project will also be discussed.

WK3

Robust Broadband Nanopositioning: A Control Systems Perspective

Srinivasa M Salapaka
University of Illinois, Urbana-Champaign

This talk will focus on control systems theoretic analysis and synthesis of new modes of operations that significantly expand the range of performance specifications of nanopositioning systems. We will present a systems theory framework to study fundamental limitations on the performance of these devices. In particular, we will characterize the inherent fundamental trade-offs between resolution, tracking-bandwidth, and reliability specifications on positioning capability of these devices. In addition to determining fundamental limitations, this framework leads to a better understanding of existing technology and in overcoming some technological hurdles that were previously thought to be fundamentally limiting.

The analysis is done in robust optimal-control setting with various architectural constraints that arise typically in nanopositioning systems imposed on both one- and two- degrees of freedom control-design frameworks. We show design strategies for improving some common existing nanopositioning systems, making nanopositioning systems insensitive to modeling uncertainties, and feedback designs that achieve simultaneously high bandwidth, resolution and robustness to modeling uncertainties.

The outcomes of this research will be corroborated with experimental results on a nanopositioning system of an atomic force microscope. Experimental results that demonstrate significant improvements in bandwidth, resolution and robustness over common existing designs will be presented.

WK3

MEMS Technology for Manipulation and Control of Optical Signals

Daniel López
Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439

A variety of applications in micro- and nanotechnologies require mechanical systems capable of very complex operations with high precision and multiple degrees of freedom. Micro-electro-mechanical systems (MEMS) enable these applications by combining excellent mechanical and optical properties with dense integration of multiple actuators and sensors on a single chip.

In this talk I will describe the fundamentals and current engineering challenges of this technology. I will present several examples of MEMS devices to illustrate the common recurrent themes in device design. These are elastic elements and pure-flexure design (with no mechanical surface contact between moving parts), electrostatic actuation and residual stress engineering. I will attempt to show different ways these themes can manifest themselves and different approaches that can be used in understanding, modeling and utilizing them. Furthermore, I will describe examples of MEMS devices under development at Argonne National Laboratory for x-ray scanning control and manipulation.

WK3

A High-stability TEM Sample Stage with Five Degrees of Freedom

Thomas Duden, Andreas Schmid
National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94708

In the framework of the TEAM project (Transmission Electron Aberration-corrected Microscope), a high-stability stage was developed. It provides three translational and two rotational degrees of freedom. The stage is a retrofit to a commercial TEM and had to satisfy spatial constraints given by the instrument. Its piezo drives allow excellent fine control of the position, which is essential to obtain a large field of view in, for example, a tomographic series. Vibration measurements allowing a comparison with the commercially available side entry stage will be presented, together with recent results that illustrate the usability of the approach. Also, methods for position detection will be discussed.

Workshop 4:
High-Speed Imaging Opportunities with X-rays

Wednesday, May 6, 2009
Bldg. 402, Rm. E1100/E1200

Organizers: Kamel Fezzaa, Wah-Keat Lee, and Jin Wang (all APS)

High-speed imaging with visible light is a well-established technique. However, in many cases, visible light has severe limitations due to reflection, refraction, and multiple scattering. Especially in optically dense systems, visible light lacks penetration ability as opposed to x-rays.

Synchrotron-based ultra-high-speed x-ray imaging is being developed at the APS, utilizing the unique parameters of this source. It is proving to be a unique and invaluable tool to image the internal structures of optically opaque systems with micrometer spatial resolution and sub-nanosecond temporal resolution. Currently, we can take x-ray edge-enhanced images of fuel jets with 100-ps exposure times, see the motion of the internal parts of a stainless steel fuel injector, and make x-ray movies of the pinch-off singularity water droplets, all at a rate of tens of thousands of frames per second.

The fundamental insights this technique can provide are not otherwise possible and are much needed in many research areas dealing with ultra-fast and transient phenomena. Examples include dynamics of multiphase flows; materials behavior under extreme conditions, such as impact or explosion; complex materials mechanics; self-propagating reactions in multi-component systems; and many other applications that are of major importance to transportation safety, national security, energy efficiency, emissions control, and development of new materials.

To promote the science that can be facilitated with the ultrafast technique at the APS, the goals of this one-day workshop are (1) to present the current capabilities this nascent technique, (2) to examine its applicability and explore its utility to existing unsolved scientific questions in a much broader community, and (3) to discuss new possibilities and identify directions for future developments.

8:55 – 9:05 Welcome and Introductory Remarks
Organizers

9:05 – 9:35 Current State of X-ray High-speed Imaging at the APS
Kamel Fezzaa, Advanced Photon Source

9:35 – 10:15 Current Challenges and Potential Application of High-speed X-ray Imaging to the Investigation of Solidification and Crystallization Dynamics
Ralph Napolitano, Iowa State University.

10:15 – 10:35 Coffee break

10:35 – 11:15 Life and Death of a Drop: What We Can Learn about Dynamic Singularities?
Sidney Nagel, University of Chicago

11:15 – 11:55 Gas-mediated Impact Dynamics on a Fine-grained Granular Bed
John Royer, University of Chicago

11:55 – 1:20 Lunch

1:20 – 2:00 Correlating the Spray Primary Breakup Process for DI Automotive Injectors to Their Design
Ming-Chia Lai, Wayne State University, Michigan

2:00 – 2:40 Study of Diesel Spray Primary Breakup
Rolf Reitz, University of Wisconsin, Madison

2:40 – 3:20 Fracture and Friction at Microscopic and Macroscopic Scales
Michael Marder, University of Texas, Austin

3:20 – 3:40 Coffee break

3:40 – 4:20 Investigation of Dynamic Failures in Metals using High-speed Photography and Infrared Thermograph
Guruswami Ravichandran, California Institute of Technology

4:20 – 5:00 Title unavailable
Steve Heister, Purdue University, Indiana

WK4

Life and Death of a Drop: What We Can Learn about Dynamic Singularities?

Sidney Nagel
The University of Chicago

The exhilarating spray from waves crashing into the shore, the distressing sound of a faucet leaking in the night, and the indispensable role of bubbles dissolving gas into the oceans are but a few examples of the ubiquitous presence and profound importance of drop formation and splashing in our lives. They are also examples of a liquid changing its topology. Although part of our common everyday experience, these transitions are far from understood and reveal delightful and profound surprises upon careful investigation. For example in droplet fission, the fluid forms a neck that becomes vanishingly thin at the point of breakup. This topological transition is thus accompanied by a dynamic singularity in which physical properties such as pressure diverge. Singularities of this sort often organize the overall dynamical evolution of nonlinear systems. I will first discuss the role of singularities in the breakup of drops. I will then discuss the fate of the drop after it falls and eventually splashes against a solid surface.

WK4

Gas-Mediated Impact Dynamics in Fine-Grained Granular Materials

John R. Royer[1], Eric I. Corwin[1], Bryan Conyers[1], Maria-Luisa Cordero[3], Mark L. Rivers[2], Peter J. Eng[1,2], and Heinrich M. Jaeger[1]
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[3] Departmento de Fisica, Universidad de Chile, Santiago, Chile

When a heavy sphere is dropped onto a bed of loose, fine sand, a large, focused jet of sand shoots upward. Experiments at reduced air pressure reveal that the jet in fact consists of two components: a wispy, thin jet that varies little with pressure followed by a thick air-pressure-driven jet. To observe the initial stages of jet formation inside the granular bed, we employ x-ray radiography using the high-intensity beams available at the Advanced Photon Source. This technique allows us to image the motion of the sphere through the bed and measure local changes in the bed packing density below the sphere. The x-ray movies reveal that gravity-driven collapse produces the initial, thin jet, while the compression of an air pocket trapped below the surface drives up the thick jet. We also find that the interstitial air alters the compressibility of the sand bed. In vacuum a visible compaction front precedes the ball, while at atmospheric pressure the sand flows out of the way of the ball, behaving more like an incompressible fluid.

Workshop 5:
Imaging Structural Hierarchy in Biological Systems

Wednesday, May 6, 2009
Bldg. 401, Rm. A1100

Organizers: Barry Lai (APS), Lee Makowski (Argonne National Laboratory), Gayle Woloschak (Northwestern University)

X-ray imaging of biomolecular systems is undergoing a revolution that will transform our understanding of biological systems at multiple length scales. X-rays have the unique capability of probing biological structures from centimeters to nanometers, often with thick samples and even in three dimensions. This workshop is designed to bring together researchers in the use of X-ray imaging to discuss the methods development and facilities upgrades that will be needed to apply the full range of imaging modalities to biological systems. The purposes of this workshop are (1) to highlight multi-length scale studies made possible by X-ray techniques, (2) bring together researchers to build the life science case for the APS Renewal, and (3) to explore synergy with other imaging modalities.

9:00 – 9:05 Welcome

9:05 – 9:35 X-ray Imaging Overview
Janos Kirz, Advanced Light Source

9:35 – 10:05 Biomedical Applications of Cellular and Structural Imaging
Lee Makowski, Argonne National Laboratory

10:05 – 10:35 Visualizing and Quantifying Fluid Flow and Respiratory Structures in Insects
Jon Harrison, Arizona State University

10:35 – 11:00 Coffee Break

11:00 – 11:30 X-ray Imaging of Bones, Teeth, and Regenerates
Thomas Diekwisch, University of Illinois at Chicago

11:30 – 12:00 Approaches to Molecular Imaging with X-ray Scatter-based Micro-CT
Erik Ritman, Mayo Clinic

12:00 – 1:30 Lunch

1:30 – 2:00 Multi-modality Image Analysis in Breast Cancer
Maryellen Giger, The University of Chicago

2:00 – 2:30 From the Cell to the Globe: What Synchrotron-based X-ray Microanalysis of Marine Bacteria and Protists can Tell Us about Biogeochemical Cycles
Stephen Baines, Stony Brook University

2:30 – 3:00 Inorganic Physiology: Subcellular Elemental Distributions in Normal and Pathological States
Tom O'Halloran, Northwestern University

3:00 – 3:20 Coffee Break

3:20 – 3:50 Probing Alterations in Bone Structure and Composition in Osteoporosis using Synchrotron-based Imaging
Lisa Miller, Brookhaven National Laboratory

3:50 – 4:20 Coherent Diffraction Imaging of Biomaterials
Ian McNulty, Argonne National Laboratory

4:20 – 5:00 Discussion and Wrap-up

Workshop 6:
Synchrotron Radiation in Chemical Science

Wednesday, May 6, 2009
Bldg. 401, Rm. A5000

Organizers: Karena Chapman (Advanced Photon Source, Argonne National Laboratory), Karen Mulfort (Argonne National Laboratory), Randall Winans (Advanced Photon Source, Argonne National Laboratory)

Overview
Applications of synchrotron radiation to problems in chemical science span the breadth of the characterization tools available at the Advanced Photon Source, and often a given chemical conundrum can only be addressed by combining the insights gleaned from several different methods. This workshop will highlight the opportunities for synchrotron-based research in chemical science by demonstrating its relevance in two topical areas of research: metal-organic hybrid materials and catalysis.

Metal-Organic Hybrid Materials
Metal-organic hybrid materials, which include metal-organic frameworks (MOFs) and related supramolecular systems, are an exciting, rapidly expanding area of materials research. This intense current interest is due to both the virtually unlimited variety of materials systems that can be synthesized and the wide range of potential applications afforded by their functional material properties, ranging from gas storage to catalysis to drug delivery. Central to the development of these metal-organic materials and associated technologies is the detailed characterization of new materials and the in-depth exploration of the structure-property relationship. In this endeavor, unsurpassed insights have been gained from the characterization tools available at the APS, which span microcrystallography and structural solution from powder, experiments under non-ambient conditions, small-angle scattering, pair distribution function methods, spectroscopy, and dynamics. As such, synchrotron-based techniques promise to play a decisive role in the advancement of metal-organic hybrid materials.

Catalysis
A grand challenge in energy research is the understanding of the mechanisms and dynamics of catalyzed transformations. Synchrotron X-ray approaches, when combined with other methods, can provide basic insight into these problems. Measurements in reaction environments are necessary but can be difficult because of the complex nature of the catalyst structure, high temperatures and pressures, and multiple reaction phases. A key issue is the characterization of solid catalysts with spatial resolution at length scales from macroscopic to atomic levels. In addition, temporal resolution needs range from fast electron transfer in the pico- and subpicosecond range to reaction rates in milliseconds to seconds. Spectroscopy has been the main tool in these studies and this workshop will explore new approaches in spectroscopy and expand to techniques in scattering.

8:55 – 9:00 Welcome

9:00 – 9:30 Complex Perovskites: Mining the Periodic Table for New Functional Materials
Patrick Woodward, Department of Chemistry, Ohio State University

9:30 – 10:00 Discovering Porous Metal-organic Framework Materials: From Micro-crystals to Methane Storage
Shengquin Ma, Chemical Sciences and Engineering Division, Argonne National Laboratory

10:00 – 10:30 A Multi-crystal Approach to Structure Solution and Refinement
Gavin Vaughn, European Synchrotron Radiation Facility

10:30 – 10:45 Coffee Break

10:45 – 11:15 Watching Chemistry Happen in Real Time: Insights from the PDF Method
Peter Chupas, X-ray Science Divsion, Argonne National Laboratory

11:15 – 11:45 Solid State NMR and Pair Distribution Function Studies to Investigate the Changes in Short-range Order in Silicon Anodes
Baris Key, Department of Chemistry, Stony Brook University

11:45 – 1:15 Lunch

1:15 – 1:45 Elucidating Structure-property Relationships in Functional Metal-organic Frameworks Using *in situ* Synchrotron-based Powder Diffraction
Gregory Halder, Materials Science Division, Argonne National Laboratory

1:45 – 2:15 High-throughput, *in situ*, and *operando* XAFS Spectroscopy from the Soft to the Hard X-ray Range
Sven Schroeder, University of Manchester

2:15 – 2:45 The Use of *in situ* Synchrotron-based Spectroscopic Methods to Characterize Functional Metal-organic Framework Materials
Carlo Lamberti, Department of Inorganic, Physical and Materials Chemistry, University of Turin

2:45 – 3:15 Coffee Break

3:15 – 3:45 Sulfur Poisoning in Rh, Ni Catalysts for Steam Reforming of Liquid Hydrocarbons
Yongshen Chen, Pennsylvania State University

3:45 – 4:15 *In situ* X-ray Scattering and Spectroscopy Studies of Model Nanocatalysts under Realistic Reaction Conditions: A New Approach for Understanding Size/Shape Effects on Catalytic Performance
Sungsik Lee, Chemical Sciences and Engineering Division, Argonne National Laboratory

4:15 – 4:45 Characterization of Photoactive Self-assembled Structures via Solution Small- and Wide-angle X-ray Scattering
Josh Vura-Weis, Department of Chemistry, Northwestern University

4:45 – 5:15 Combining Molecular Dynamics Simulations and Solution-phase X-ray Scattering to Investigate Conformational Changes
Kristy Mardis, Department of Chemistry and Physics, Chicago State University

5:15 Workshop concludes

WK6

Complex Perovskites: Mining the Periodic Table for New Functional Materials

Patrick M. Woodward
Department of Chemistry, Ohio State University

Compounds that belong to the perovskite family exhibit remarkable versatility, both in terms of their chemical composition and their physical properties. Not only can one prepare perovskites containing elements ranging from hydrogen to plutonium, the physical properties of perovskites are incredibly varied. Superconductivity, ferrolectricity, piezoelectricity, ferromagnetism, antiferromagnetism, colossal magnetoresistance, half-metallicity, proton conductivity—all these properties can be found in members of the perovskite family. In this talk I will discuss our studies of complex perovskites with a particular emphasis on atomic ordering over various length scales and its impact on the structure and properties of these materials.

WK6

Discovering Porous Metal-organic Framework Materials: From Micro-crystals to Methane Storage

Shengqian Ma
Chemical Sciences & Engineering Division, Argonne National Laboratory

Porous metal-organic frameworks (MOFs) represent a new type of functional materials and have recently been considered one of the most promising candidates for hydrogen and methane storage applications. Because the uptake of hydrogen or methane is usually directly related with the size, shape and volume of the pores as well as the geometry of the secondary building units and topologies of the porous MOF materials, it is thus of crucial importance to obtain the detailed structures of the porous MOF crystals. Albeit of their excellent hydrogen and methane storage performances, porous MOFs with huge unit cells or with small crystal sizes usually suffer from their structural determination when utilizing a Mo/Cu radiation source. Fortunately, with the assistance of synchrotron micro-single-crystal X-ray diffraction at ChemMatCARS beamline of APS, the structures of some porous MOFs with high hydrogen and methane uptakes have been determined, which will be very helpful for future design of porous MOFs with even higher hydrogen and methane storage capacities.

WK6

Solid State NMR and Pair Distribution Function Studies to Investigate the Changes in Short-range Order in Silicon Anodes

Baris Key[1], Rangeet Bhattacharyya[1], Mathieu Morcrette[2], Vincent Seznec[2], Jean-Marie Tarascon[2], and Clare P. Grey[1]
[1] Department of Chemistry, SUNY at Stony Brook, Stony Brook, NY, 11794
[2] LRCS, Universite de Picardie Jules Verne, 80039 Amiens, France

Conventional diffraction techniques provide limited information for systems that involve structural changes in amorphous phases, such as those that occur during the electrochemical lithiation of crystalline silicon in a lithium-ion battery (LIB). Local structure techniques are ideal methods for investigation of these reactions. Magic angle spinning solid state nuclear magnetic resonance (MAS-SSNMR) spectroscopy is an extremely useful tool to investigate the local structure of a wide range of crystalline and amorphous materials. Additionally the use of the high energies available at synchrotron sources has opened up the possibility of going beyond traditional diffraction experiments and performing high-resolution scattering experiments, such as pair distribution function (PDF) analysis, which can yield extremely valuable short-range structural information from complex materials [1]. Here, we demonstrate the use of various local structure probes, such as *in situ* and *ex situ* solid state NMR and *in situ* and *ex situ* X-ray PDF and analysis, to understand the structural changes that occur for bulk and nanoparticulate silicon anodes.

Crystalline silicon undergoes a crystalline-to-amorphous phase transformation in the beginning of the first discharge when it is cycled versus Li metal, as can be seen in Figure 1 (I). The long plateau at 0.09V vs. Li is associated with the formation of a lithiated amorphous (a)-Li_xSi. Larger particles then crystallize to form the metastable intermetallic $Li_{15}Si_4$ phase [2]. We investigated the lithiation of crystalline Si and the formation of the lithiated amorphous phase during the plateau between I and II and the formation of the recrystallized intermetallic metastable phase $Li_{15}Si_4$ at the end of discharge (III) by *ex situ* MAS-SSNMR. The discharged electrode spectra were compared with model

intermetallic compounds in Li-Si system in order to provide ^{7}Li and ^{29}Si peak assignments (Figure 2). The data obtained were compared with the *in situ* static SSNMR data of an actual working LIB, which allowed the local structures present in the first amorphous phase to be determined and then the changes in local structure to be followed. In addition, the *in situ* method revealed a second process at low voltages, which was not visible by using *ex situ* methods [3]. The combination of the two techniques was then used to investigate the processes that occur during the first charge and second discharge.

Ex situ PDF analysis of model intermetallic compounds and Si electrodes stopped at various charged/discharged stages showed changes in Li-Li, Si-Si and Li-Si distances, and could be used to follow the transformation from crystalline to amorphous phases (Figure 3). By comparing the interatomic distances with those found in model intermetallic compounds, local structures for the amorphous phases were proposed. The local structures of the deep discharge and the charged stages were identified. A preliminary fitting was performed to explain the change in Si-Si correlations (2.35 and 3.8Å).

This work was supported by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies of the U.S. DOE under Contract No. DE-AC03-76SF00098, via subcontracts No. 6517748 and 6517749 with the Lawrence Berkeley National Laboratory. This work has benefited from the use of the Advanced Photon Source (APS) at Argonne National Laboratory. We thank Peter Chupas, Karena Chapman and Evan Maxey for their help in data collection at the APS.

References:

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[2] M. N. Obrovac, L. Christensen, *Electrochem. Solid-State Lett.*, **7**, A93, 2004.

[3] B. Key, R. Bhattacharyya, M. Morcrette, V. Seznec, J.-M. Tarascon, C. P. Grey, *J. Am. Chem. Soc., in press* Figure 1. Electrochemical profile of first discharge of crystalline silicon and carbon SP composite electrode vs. Li.

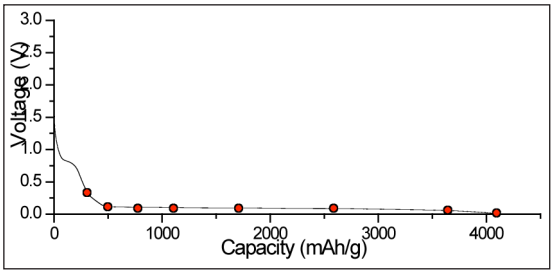

Figure 1. Electrochemical profile of first discharge of crystalline silicon and carbon SP composite electrode vs. Li.

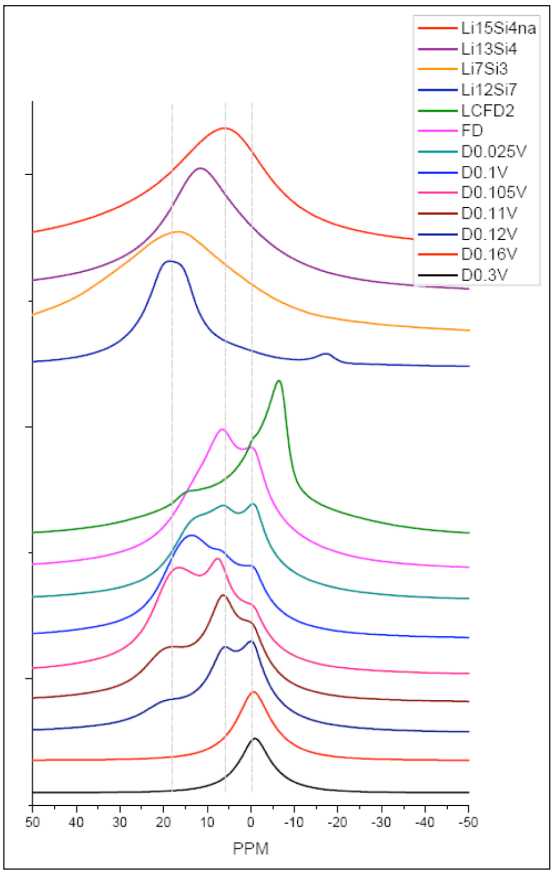

Figure 2. ^{7}Li MAS-SSNMR spectra of selected Li-Si intermetallic compounds and Si electrodes stopped at different discharge states (approx. discharge voltage denoted in vertical sequence in legend).

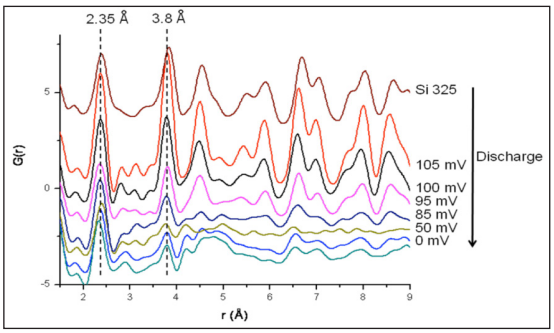

Figure 3. Pair distribution function analysis of crystalline Si (Si 325) and Si electrodes stopped as different discharge states.

WK6

Elucidating Structure-property Relationships in Functional Metal-organic Frameworks using *in situ* Synchrotron-based Powder Diffraction

Gregory J. Halder
Materials Science Division, Argonne National Laboratory

The targeted development of functional metal-organic frameworks, is not only a synthetic challenge but requires parallel characterization of their often complex structure-property relationships. Among strategic efforts to introduce specific function to these materials, the incorporation of spin crossover switching centers allows for the development of new advanced functional materials for molecular-scale electronic switching and sensing devices. Recent work has shown that such spin crossover framework materials (SCOFs) can incorporate an additional level of functionality associated with their often porous natures; guest sorption/desorption in such systems, and the associated perturbation of the iron(II) coordination environment and framework structure, provide a unique avenue for investigating the spin crossover phenomenon in the solid state. To this end we have developed *in situ* synchrotron-based powder diffraction methods to simultaneously probe their guest-dependent structural and magnetic properties.

WK6

High-throughput, *in situ*, and *operando* XAFS Spectroscopy from the Soft to the Hard X-ray Range

Sven L. M. Schroeder
The University of Manchester, School of Chemical Engineering and Analytical Science & School of Chemistry, Manchester, M60 1QD

Using commercially available standard technologies we have developed flexible, inexpensive and modular platforms for high-throughput *in situ/operando* XAFS of catalytic processes in (i) homogeneous liquid phase and in (ii) heterogeneous gas-solid systems. Automated gas and liquid handling of feedstocks via reaction/formulation vessels to the in situ XAFS reactor stages facilitates measurements on air- or moisture-sensitive reactants. The functionality of the *in situ* systems will be demonstrated using examples of (i) dialkyl-zinc-catalyzed selective oxidations in homogeneous phase, (ii) ternary supported metal catalysts for the oxidation of carbon monoxide, and (iii) the early stages of crystallisation and self-aggregation of imidazole in aqeuous solution.

WK6

The Use of *in situ* Synchrotron-based Spectroscopic Methods to Characterize Functional Metal-organic Framework Materials

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Metal-organic frameworks (MOFs, also known as "coordination polymers") are crystalline nanoporous materials comprised of metal-containing clusters connected three-dimensionally by poly-functional organic ligands. The ligands act as spacers, creating an open, porous three-dimensional structure, with very high pore volume and surface area. This hybrid architecture opens the possibility to design and synthesize a great variety of new porous materials, which are in principle able to display novel functionalities that are potentially exploitable for a number of applications in catalysis, ion exchange, nonlinear optics, sensors, and gas separation and/or storage.[1-10]

The key step for most of the foreseen applications is the solvent removal treatment, which makes the large pore volume accessible to the desired molecules. For several cases (among all MOF-5, HKUST-1, CPO-27-Ni, UiO-66), the combined use of XRD, EXAFS, XANES, UV-vis, IR and Raman techniques allowed us to obtain a complete understanding of the structural, electronic, and vibrational properties of MOF materials. The adoption of *in situ* experimental set-ups guarantees the possibility to follow the evolution of such properties along the solvent removal process and the successive interaction with increasing amount of desired adsorbate (H_2, N_2, CO, NO, CO_2, etc.).[8-14]

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WK6
Sulfur Poisoning in Rh, Ni Catalysts for Steam Reforming of Liquid Hydrocarbons

Yongsheng Chen, Chao Xie, Yan Li, Chunshan Song, Trudy B. Bolin

This work addresses a decades-old sulfur poisoning mechanism in the steam reforming reaction, which is of great interest to the catalysis community. The residual sulfur species in sulfur-poisoned Ni and Rh catalysts were identified by x-ray absorption near edge structure (XANES) spectroscopy, and the major species included metal sulfide, organic sulfide (-C-S-C-), sulfonate, and sulfate. Total sulfur and carbon amounts in the catalysts were determined chemically. For Ni catalyst, the formation of nickel sulfide caused the loss of about one third of the steam reforming activity in the first five hours, and two thirds of the activity gradually vanished subsequently in another 50 hours due to the carbon deposition. Rh catalyst had much better performance under our reaction conditions; within the test time, over 80% of steam reforming activity remained and carbon deposition was 10 times less than what was observed on Ni catalyst. Sulfur chemistry was different on Rh catalyst, which might be the key to its better sulfur tolerance compared to Ni catalyst.

WK6

In situ X-ray Scattering and Spectroscopy Studies of Model Nanocatalysts under Realistic Reaction Conditions: A New Approach for Understanding Size/Shape Effects on Catalytic Performance

Sungsik Lee
Chemical Sciences and Engineering Division, Argonne National Laboratory

Catalytic performance of nanocatalysts has been the subject of extensive theoretical and experimental studies within the surface science and catalyst community. To gain detailed understanding of the size-function relationship on molecular level, monitoring the chemical/physical properties of atomically controlled nanoparticles during the course of the catalytic reaction is of great importance. This work focuses on synthesis techniques to control the size of catalyst from few-atom clusters to nanoparticles and *in situ* surface X-ray analysis, grazing incidence small angle X-ray scattering (GISAXS) and grazing incidence X-ray absorption spectroscopy (GIXAS) combined with temperature-programmed reaction (TPRx). The powerful combination of these techniques will be illustrated on selected examples of sub-nanometer clusters and larger nanocatalysts in various reactions.

WK6

Characterization of Photoactive Self-assembled Structures via Solution Small- and Wide-angle X-ray Scattering

Josh Vura-Weis
Department of Chemistry, Northwestern University

To create new materials for solar energy conversion, we employ bio-inspired molecular designs based on self-assembly that are economical and versatile, yet sufficient to mimic the functionality of the natural system. Using this strategy we have prepared a variety of self-assembling artificial nanostructures that mimic the key photosynthetic functions. Structural studies in solution by x-ray scattering techniques using synchrotron radiation have emerged as the most valuable methodology for characterizing our systems. Our SAXS/WAXS studies conducted at APS have resulted in the elucidation of structure of these systems at the actual conditions at which we performed photophysical measurements on them, providing invaluable insights into their structure-function relationships.

WK6

Combining Molecular Dynamics Simulations and Solution-Phase X-Ray Scattering to Investigate Conformational Changes

Kristy Mardis
Department of Chemistry and Physics, Chicago State University

Electron transfer events are crucial for a wide range of processes including photosynthesis, respiration, and designing new solar energy devices. Understanding such events requires the development and application of methods that can investigate solution-phase conformational changes. Recent advances combining x-ray scattering measurement with coordinate-based modeling methods have created opportunities for enhancing our understanding of structural dynamics of supramolecular assemblies in solution. This talk will highlight correlations between solution x-ray scattering results and ensembles of molecules created through molecular dynamic simulations. Examples will include interactions between solvent molecules and zinc-porphyrin hexamers, the ability to distinguish conformational changes in cytochrome c_7 proteins upon oxidation, and hydrogen bonding interactions in primary alcohols. The combination of atomic-scale molecular ensembles created through molecular dynamics simulations and solution-phase x-ray scattering offer possibilities for resolving the structure of dynamic supramolecular assemblies that can not be fully addressed by crystallographic techniques. In addition, this approach addresses the need to determine fundamental mechanisms for solar energy conversion by mapping out structures under conditions necessary for electron transfer.

Workshop 7:
High Pressure Synchrotron Science: Future Frontiers

Wednesday, May 6, 2009, through Friday, May 8, 2009
Bldg. 362, Auditorium (Thursday and Friday: Bldg. 402, Lecture Hall)

Organizers: Yang Ding, Malcolm Guthrie, and Michael Lerche (all HPSynC)

The natural resonance between synchrotron sources and high-pressure science has led to dramatic advances across surprisingly diverse scientific fields in previous decades. At present, major renewal programs are either underway or imminent at existing third-generation synchrotrons, and fouth-generation sources are just on the horizon. Given this context, now is a critical time to explore the full potential of high-pressure science at synchrotron sources. The goal of this workshop is to use the current state of the art as a starting point to explore the future of high pressure science. As part of the forward-looking theme of the workshop, the program will comprise both the future evolution of established techniques and entirely novel uses of the high pressure variable.

For full details and the program for Thursday and Friday, please see the workshop web page, http://www.hpsync.org/links/HiPreSS/.

8:30 – 8:45 Opening remarks
H-K. Mao, Carnegie Institution of Washington, DC

8:45 – 10:30 Fundamental Matter under Extreme Conditions
Chair: H-K. Mao, Carnegie Institution of Washington
E. Gregoryanz, University of Edinburgh, UK
P. Dera, University of Chicago, Illinois
R. Ahuja, Uppsala University, Sweden

10:30 – 10:45 Coffee Break

10:45 – 12:30 Extreme Biology: X-ray Techniques and the Study of Proteins under Pressure
Chair: K. Brister, Northwestern University
R. Winter, Technische Universität Dortmund, Germany
C. U. Kim, Cornell University, New York
R. Fourme, Synchrotron Soleil, France

12:30 – 2:15 Lunch

2:15 – 4:00 Physics and Chemistry of Earth and Planetary Interiors
Chair: V. Prakapenka, University of Chicago, Illinois
L. Dubrovinsky Universität Bayreuth, Germany
R. Boehler, Max Planck Institute for Chemistry, Germany
T. Duffy, Princeton University, New Jersey

4:15 – 6:00 Technical Session: Future High-Pressure Science Using Nano-beams
Chair: Z. Cai, Argonne National Laboratory
L. Wang, Carnegie Institution of Washington, DC
W. Yang, Carnegie Institution of Washington, DC
W. Liu, Argonne National Laboratory

Scientific Advisory Committee:

Ercan Alp, Chris Benmore, Keith Brister, Daniel Haskel, Russ Hemley, Wenjung Liu, Dave Mao,Vitali Prakapenka, Guoyin Shen, Yanbin Wang

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POSTER ABSTRACTS

A1

Micron- and Nano-scale Computed Tomography of Zebrafish

Darin Clark[1], Roland Myers[2], Timothy Sledz[3], Jean Copper[1], Margaret Hubley[1], Robert Winarski[4], Francesco De Carlo[4], Patrick La Riviere[5], and Keith Cheng[1, 6, ‡]
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The length scale of and the genetic toolset established for the zebrafish make it an ideal vertebrate model system for answering an overarching biological question: "How do genes result in the production of organelles, cells, and organs?" Part of this effort is an on-line atlas of zebrafish microanatomy (http://www.zfatlas.psu.edu/) that currently focuses on histology as a basis for recognizing normal, mutant, and diseased zebrafish. We propose to use micron- and nano-scale computed tomography (micro- and nano-CT) to create three-dimensional models of zebrafish at crucial developmental stages. These data will serve as a reference point for comparisons at resolutions both above and below the length scales offered by histology and complement data collected by three-dimensional optical, magnetic resonance, and electron-based tomographic techniques. We report progress developing a protocol for high-resolution hard and soft tissue imaging of zebrafish through the use of osmium tetroxide and/or uranyl acetate staining and plastic embedding. Edge subtraction and phase contrast enhancement techniques are also being pursued. This work will contribute reference images to the atlas, and will someday allow zebrafish investigators to connect genetic and reverse genetic deficits with changes in organelles, cells and organs.†

† Use of the Advanced Photon Source at Argonne National Laboratory was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

‡ Funding provided by the Jake Gittlen Cancer Research Foundation of the Penn State College of Medicine and by the NIH (Grant #RR017441).

A2

On-axis Sample Visualization Using Reflective Optics

Kazimierz J. Gofron, Michael Molitsky, and Andrzej Joachimiak
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The on-axis viewing is used for high-precision visualization of micrometer-size biological crystals and non-distorted (no parallax) view of the x-ray mini beam at the sample location. We have developed an on-axis (along the synchrotron x-ray beam) sample visualization system and implemented the microscope at 19-ID beamline. The on-axis visualization utilizes Questar QM100 reflective microscope and a 45º mirror. The on-axis microscope permits a convenient long working distance of 5–16 inches, and provides variable optical magnification of 2x–34x. The 45º mirror has a small hole to pass the x-ray beam. Since the on-axis optics is reflective (mirror based) it results in non-dispersive (no chromatic aberration) image quality capable of focusing light with wavelengths from deep UV (UVB) to IR. Using the on-axis microscope we investigated UV fluorescence from biological crystal with intention of utilizing it for crystal location.

This work was supported by the U. S. Department of Energy, Office of Biological and Environmental Research, under contract DE-AC02-06CH11357.

A3

Diffraction/Fluorescence Rastering and Automated on-the-fly Fluorescence Scanning at GM/CA-CAT

M.C. Hilgart, S. Stepanov, R. Sanishvili, N. Venugopalan, D. Yoder, and R. Fischetti
GMCA-CAT, Biosciences Division, Argonne National Laboratory, Argonne, IL 60439

GM/CA-CAT's "mini-beam" capability allows for successful solution of structures that would have otherwise not been possible. This includes collecting data of crystals ~5μm or less in size, finding "good" spots on twins and poorly diffracting crystals and collecting partial complementary datasets from several spots on radiation sensitive crystals.

To facilitate this technique, we have developed rastering software that helps users in finding the best diffracting areas of crystals or locating crystals that can't be seen visually. The software samples a user-defined grid and takes diffraction or fluorescence data for each cell.

Users choose either diffraction or fluorescence rastering in GM/CA's beamline control software, BluIce-EPICS. For diffraction rastering, data are automatically scored by an external tool called distl that counts diffraction spots at each location, and the results are shown in a table. Attenuation is kept very high to prevent radiation damage and this way only the strongest reflections are counted.

Fluorescence rastering is faster than diffraction rastering by about four times due to deployment of on-the-fly scanning. It is most useful when samples contain Se or other anomalous reference scatterers or when radiation damage is critical. Results are shown in a color map, which is displayed over the sample image and moves with the sample.

We have also developed three-band on-the-fly edge scans for determining the inflection points in multiple wavelength anomalous diffraction data collections. These scans typically take two minutes compared to 6.5 min with steps scans. For users' convenience, several automations like ROI presets for selected absorption edge and fluorescence detector optimization have been added.

A4

Investigating Barium and Strontium Selectivity in Desmid Green Algae

Minna Krejci[1, 2], Stefan Vogt[2], Barry Lai[2], and Derk Joester[1]
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[2] X-ray Microscopy and Imaging, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439

There is currently no cost-effective and selective separation method available to remove radioactive ^{90}Sr from either nuclear waste or the environment. The difficulty is caused by the chemical similarity of Ca^{2+}, Sr^{2+}, and Ba^{2+} cations, which also leads to indiscriminate transport (and incorporation into bone) in most animals. However, desmid green algae of the genus *Closterium* have the ability to selectively precipitate (Ba, Sr)SO_4 crystals within vacuoles in a calcium-rich environment, indicating that they possess a unique capability for Sr and Ba selective transport. The present work involves an investigation of the basic methods of Sr and Ba selectivity and transport in these organisms, using synchrotron x-ray fluorescence microscopy elemental mapping to identify routes of uptake and locations of selectivity filters. With this technique, we have verified the uptake and sequestration of Ba and Sr into crystals and have observed time-dependent changes in intracellular Sr concentrations. Preliminary results indicate that Sr is compartmentalized in between the lobes of the chloroplasts, which may indicate vesicular transport or an extensive vacuolar network. An elucidation of the mechanisms of selective transport in these organisms will enable and inspire innovation of chemical and bioremediation approaches to ^{90}Sr cleanup operations.

A5

Cytochrome c Compressibility Measured with Two Synchrotron-based Experiments

Bogdan M. Leu[1], Ahmet Alatas[1], Harald Sinn[2], E. Ercan Alp[1], Ayman Said[1], Hasan Yavas[1], Jiyong Zhao[1], J. Timothy Sage[3], and Wolfgang Sturhahn[1]
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[3] Department of Physics, Northeastern University, Boston, MA 02115

Poster displayed.

A6

Characterizing the Unsolved Proteins of the PSI Using WAXS

Lee Makowski, Robert Fischetti, Diane Rodi, and Suneeta Mandava
Biosciences Division, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439

We are developing methods for using wide-angle x-ray solution scattering (WAXS) to characterize proteins expressed but not solved by the Protein Structure Initiative (PSI). The PSI has constructed expression clones for over 110,000 proteins, most of which remain unsolved, representing a collection of proteins whose structures would have significant value in defining the universe of protein structures. In an experiment typically lasting one or two minutes and utilizing less than a milligram of protein, WAXS provides a data set containing substantial information. This makes it is possible to collect structural information on a great many proteins in a relatively short period of time. WAXS can be used to determine size, shape, and oligomeric state of a protein. Furthermore, by comparing the WAXS patterns of proteins to those from proteins of known folds, it may be possible to identify those targets with structures very similar to known structures as well as those with the highest probability of exhibiting entirely novel folds. Proteins giving rise to completely novel WAXS patterns may well have the highest probability of exhibiting completely novel folds.

A7

Feasibility of X-ray Fluorescence Computed Tomography Using Emission Tomography Systems

L.J. Meng[1], P.J. La Riviere[2], G. Fu[1], Peter Eng[3], and Matt Newville[3]
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The overall goal of this work has been to develop and implement faster and more accurate synchrotron-based x-ray fluorescence computed tomography (XFCT) methods for the mapping of trace metals in biological samples. Many endogenous metals play critical roles in signal transduction and reaction catalysis, while others are quite toxic even in trace quantities. In addition, exogenous metals are often critical components of new *in vivo* molecular imaging agents. The study of these metals in biology would benefit greatly from the three-dimensional (3D) spatially resolved maps of trace element distribution provided by the methods being explored.

Specifically, this paper presents a feasibility study of using emission tomography (ET) systems for synchrotron XFCT. The proposed detection system combines high-resolution semiconductor detectors with multiple-pinhole apertures. The key advantage of using an ET-based detection system is that 3D distributions of trace elements can be built up with much reduced scanning motion and potentially without need for tomographic reconstruction. In comparison to the conventional line-by-line scanning scheme, the ET-based imaging system allows a great reduction in imaging time, which has been one of the major hurdles for current XFCT studies.

A8

The First Structural Studies of Bacterial Polyphosphate Kinase 2

B. Nocek, J. Osipiuk, E. Evdokimova, A. Savchenko, A. Edwards, A. Yakunin, and A. Joachimiak
The Midwest Center for Structural Genomics and Structural Biology Center, Biosciences, Argonne National Laboratory, 9700 South Cass Ave., Bldg. 202, Argonne, IL 60439

The Midwest Center for Structural Genomics (MCSG), one of four large-scale centers of the Protein Structure Initiative, provides structural coverage of major protein superfamilies. The objective of MCSG is to develop and optimize new methods for high-throughput determination of protein structures of important biological targets through x-ray crystallography. Recently, our center has cloned, purified, and functionally characterized several members of the polyphosphate kinase 2 (PPK2) family of proteins. PPK2 kinases are considered to be potent polyphosphate-dependent generators of nucleoside triphosphates and are involved in many aspects of cellular metabolism.

We have demonstrated that the PPK2 family members could be grouped into two classes: short (~30 kDa) PPK2, and long (~60 kDa) PPK2, comprised of two fused PPK2 domains. We have shown that the first class, which contains a single PPK2 domain, catalyzes the poly-P-dependent phosphorylation of nucleoside diphosphates (ADP, GDP) to nucleoside triphosphates, whereas the class of proteins with two fused PPK2 domains catalyzes phosphorylate nucleoside monophosphates (AMP, GMP) to the respective diphosphates. Crystal structures of representative of each group of PPK2 family were determined and site-directed mutagenesis identified the conserved residues important for catalysis. Details of the PPK2 structures and insights into the possible catalytic mechanism will be presented.

This work was supported by National Institutes of Health grant GM074942 and by the U.S. Department of Energy, Office of Biological and Environmental Research, under contract DE-AC02-06CH11357.

A9

GM/CA-CAT Tools for Remote Controls: Automated Screening, Robotic Sample Changer, WebIce, Beam Alignment, and Crystal Centering Software

S. Pothineni, O. Makarov, N. Venugopalan, C. Ogata, M. Hilgart, S. Stepanov, and R. Fischetti
GMCA-CAT, Biosciences Division, Argonne National Laboratory, Argonne, IL 60439

Remote user operations are quickly ramping at the GM/CA-CAT beamlines. Advanced Light Source (ALS)-style robotic sample changers are used for this purpose. Software is being developed to provide convenient automations of all parts of the experiments, especially unattended fully automated crystal screening. A robot server software has been developed for mounting the samples as a part of this project. BluIce-EPICS beamline control user interface has been upgraded to communicate with the robot server. BluIce-EPICS GUI allows users to load spreadsheets containing sample information and to assign the samples for screening. The automated crystal centering is implemented with the XREC and C3D software packages.

Crystal scoring software WebIce, which is developed at the Stanford Synchrotron Radiation Lightsource and ALS, is being adapted to GM/CA beamlines. After being collected images are sent to the WebIce for processing. WebIce indexes them with LABLEIT and generates a crystal score from an empirical formula to select the best crystals for data collection. It also creates data collection strategy using BEST/MOSFLM.

These developments allow users to work remotely very efficiently when connecting to GM/CA-CAT computers using NOMACHINE software. Various automation tools like automated diffraction and fluorescent sample rastering, automated minibeam collimator replacements, minibeam adjustment with on-the-fly scanning of piezo actuators, and others provide further facilitation of remote beamline operations.

A10

Mechanism of Interaction between the Volatile Anesthetic Halothane and a Model Ion Channel Protein in Langmuir Monolayers Studied by X-ray Reflectivity

Joseph Strzalka[1], Jing Liu[1], Andrey Tronin[1], Inna Y. Churbanova[1], Jonas S. Johansson[2], and J. Kent Blasie[1]
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Poster displayed.

A11

X-ray Diffraction Study of DMPE:Dchol Monolayers using a 1D Pinhole Geometry

Kathleen D. Cao[1], Luka Pocivavsek[1], Mati Meron [2], Binhua Lin[2], and Ka Yee C. Lee[1]
[1] Department of Chemistry, Institute for Biophysical Dynamics, and The James Franck Institute, The University of Chicago, Chicago, IL 60637
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Grazing incidence x-ray diffraction of mixed dimyristoylphosphoethanolamine (DMPE) and dihydrocholesterol (Dchol) monolayers provides evidence of a lipid ordered phase. Two trends for the intermolecular distances (d-spacing) were found as a function of lipid composition. For Dchol amounts less than 20 mole percent, there is little change in the d-spacing. However, for monolayers with more than 20 mole percent Dchol, a linear increase in d-spacing is observed with increasing Dchol concentration. Unlike traditional experiments, an area detector was used rather than a linear detector. Employing a geometry similar to a pinhole camera, data with point densities more than five times greater compared to a linear detector were collected.

A12

Multi-phase Behavior in Coordination Framework Materials at High Pressure

Karena W. Chapman[1], Gregory J. Halder[2] , and Peter J. Chupas[1]
[1] X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439
[2] Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

Compared to traditional solid state materials based on corner-, edge-, and face-sharing polyhedra, coordination framework materials form expanded network structures in which the structural building units are bridged via molecular ligands (e.g., -CN- or 4,4'-bipyridine) rather than single atoms (e.g., -O-, -F-, -S-). As such, distortions of the framework, involving the translation and/or rotation of connected building units, are decoupled. This is associated with greatly enhanced structural degrees of freedom. Here, we present the comprehensive synchrotron-based investigation of the high-pressure behavior of a coordination framework material (<10 GPa), highlighting the structural versatility of these materials. Specifically, for the sample contained within a diamond anvil cell, several distinct high-pressure phases were accessible at a given pressure under different experimental conditions.

A13

Catalyst Center at the Advanced Photon Source Beamline 9-BM

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Catalyst research has been an important component of research at the Advanced Photon Source. Catalysis is an essential technology for economic prosperity, energy security, and environmental preservation in the twenty-first century. The importance of catalysis research to meet both the energy needs of the nation and the central role of Department of Energy (DOE) facilities in advancing catalysis science has been specifically identified in the Energy Policy Act of 2005. One of the recent DOE reports on research needs is entitled "Basic Research Needs: Catalysis for Energy" from a BES workshop held August 6–8, 2007. In addition, catalysis can be expected to play a major role in a number of other research needs identified in this series of reports. These reports include "Basic Research Needs for Solar Energy Utilization," "Basic Research Needs for Clean and Efficient Combustion of 21st Century Transportation Fuels," and "Basic Research Needs for the Hydrogen Economy." As part of the reports on specific research needs, the DOE also identified "Five Challenges for Science and the Imagination." Mastery of catalysis and catalytic systems will require advancement in all five of these challenges.

A workshop titled "Catalysis Research at the APS" was held on September 12–13, 2005, in order to assess the requirements and opportunities for supporting catalysis research at the Advanced Photon Source. In response to the major recommendations of this report, a Laboratory Directed Research and Development proposal was submitted. It received funding in FY2009. This proposal includes plans for upgrading beamline 9-BM and support equipment for *in situ* studies of operating catalysts over a wide energy range (2.1 to 24 keV). Key features are the addition of the capability for concurrent *in situ* x-ray absorption fine-structure and diffraction characterization, and the upgrading of facilities for catalyst preparation and characterization in nearby laboratories. This project is the first step in establishing a center to facilitate catalyst studies at all APS beamlines.

A14

Elucidating Structural Dynamics and Excited State Pathways of Transition Metal Complexes Using Laser-initiated Time-Resolved X-ray Absorption Spectroscopy (LITR-XAS)

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Laser-initiated time-resolved x-ray absorption spectroscopy (LITR-XAS) is employed to probe the excited state structural changes of transition metal complexes. The correlation between electron density redistribution and structural change along excited state pathways is particularly important to establish for understanding the photochemistry of coordination complexes since metal oxidation state changes often dictate the photochemical excited state pathways. Under investigation are a copper(I) bisphenanthroline complex and a bimetallic complex consisting of two square planar platinum(II) units connected by pyrazolate bridging ligands. LITR-XAS is used to probe the metal electron configurations and excited state molecular structural changes upon laser photon absorption into the lowest energy metal to ligand charge transfer states. The XAS results are presented along with theoretical analysis to show excited state metal oxidation state changes and corresponding Jahn-Teller distortion in the case of the copper(I) system and changes in metal-metal distance for the platinum dimer molecule.

A15

Structure and Stability of Lipid Monolayers on Binary Fluids: X-ray and Neutron Reflectivity Study

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Interfaces are ubiquitous in nature and absolutely key for life as illustrated by such complex interfaces as the cell membrane and the endothelial and epithelial linings of tissues. The mechanical properties of these interfaces play an important role in their biological functions. In this talk, we describe our work over the last two and a half years using geometry, continuum mechanics, physical chemistry of surfaces, and x-ray/neutron scattering as a tools for studying the behavior and structure of complex interfaces, in particular self-assembled lipid monolayers. What has emerged form our endeavor is a physical model about the general behavior of supported elastic interfaces. We show that elastic interfaces be it a ten micron thick piece of polyester or a nanometer thin lipid monolayer follow the same scaling laws which govern their response to mechanical stress. Moreover, we show that the mechanical response of self-assembled membranes like lipids becomes increasingly sensitive to the structure of the bulk fluid upon which it rests. Using x-ray and neutron reflectivity to probe the atomistic structure of lipid membranes at binary fluid interfaces of water and glycerol, a model is constructed which takes into account bulk fluid effects on membrane mechanics.

A16

X-ray Absorption Spectroscopy Analysis of Ru-based Water Oxidation Catalysts

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Sunlight provides by far the largest of all carbon-neutral energy sources. Production of solar fuels by artificial photosynthesis will play an important role in a future energy economy. Water oxidation is a key half reaction of artificial photosynthesis. In nature this reaction is carried out in the oxygen-evolving complex of Photosystem II. Molecular catalysts capable of water oxidation include ruthenium and iridium complexes. However, the performance of all known catalysts is far inferior to that of Photosystem II. Coupling the multi-electron nature of water oxidation to single photon solar absorption poses a significant mechanistic challenge. Our limited understanding of the mechanisms of catalytic water splitting is a crucial barrier to future practical advances of artificial photosynthesis.

Ru L- and K-edge x-ray absorption spectroscopy is here used to characterize the electronic states and molecular geometries of Ru-based catalysts (Ru "blue dimer" and single-site Ru complexes) of water oxidation. Intermediates of water oxidation were prepared from the Ru-complexes by oxidation with Ce(IV). L-edges x-ray absorption near-edge spectroscopy demonstrated an increase in the Ru oxidation state prior to the water splitting step. Extended x-ray absorption fine structure at the Ru K-edge revealed a shortening of the Ru-O bond in the oxidized intermediates. Comparison of the experimental and density functional theory optimized structures will help us understand the mechanism of water oxidation by Ru-complexes.

A17

Steam Reforming of Liquid Hydrocarbons over Supported Transition Metal Catalysts for Fuel Cell Applications

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On-board liquid hydrocarbon reforming over supported transition metal catalysts is attracting considerable attention as it is very promising for providing H_2 to fuel cell-powered vehicles. However, the catalyst deactivation due to sulfur poisoning and carbon deposition is a big challenge for its practical applications. In this study, the deactivation of Al_2O_3 supported Rh, Pt, Ru, Pd, and Ni catalysts is investigated through reforming of sulfur-containing liquid hydrocarbons in the presence of sulfur at 800°C. With the aid of x-ray absorption near edge spectroscopy, system by

using the extended x-ray absorption fine structure spectra to determine the specifics different sulfur species including metal sulfide (M-S), organic sulfide (C-S-C), sulfonate ($C\text{-}SO_3^-$), and sulfate (SO_4^{2-}) have been identified over the spent catalysts. It was found that sulfur preferred to be converted to sulfonate and sulfate, while metal and organic sulfides are the prevailing sulfur species for the other catalysts. The different sulfur conversion pathways may be related to the exceptional sulfur tolerance of Rh compared with the other metals for reforming of sulfur-containing liquid hydrocarbons at 800°C. Moreover, K addition can effectively enhance the catalytic performance of the Ni catalyst for the reaction, whereas little promotion effect is observed for the K-modified noble metal catalysts.

A18

Ultrafast/Ultrasmall Laser/X-ray Techniques at High Repetition Rate

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We are combining novel high-repetition-rate ultrafast lasers with a hard x-ray microprobe to study and control behavior of atoms and molecules in strong optical fields and to study photoinduced structural dynamics of isolated molecules in the solution phase. Typically, these laser pump/x-ray probe studies are done at kilohertz repetition rates because the high peak intensities required (10^{12}–10^{13} W/cm^2) necessitate the use of large scale solid state amplifiers.

The mismatch in laser (1–5 kHz) and x-ray (6.5 MHz) repetition rates results in a very inefficient use of the Advanced Photon Source and, consequently, photon starved experiments. We make efficient use of *both* x-ray and laser power by using an integrated picosecond master oscillator/power amplifier laser system (where repetition rates can be varied between 50 kHz and 6.5 MHz) and microfocused beams. The high repetition rate is suitable for studying rapidly reversible or replenishable systems (e.g., gas jets, liquid jets, and nanoscale devices). To attain strong fields at the maximum repetition rate, a phase-locked enhancement cavity where coherent addition of successive pulses can increase the peak intensity by ~500-fold is being developed in collaboration with the JILA group.

A19

Levitated Supercooled Liquids

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A supercooled liquid can be classified as 'strong' or 'fragile' according to the rapidity with which its viscosity and relaxation times change as it approaches the glass transition. In strong liquids the viscosity behaves in a nearly Arrhenius fashion, whereas fragile liquids display a more complex behavior, with the viscosity changing very rapidly at temperatures near the glass transition temperature. The goal of this work is to connect the structure of the liquid with the kinetics of glass behavior and the underlying thermodynamics. Containerless levitation techniques provide a means of accessing the deeply supercooled liquid state. In this poster we describe our recent advances using acoustic and aerodynamic levitation techniques, integrated onto the high-energy x-ray beamline 11-ID-C. Silicate liquids play an important role in modeling the melt during the formation of the early Earth. High-temperature aerodynamic levitation measurements have been performed on a series of laser heated alkaline earth- and metal-silicate liquids. Their relative viscosities are related to the structural units present. For low-temperature melts an acoustic levitator used in tandem with a cryostream is described. This technique is still under development but is starting to provide detailed structural information on deeply supercooled fragile hydrocarbon liquids.

A20

Enhancement of Diffuse X-ray Scattering from Supported Phospholipid Bilayers using Au Nanoparticle Labeling

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Supported phospholipid bilayers provide a simple model system through which properties of real cellular membranes can be studied in a controlled manner. An important goal of such research is to elucidate the mechanisms by which membrane proteins interact with their lipid environment. This could lead to important biomedical applications such as enhanced drug delivery or antimicrobial drugs. Supported bilayers are only stable under water, which makes grazing-incidence small-angle x-ray scattering (GISAXS) studies challenging due to high background. In the present work we have investigated using Au nanoparticles to label both single component bilayers as well as one lipid in multicomponent bilayers in order to enhance the scattering signal. GISAXS from labeled and unlabeled systems are compared. In addition, specular reflectivity of both systems was measured in order to understand how the Au labeling may modify the bilayer structure.

A21

Configuration of PKCalpha-C2 Domain Bound to SOPC:SOPS Lipid Monolayers

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X-ray reflectivity measurements are used to determine the configuration of the C2 domain of protein kinase C alpha (PKCalpha-C2) bound to a lipid monolayer of a 7:3 mixture of SOPC and SOPS supported on a buffered aqueous solution. The configuration is described by theta = 35° ± 10°, phi = 210° ± 30°, and a domain penetration of 7.5 ± 2.2 Å into the lipid layer. This configuration of PKCalpha-C2 determined by our x-ray reflectivity is consistent with many previous findings, particularly mutational studies, and also provides new molecular insight into the mechanism of PKCalpha enzyme activation. Our modified data analysis methodology, which allows us to test the whole range of protein orientations, shows that our data cannot be explained by a protein that is orientated parallel to the membrane, as proposed by crystallographic and Einstein-Podolsky-Rosen experiments.

A22

Structure of Silicon-supported Phospholipid Bilayers Measured via X-ray Specular Reflectivity

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Silicon-supported phospholipid bilayers provide a model system for exploring the phases and interactions of cellular membrane components. One advantage of a silicon support is that it provides an orientation to the bilayer allowing structural studies via x-ray specular reflectivity. We have performed specular reflectivity on a number of biologically important lipid bilayers supported on ultra-smooth silicon substrates. Density profiles of the lipids are obtained from the reflectivity using two different methods. One method employs a maximum entropy search through a parameterized layer model, and the second method employs a phase-guessing direct inversion algorithm. In addition, membranes were studied both with and without Au nanoparticle labels used to enhance contrast. The resulting membrane structures and their significance to the cell membrane phases will be discussed.

A23

Electronic Interband Transitions in Doped Cuprates Measured with Resonant Inelastic X-ray Scattering

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Electronic interband excitations have been observed with resonant inelastic x-ray scattering (RIXS) in the hole-doped cuprate material $La_{2-x}Sr_xCuO_4$ (x = 0.0, 0.05, 0.07, 0.10, 0.12, 0.17, 0.25, 0.30, and 0.35). In this poster, we present the progression of the spectra as the hole doping is increased from the undoped to the overdoped region of the phase diagram. Three main features are prominent in the spectra; a ~ 3.5 eV peak which is suppressed and shifts to higher energy with increased doping, a 4.5 eV peak which is nearly doping independent and a 1.6 eV peak which exhibits doping dependent width change. This latter peak is enhanced in $La_{1.875}Ba_{0.125}CuO_4$, raising the question a possible association with stripe electronic structure. The temperature dependence of the1.6 eV peak in $La_{1.875}Ba_{0.125}CuO_4$ shows gradual change between 25 K and room temperature.

A24

X-ray Diffraction from Liquid Water up to ~5 GPa

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Water can lay a strong claim on being the most studied liquid in history. However, its structure under high pressure remains poorly understood. A wealth of diffraction studies of both amorphous ices and liquid water at relatively low pressures highlight the importance of changes in the local oxygen environment. As the x-ray diffraction signal is dominated by oxygen-oxygen correlations, such measurements provide an ideal probe of pressure-induced modification of the water networks. To date, however, the poor diffraction signal of water and the challenges associated with high-pressure measurements have limited quantitative x-ray diffraction measurements to < 1.1 GPa. Here we present data collected from the liquid at 4.7 GPa and 477 K and examine the consequences for the structure of the liquid under these conditions.

A25

Cholesterol-phospholipid Interactions: New Insight from Surface X-ray Scattering Data

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Cholesterol-enriched domains in cell membranes are involved in a wide variety of cellular activities including protein sorting, signal transduction, and host-pathogen interactions. Although a number of conceptual models exist, there is no consensus on the structural organization of cholesterol-phospholipid membranes. Here we present a systematic structural study of cholesterol-DPPC mixed monolayers at the air-liquid interface by surface pressure-molecular area Langmuir isotherms, as well as x-ray reflectivity (XR) and grazing incident-angle x-ray diffraction (GIXD) using synchrotron radiation. Electron density distribution across the monolayer, derived from XR data, demonstrates that cholesterol moiety is located within DPPC acyl chains at low mole fractions of cholesterol (χ_{CHOL}) 0.13-0.46, promoting thickening of the phospholipid hydrophobic core, whereas shifts towards the aqueous milieu at higher χ_{CHOL}. Analysis of x-ray data and Langmuir isotherms provide explanation for well-known "condensing" effect of cholesterol. According to GIXD, at $\chi_{CHOL} \leq 0.13$ the ordered part of cholesterol-DPPC mixed monolayer is presumably represented with pure DPPC and at $\chi_{CHOL} \geq 0.85$ with pure cholesterol. At the intermediate sterol concentrations, cholesterol and DPPC self-organize into a new, cooperative molecular structure with a short-range order and intermolecular spacing changing according to stoichiometry

A26

Direct Measurement of Charge-transfer Gap, Mott-Hubbard Gap, and Crystal Field Splitting in $FeBO_3$ by Fe K-edge Resonant Inelastic X-ray Scattering

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Momentum-resolved resonant inelastic x-ray scattering (RIXS) spectroscopy in the horizontal scattering geometry is applied at the Fe K-edge, successfully for the first time. RIXS spectra in $FeBO_3$ single crystals reveal wealth of information on low-energy ~ 1–10 eV electronic excitations. $FeBO_3$, is a large gap insulator with T_N=348 K. Fe^{3+} ions are centered in a slightly distorted octahedra where the energy of the 3d orbitals into t_{2g} and e_g states. Five 3d electrons fill these crystal filled levels in the high-spin configuration. It was found that IXS signal resonates both when the incident photon energy approaches the 1s-3d or 1s-4p transitions. The 1s-3d and 1s-4p RIXS spectra show qualitatively different dependences on the incident photon energy, momentum transfer, polarization, and temperature. The 1s-3d RIXS spectra show a ~ 0.5 eV dispersion in Γ–A direction, while no significant dispersion of the 1s-4p RIXS spectra is observed. Drastic photon polarization dependence is observed only for the 1s-3d RIXS spectra. Change in the 1s-3d RIXS spectra is observed above the N´eel transition temperature, indicating an intersite magnetic correlation. Taken altogether, the 1s-3d and 1s-4p spectra electronic excitations are interpreted as Mott-Hubbard and charge-transfer excitations, respectively. The charge-transfer gap Δ_{CT} = 3.69 eV, the Mott-Hubbard gap U_{eff} = 3.92 eV, and the crystal-field splitting $10Dq$ = 1.35 eV are determined, model independent from the experimental data. The Mott-Hubbard energy U = 2.47 eV, and Hund exchange J = 0.85 eV are derived using the multi-orbital Hubbard model.

A27

Effects of Chitosan on the Alignment, Morphology, and Shape of Calcite Crystals Nucleating under Langmuir Monolayers

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The growth of calcium carbonate crystals under Langmuir monolayers was investigated in the presence of chitosan, a soluble derivative of chitin added to the subphase to better simulate the polyelectrolyte-containing *in vivo* environment. Chitosan causes distinct concentration-dependent changes in the orientation, shape, and morphology of the calcite crystals nucleating under acid and sulfate monolayers. Our results suggest that polyelectrolytes may play essential roles in controlling the growth of biogenic calcite crystals.

A28

Isolation of Exchange- and Spin-orbit-driven Effects via Manipulation of the Axis of Quantization

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Double polarization photoelectron spectroscopy (DPPS) using circularly polarized x-rays and true spin detection has been performed using the 2p core levels of ultrathin films of Fe and Co. This includes both the separation into magnetization- and spin-specific spectra and an instrumental asymmetry analysis. How to selectively manipulate the manifestation of exchange and spin-orbit effects simply by choosing different axes of quantization is shown. Furthermore, the underlying simplicity of the results can be confirmed by comparison to a simple yet powerful single-electron picture.

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A29

In-field Scattering Studies Using a Cryogen-free 4-tesla Magnet at APS Beamline 4-ID-D

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This poster presents an overview of four different scattering studies performed using a 4-tesla cryogen-free superconducting magnet at APS beamline 4-ID-D. The studies described include a determination of the London penetration depth and critical-field anisotropy constants in superconducting MgB_2 using field-induced texturing in a powder sample [1], measurements of the spin-flop transition in the magneto-caloric Gd_5Ge_4 compound using resonant magnetic scattering [2], observations of the charge-order melting in multi-ferroic $LuFe_2O_4$ using single-crystal diffraction [3], and growth of magnetic domains through a meta-magnetic transition in $GdNi_2Ge_2$ using diffraction-contrast imaging [4]. The diverse set of materials-physics problems successfully studied using this instrument highlight the flexibility offered by the cryogen-free magnet design and illustrate the usefulness of in-field scattering studies.

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A30

Atomic and Electronic Structures of Copper-doped ZnO Thin Films

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Reports from several groups of observation of ferromagnetism in copper-doped ZnO thin films prepared under various conditions prompt this study of the atomic and electronic structures of these films using x-ray techniques. In this report we present the findings from these studies and discuss their implications to magnetic properties.

A31

In situ Lithium Ion (De)intercalation in MO_x: Application of X-ray Absorption Spectroscopy and Non-resonant Inelastic X-ray Scattering

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Due to their high energy and power density, lithium ion batteries are widely used for a variety of existing and emerging technologies. Comprehensive understanding of the local electronic structure and the mechanism of charge transfer upon intercalation/deintercalation of lithium ions is important to identify new battery materials with superior properties and to design battery with improved performance. The advent of third-generation synchrotron sources coupled with dedicated and specialized instruments for specific types of x-ray scattering studies has opened a new window to systematically investigate the structure-property relationship of advanced materials under operating conditions. By means of a combination of x-ray absorption spectroscopy and x-ray Raman scattering methods, we seek to provide definitive characterization of the redox chemistry of various metal oxides in operando batteries, a topic of continuing fundamental and applied interest.

A32

Ultrafast X-ray Phase Contrast Imaging of Gasless Reactive Systems at the Advanced Photon Source

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We report the results of an ultrafast x-ray phase-contrast imaging study of a gasless composite reactive system undergoing high heating rates (10^4–2.5×10^5 K/s), undertaken at the Advanced Photon Source (APS) at Argonne National Laboratory. Construction of an imaging system utilizing a high-speed CMOS camera (Vision Research Phantom v7.3) and the third-generation synchrotron at the APS allows for imaging of microstructural changes of the reactive system over previously unstudied time and length scales (<30 microsecond temporal resolution and ~2.2 micron spatial resolution). Using computer-assisted electrothermography (CAE), the heating rate of the gasless reactive system W-Si is controlled and its kinetics are measured. A physical description of the changes undergone by the system during melting and reaction are captured by the high-speed imaging system and temporally correlated to the recorded CAE data. These changes include microscale nucleated melting and the formation of fluid instabilities in the reaction products. Images of these events can, for the first time, allow for direct optical measurement of events that affect the kinetics of both reaction and phase transformation in gasless composite reactive system.

A33

The Hard X-ray Nanoprobe Goes Green

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We have utilized the hard x-ray nanoprobe (HXN) at 26-ID to obtain fluorescence maps of a hydroxide-activated geopolymer (fly ash + KOH solution). Geopolymers are currently being developed as an environmentally beneficial replacement to Portland cement for concrete production, offering comparable performance and cost while reducing greenhouse emissions by approximately a factor of 5. Given that cement production is responsible for up to 8% of global anthropogenic CO_2 emissions, this equates to the opportunity to reduce CO_2 by at least tens of millions of tons per annum worldwide. The exceptional spatial resolution and penetration power of the HXN enables the study of the poorly understood heterogeneity in order to tailor geopolymer formulations to ensure optimal performance for materials based on the available precursor materials. This study provides, for the first time, direct evidence of the formation of discrete high-calcium particles within the binder structure of a geopolymer synthesized from a low-calcium (2.6 wt.% as oxides) fly ash.

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A34

Gas-mediated Impact Dynamics in Fine-grained Granular Materials

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When a heavy sphere is dropped onto a bed of loose fine sand, a large focused jet of sand shoots upward. Experiments at reduced air pressure reveal that the jet in fact consists of two components: a wispy, thin jet that varies little with pressure followed by a thick air-pressure-driven jet. To observe the initial stages of jet formation inside the granular bed, we employ x-ray radiography using the high-intensity beams available at the Advanced Photon Source. This technique allows us to image the motion of the sphere through the bed and measure local changes in the bed packing density below the sphere. The x-ray movies reveal that gravity-driven collapse produces the initial thin jet, while the

compression of an air pocket trapped below the surface drives up the thick jet. We also find that the interstitial air alters the compressibility of the sand bed. In vacuum, a visible compaction front precedes the ball, while at atmospheric pressure, the sand flows out of the way of the ball, behaving more like an incompressible fluid.

A35

X-ray Scattering Experiments on Solid Helium

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Using x-ray synchrotron radiation, we have studied the nature of crystals and the properties of the defects in solid ^{4}He at temperatures down to 50 mK. Measurements of peak intensities and lattice parameters do not show indications of a supersolid transition. Scanning with a small (down to 10 □ 10 μm^2) beam, we resolve a mosaic structure consistent with numerous small-angle grain boundaries. The mosaic shows significant motion even at temperatures far from melting. When grown in aerogel, solid ^{4}He is polycrystalline with a hcp crystal structure (as in bulk) and a crystallite size of approximately 100 nm. In contrast to the expectation that highly disordered solid will have a large supersolid fraction, torsional oscillator measurements show a behavior that is strikingly similar to high quality crystals grown from the superfluid phase. The low temperature supersolid fraction is only ~3 □ 10^{-4} and the onset temperature is ~100 mK. When quenched from the liquid state, in addition to well-defined crystals, the solid develops a significant amorphous fraction. The supersolid state may well occur in the amorphous fraction.

A36

Small-angle X-ray Scattering (SAXS) Studies of Self-assembled Avian Photonic Nanostructures

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Organism structural colors are ubiquitous and are frequently used in intersexual signaling. Non-iridescent structural colors in avian feather barbs are produced by "quasi-ordered" or "amorphous" photonic nanostructures composed of beta-keratin (r.i. 1.54) and air. These nanostructures occur either as interconnected bicontinuous channels (spinodal decomposition like), or as a glass-like ordering of air spheres in beta-keratin (nucleation and growth like). Until recently, such amorphous biophotonic nanostructures were erroneously thought to produce color by incoherent Tyndall or Rayleigh scattering as opposed to coherent scattering.

Biologists are keen to characterize the physical mechanism of organism structural color production to understand the optical function and evolution of such biological signals. However, we need a precise knowledge of their three-dimensional ultrastructure for this, and current techniques like transmission electron microscopy have not provided sufficiently accurate data. We use small-angle x-ray scattering (SAXS) to characterize the spatial organization of mesoscale photonic materials in avian feather barbs. We present the first SAXS data on any bio-photonic material and discuss the results in light of the putative self-assembly of these avian barb nanostructures through phase separation kinetics of beta-keratin from the cellular cytoplasm. Self-assembled, optically isotropic feather barbs evolved for a consistent optical function, may thus provide a useful template for biomimetism.

A37

Pressure-induced Electronic Mixing and Enhancement of Ferromagnetic Ordering in Eu_X (X = O, S, Se, Te) Magnetic Semiconductors

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Monochalcogenides Eu_X (X = O, S, Se, Te) materials have attracted renewed interest for displaying fully spin-polarized bands and colossal magnetoresistance more pronounced than in manganite compounds making them attractive for potential use in spintronic devices. Although the ferromagnetic (FM) ordering temperature (T_C) is below 70 K in the bulk limiting its applications, a lattice contraction induced by pressure, chemical doping, or interfacial strain significantly increases T_C toward room temperature. An understanding of the changes in electronic structure responsible for the strengthening of the indirect FM exchange interactions, while key to enable further developments, remains elusive with models alternatively centered on f-d, s-f, or p-f mixing being proposed.

We exploited the element and orbital selectivity of Eu L-edge x-ray absorption spectroscopy in electric-dipole and electric-quadrupole channels to probe the spin-polarized electronic structure of Eu 4f (valence) and 5d (conduction) states as the lattice is contracted with chemical (Te -> O) or physical pressure in a diamond anvil cell. Our results pinpoint the relevant changes in electronic structure regulating T_C in these materials, and should help guide efforts aimed at tailoring exchange interactions in monochalcogenide thin films through manipulation of interfacial strain and chemical doping.

A38

Thermal Expansion of Diamond at Low Temperatures: High-energy-resolution X-ray Diffraction Studies

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Measuring thermal expansion of diamond at low temperatures is a challenging experiment due to the extremely low thermal expansion coefficient. Until now, existing experimental studies showed that in the temperature range below 100 K the values are on the order of 10^{-7} K^{-1} while the measurement uncertainty is about the same or greater. These data lack precision necessary to draw conclusions on utilization of diamond (C) as a diffracting crystal in many high-energy-resolution applications. Also, the large uncertainties may prevent observation of negative thermal expansion that exists for many compounds with diamond structure. We have recently measured thermal expansion of a diamond crystal using x-ray diffraction from C (9 9 5) planes in backscattering configuration. In the conducted experiment, a unique high-energy-resolution x-ray optics at sector 30 has enabled determination of the temperature dependent lattice parameter with relative accuracy 1×10^{-8}. It was found that in a studied temperature range (50–300 K) the temperature dependence of the thermal expansion coefficient follows a cubic law and the value can be as small as 3×10^{-9} K^{-1} at the low temperatures. No evidence of negative thermal expansion was observed.

A39

Evidence of Dynamical Spin Shielding in Ce from Spin-resolved Photoelectron Spectroscopy

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Using Fano effect measurements on polycrystalline Ce, we have observed a phase reversal between the spectral structure at the Fermi edge and the other 4f derived feature near a binding energy of 2 eV. The Fano effect is the observation of spin polarized photoelectron emission from nonmagnetic materials under chirally selective excitation such as circularly polarized photons. The observation of phase reversal between the two peaks is a direct experimental proof of Kondo shielding in Ce, confirming the predictions of Gunnarsson and Shoenhammer, albeit with a small modification.

[See Tobin et al., *EuroPhysics Letters*, **77**, 17004 (2007).]

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A40

Charge Excitations in the Stripe-ordered $La_{5/3}Sr_{1/3}NiO_4$ and $La_2(Ba, Sr)_xCuO_4$ Superconducting Compounds

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Charge excitations in stripe-ordered 214 compounds, $La_{5/3}Sr_{1/3}NiO_4$ and 1/8-doped $La_2(Ba, Sr)_xCuO_4$ are studied using resonant inelastic x-ray scattering in hard x-ray regime. We are observing ~1 eV excitations with the momentum transfer corresponding to the charge stripe spatial period both for the diagonal (nickelate) and parallel (cuprates) stripes. They are interpreted as collective stripe excitations or anomalous softening of the charge excitonic modes of the in-gap states.

A41

Resonant Inelastic X-ray Scattering in CE-ordered Bilayer Manganite

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Resonant inelastic x-ray scattering (RIXS) has recently emerged as valuable tool in the study of orbital excitations in transition metal oxides. We have performed RIXS measurements at the Mn K-edge in the half-doped bilayer manganite $LaSr_2Mn_2O_7$. Our sample was a non-reentrant single crystal with long range CE order down to lowest temperatures. We made wave vector dependent energy loss scans with energy transfers up to 15 eV in the (110) direction at three different temperatures, i.e., T = 75K (AFM CE ordered), 175K (PM CE ordered) and 250K (PM and no orbital order). In particular, we compare the temperature dependence of the 2eV peak with previous results on manganite perovskites [1].

[1] S. Grenier et al., *Phys. Rev. Lett.*, **94**, 047203 (2005).

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A42

The Momentum of Light

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We report the first time-resolved x-ray diffraction measurements of the transient acoustic perturbation produced by reflecting intense laser light off a near-perfectly reflective film. The sample consists of a high damage threshold broadband reflection coating deposited onto c-axis sapphire wafers. A model of the strain generated by transfer of momentum from light into the sample agrees quantitatively with the data, which shows an impulsive strain wave appearing in the sapphire substrate following propagation of the strain away from the coating layer. This work ultimately seeks to answer the question: "How much momentum does light transfer to a material through which it passes?" Future experiments on measuring the momentum of light in different geometries will be described, along with the instrumentation developments that have been undertaken to make this type of high speed yet sensitive measurement possible.

A43

New Method Uses X-ray Calorimetry to Map Phonon Dispersion Relations

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Phonon dispersion relations in crystalline materials are a manifestation of the interatomic bonding forces; as such, they play a fundamental role in many physical effects and phenomena. Direct mapping of phonon dispersion relations is traditionally carried out by inelastic x-ray scattering (IXS) or neutron scattering, but the data acquisition rates of these techniques are low. This talk reports a new method based on x-ray calorimetry that can be much simpler and more efficient. X-ray scattering intensities are measured at selected points in reciprocal space with suitably chosen polarization configurations; the thermal part of the scattering intensity is extracted by scanning the temperature of the sample. The intensity variations, governed by the phonon populations, are directly related to the energies of the phonons. Conceptually, the energy analysis in IXS is replaced by a thermal (calorimetric) analysis in the present method, with the heat content in the system at each point in momentum space measured by the thermal scattering intensity. Experiments on copper as a test case have yielded excellent results. This new method should be a useful addition to the arsenal available for studies of phonons and quantum phase transitions in solids.

A44

Carbon Dioxide Gas Storage in Bentonite and Sandstone Rocks Studied with Small-angle X-ray Scattering

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Capture and storage of carbon dioxide gas are important processes for the reduction of this green house gas in the Earth's atmosphere. Technologies to capture carbon dioxide gas from power plant facilities, for example, are becoming available and the challenge now lies in the storage of the captured gas. One potential area for storage is in deep geological formations where porous sandstone layers may be capped by low porosity shale rocks. We are investigating the carbon dioxide storage potential of sandstone and clay rich rocks using time-resolved small-angle x-ray scattering. Our *in situ* experimental approach is to apply 900 psi carbon dioxide pressure to a sample and collect data once every few minutes. Analysis of the small-angle scattering curve for these data yields information on the evolution of pore sizes or layer spacing for rocks with sheet-like structures. The evolution of the invariant, a quantity reflecting differences in electron density, shows how quickly pores become filled with carbon dioxide. These data are necessary to evaluate the potential for carbon dioxide storage in the pore spaces of rocks buried deep within sedimentary basins such as the one under our feet, the Illinois basin.

A45

X-ray Tomography of Damaged Bone Tissue Labeled with Functionalized Gold Nanoparticles

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Understanding of microdamage accumulation mechanisms and concomitant mechanical property degradation in bone tissue is limited *in vivo* due an inability to noninvasively detect damage. Functionalized gold nanoparticles (Au NPs) show potential as damage-specific x-ray contrast agents due to a relatively high attenuation compared to bone, which would allow for nondestructive and three-dimensional imaging. Human bone samples were machined to 2 mm in diameter, prestained with a calcium-specific fluorophore, and loaded in cyclic uniaxial tension to a 10% degradation in secant modulus, which is known to lead to the accumulation of microdamage. Samples were then stained with alendronate functionalized Au NPs to enable calcium-specific binding by bisphosphonate ligands. In order to investigate detection limits, photolithography was used to prepare imaging phantoms comprising gold islands of varying size deposited on an aluminum substrate. Aluminum was chosen due to its similar x-ray attenuation to cortical bone. Specimens were imaged by micro-computed tomography to determine feasibility of detecting gold as an x-ray contrast agent in bone, using either a commercial polychromatic or monochromatic synchrotron x-ray source. Commercial microtomography was unable to detect Au NPs but synchrotron radiation around the LIII edge of gold showed that functionalized Au NPs stained damaged regions in human bone.

A46

Structural Stability of Terbium Oxide under High Pressure

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Poster displayed.

A47

Ultra-incompressible Carbon Phase Transforms from Solvated Structured C_{60}

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Poster displayed.

A48

Improved Focusing Capability for Inelastic X-ray Spectrometer at Sector 3: A Combination of Toroidal and Kirkpatrick-Baez (KB) Mirrors

A. Alatas, E.E. Alp, L. Gao, B. Leu, W. Sturhahn, T.M. Toellner, H. Yavas, and J. Zhao
Advanced Photon Source, Argonne, IL 60439

Poster displayed.

A49

Single- and Multi-element Drift Detectors for Specialized X-ray Applications

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A 50 mm^2 silicon drift detector (SDD) has been successfully applied to x-ray fluorescence, total reflection x-ray fluorescence, synchrotron, and microanalysis applications. The SDD offers a large solid angle, excellent energy resolution, and high-count-rate performance. Several unique four-element systems have been developed and are currently being used in high-count-rate synchrotron applications at the Advanced Photon Source at Argonne National Laboratory. We are pursuing several approaches in its development plan to achieve the required improved performance:

A. Reducing entrance window thickness—a shallower entrance window results in a better peak-to-background (P/B) ratio, as well as better low energy detection. A significantly improved P/B ratio, as well as the detection of C, B, and Be has been achieved with the new shallower entrance window SDD.

B. A thicker device would enable the detector to be more efficient at higher energy. The current device's thickness is 0.35 mm with an efficiency of 0.28 and 0.08 for 20 keV and 30 keV, respectively. A recent experiment with a 1-mm-thick SDD showed an efficiency that matches the theoretical values of 0.6 and 0.4 for 20 keV and 30 keV, respectively. Spectra of ^{109}Cd (22 keV) from 0.35- and 1.0-mm-thick SDDs are shown in Figure 1.

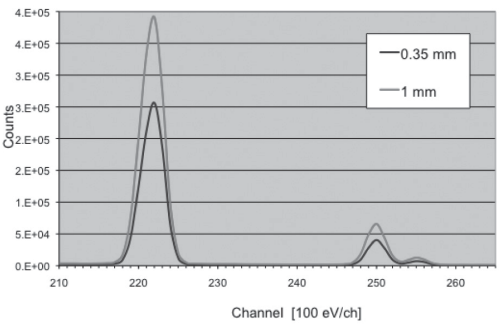

Fig. 1 - Comparison of 109Cd spectra from 0.35mm and 1mm thick SDDs.

C. The characterization of nanoscale materials at high spatial resolution has become increasingly important to state-of-the-art research. A number of instruments ranging from Argonne's subangstrom electron-optical instrument to the x-ray nanoprobe are capable of extraordinary resolution to study these materials. A special 50 mm^2 SDD with a larger solid angle for use in nanotechnology characterization was designed and assembled into a scanning electron microscope at the Electron Microscopy Center at Argonne. The detector is shown in Figure 2.

D. A detector array larger than four elements is a current project at SNTUS. Several designs are considered in which one of them is an array composed of multiples of four elements each.

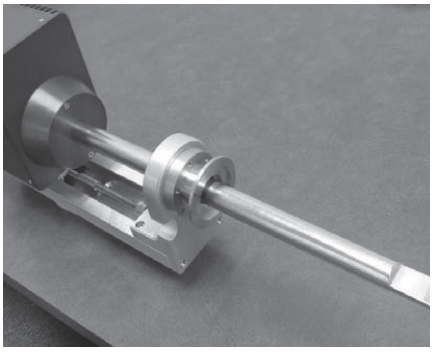

Fig. 2 - Photo of custom Vortex-EM for ANL SEM.

A50

Microwave Kinetic Inductance Detector (MKID) Development for APS X-ray Focal Plane Arrays

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We are developing energy-resolving focal plane arrays utilizing superconducting detectors and a simple, scalable, readout scheme. The concept can deliver an x-ray detector with large pixel counts, high sensitivity, and Fano-limited spectral resolution. The detection scheme employs the monotonic relation between the kinetic surface inductance Ls of a superconductor and the density of quasiparticles generated by photon absorption n, which holds even at temperatures far below T_c. This allows a sensitive readout of the number of excess quasiparticles in the detector via monitoring of the transmission phase of a microfabricated thin film resonant circuit. The approach is known as a microwave kinetic inductance detector (MKID). The response time of an x-ray MKID sensor is ~10 μs, with an energy resolution of ~30 eV or better at 5.9 keV. Passive frequency multiplexing can be employed to read out ~1,000 pixels with a single high electron mobility transistor amplifier. We will describe and discuss our preliminary results with Al/Sn MKIDS fabricated in Argonne's Materials Science Division and Center for Nanoscale Materials and tested at University of California, Santa Barbara.

A51

Current Status of the 7-BM Fuel Spray and Time-resolved X-ray Imaging Beamline

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We are developing a new bending magnet (BM) beamline at sector 7 of the Advanced Photon Source specializing in ultrafast x-ray imaging based on absorption. The instruments in 7-BM-B will provide capabilities for microsecond x-ray radiography and tomography of liquid sprays. These techniques require a highly intense monochromatic x-ray beam with photon energy tunable between 6 and 12 keV. The x-ray beam is provided by a bending magnet beam monochromatized by a double-multilayer monochromator with a 2% bandpass. This presentation will show the current plan for the facility and preliminary performance data on the beamline optics.

A52

Microcrystallography Developments at GM/CA-CAT

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Recently several important structures have been solved using microcrystallography techniques that could not have been solved otherwise with conventional crystallography. At GM/CA-CAT we continue to develop microcrystallography capabilities for difficult problems such as membrane proteins grown in the lipidic cubic phase. Our "mini-beam" collimators have been redesigned to improve reliability and robustness. We now offer beam sizes of 5, 10, and 20 microns at the click of a button. We have also designed and implemented a goniometer with a 1-micron peak-to-peak sphere of confusion that can carry sample position stages with nanometer resolution and repeatability. A Fresnel zone plate can be positioned before the goniometer providing a 1-micron beam for studies on radiation damage and applicability to microcrystallography of crystals with largest dimension on the order of 1 micron. Results will be reported from our initial development efforts to produce a high-intensity microfocus beam by successive demagnification, i.e., re-imaging a virtual source.

A53

A High-field Pulsed Magnet Instrument for X-ray Studies of Materials

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We present an extremely high-field pulsed magnet system for x-ray studies of materials at the Advanced Photon Source. High-field pulsed magnets for synchrotron x-ray applications are not the solution to all problems requiring high magnetic fields, but they are the only approach to many. The high-field environment capabilities provided by this instrument for scattering and spectroscopy studies are unique in the United States. Currently, 30-tesla split-coil and long-pulse solenoid magnets are in use for scattering and spectroscopic experiments, respectively. They have been developed at Tohoku University using high-tensile strength and high conductivity CuAg wires. Pulsed fields (1–10 ms in duration) are generated using a configurable bipolar capacitor bank (3 kV, 40 kJ). Low-energy, small-bore split coils are mounted on the cold finger of a closed-cycle He cryostat capable of a repetition rate of ~10–20 minutes for peak fields in the range of 20–30 tesla. Long-pulse large-bore solenoids are designed to be cooled by liquid nitrogen. Time-resolved scattering data are typically collected using a fast APD detector or a strip detector. Results from preliminary scattering studies of structural effects and magnetostriction in a geometrically frustrated magnet will be presented and future opportunities in experiments and instrumentation will be discussed.

Use of the Advanced Photon Source is supported by the U. S. Department of Energy, Office of Science, under Contract No. DE-AC02-06CH11357.

A54

Multilayer Laue Lenses for Nanofocusing of Hard X-rays

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A multilayer Laue lens (MLL) is an x-ray focusing optic fabricated from a multilayer structure consisting of thousands of layers of two different materials produced by thin-film deposition. We have found that WSi_2/Si is a promising material system due to the low film stress between deposited layers and high atomic number difference of the system. The sequence of layer thicknesses is controlled to satisfy the Fresnel zone plate law. The latest MLL deposited consist of 5165 alternating layers of Si and WSi_2. The thicknesses of layers on each side vary monotonically from 4 nm to 160 nm and the total thickness of the stack is about 40 microns.

The challenges we face in this project are the deposition of thousands of graded layers with accurate thickness control and the challenging sectioning and polishing of such a delicate and small sample for use with hard x-rays in a nanofocusing experiment. With the help, training, and support of experienced EMC staff, we were able to overcome the

challenges. High-resolution images at very high magnification were obtained from the Hitachi S-4700 SEM in the EMC facility over last several years. These images were essential as feedback for our deposition and sectioning process. Sequences of overlapped images were used in accurate image processing in order to achieve accurate thickness and position data for each single layer.

We have measured a line focus of 16nm width with an efficiency of 31% at a wavelength λ = 0.064 nm (19.5 KeV) using partial MLL structure with an outermost zone width of 5 nm. This is the hard x-ray nanofocusing world's record [1].

[1] H. C. Kang, Hanfei Yan, Robert P. Winarski, Martin V. Holt, J. Maser, C. Liu, R. Conley, S. Vogt, A. Macrander, and G. B. Stephenson "Focusing of Hard x-rays to 16 nanometers with a multilayer Laue lens," *Applied Physics Letters*, **92**, 2211114 (2008).

A55

Laboratory Simulations of Astrophysical Plasmas

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We report on preparations for future experiments to investigate the x-ray photoionization of highly charged ions. For these experiments, we will bring an electron beam ionization trap to the APS and install it on the 12-ID beamline together with a metal vapor source and a high-resolution x-ray microcalorimeter. These state-of-the-art instruments will permit the first laboratory measurements of the x-ray spectra following *K*-photoionization in a plasma of highly charged metallic ions. Satellite-based x-ray telescopes and spectrometers are returning exciting new observations from a wide variety of cosmic sources such as stellar coronae, supernova remnants, galaxies, clusters of galaxies, active galactic nuclei, and x-ray binaries. Understanding of the physical conditions of these astrophysical objects comes primarily from the spectroscopic information that is gathered and compared to modeling calculations. Our experiments will serve as an important benchmark for the existing theoretical atomic data in order to gauge the accuracy and improve the modeling of those x-ray spectroscopic measurements. During the next year, we plan to measure high-resolution x-ray fluorescence spectra following *K*-photoionization of Ne-like Fe ions, a ubiquitous constituent of astrophysical plasmas. We will describe our preparations and show the results of the commissioning of the equipment.

A56

Highly Sensitive, Nondamaging Imaging of Protein Crystals at Cryogenic Temperatures

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Second-order, non-linear optical imaging of chiral crystals (SONICC) was applied to protein crystal detection and characterization at cryogenic temperatures. The need for automated crystal centering has motivated the development of many techniques that determine the position of protein crystals frozen in loops (e.g., brightfield image analysis, birefringence, intrinsic UV fluorescence, and x-ray diffraction-based centering). These techniques have been limited by a lack of contrast, minimum detectable crystal size, and sample damage, respectively. SONICC provides the necessary contrast by effectively eliminating the background from centrosymmetric materials (e.g., amorphous water and cryoprotectants). SONICC has been used to detect two-dimensional protein crystals down to 2 microns squared in size. In addition, diffraction studies showed no measurable laser damage to crystals that were exposed for fifteen minutes with ~500 mW laser power, where a typical image requires three minutes exposure at ~100 mW. Images taken at multiple angles of both soluble and membrane protein crystals frozen in loops demonstrate the general applicability of SONICC. High image contrast, even for small crystals, and the lack of measurable crystal damage make this technique well suited for automated centering.

A57

SBCserver—A General Beamline Software Interface

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The Structural Biology Center's experiment control software has recently been redesigned and implemented with the main goal of ensuring the flawless automated operation of wide range of beamline tasks including robotic sample mounting, automatic crystal alignment, automated crystal evaluation and fluorescence scanning, automated energy changes, and various batch movements of different beamline components triggered by well defined beamline events. That achievement was made possible by careful implementation of the three-tier software architecture: the modified EPICS layer; the SBCserver as a middleware; and the variety of clients such as the SBCcollect, the main beamline user operations GUI, HKL3000 data analysis, processing and structure determination software, and others. The presentation shows details of SBCserver architecture, its main features, interactions with the beamline components and software clients, as well as its modularity and flexibility. The SBCserver's effectiveness and reliability has been evaluated using robotic sample mounting and the automatic cryoloop alignment. The automated procedure involving automounting and autocentering of 700 crystals showed a 93% success rate for centering on the loop and a 76% success rate for centering on the crystal. This system has been in operation since 2007 on both SBC beamlines and has been used for the alignment of more than 21,300 samples.

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A58

Current State of the 11-ID-B Dedicated Pair Distribution Function Beamline at the APS

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We describe the current status of the 11-ID-B beamline including pair distribution function capabilities and recent updates and developments of scattered beam detection, sample environment including high-pressure equipment, and data acquisition software.

A59

X-ray Detector Development Projects

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We describe several x-ray detector projects currently in development for timing applications at the early stages of engineering design or testing. In all cases we work closely with small U.S. companies and tailor the specifications around specific Advanced Photon Source beamline applications, timing modes, and fill patterns. These detectors utilize custom sensors or have application-specific integrated circuits and serve as focal points for our learning in these areas. This learning in turn feeds into the curriculum in our university's electrical engineering department. The projects involve a P0, 3.6-microsecond readout strip detector CCD; a 12-nanosecond, two-energy resolving, pixel array detector; a subnanosecond avalanche photodiode array with GHz analog-to-digital conversion for time resolution measurements; and two-microsecond range patterned photodiode arrays.

A60

Design and Test of a Two-dimensional Precision Weak-link Stage System with Subcentimeter Travel Range and Subnanometer Resolution

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Recent developments in hard x-ray focusing on the nanometer scale with linear multilayer Laue lenses (MLLs) as reported by H. C. Kang et al. [1] have demonstrated a promising new x-ray optic for focusing hard x-rays to a spot in few nanometers. To use x-ray optics with nanometer resolution limit, scanning x-ray nanoprobes with corresponding mechanical positioning capability need to be designed. In particular, positioning stages with both subnanometer resolution and a positioning/scanning range of several millimeters is required.

Based on our design of precision weak-link stages with interferometric encoders for the CNM/APS hard x-ray nanoprobe at APS sector 26 [2, 3], we have developed a new two-dimensional (2D) weak-link stage system for nanopositioning of a specimen holder. This system provides subnanometer resolution, coupled with subnanometer metrology, at a travel range of several millimeters. The two-dimensional weak-link stage system is designed with high structure stiffness using laminar overconstrained weak-link mechanisms [4, 5].

In this paper we present a novel mechanical design for laminar weak-link mechanisms with subcentimeter travel range and subnanometer positioning resolution. Finite-element analyses and test results with a laser Doppler displacement meter are also presented [6].

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

[2] J. Maser et al., *Proc. 8th Int. Conf. X-ray Microscopy, IPAP Conf. Series 7*, 26–29, (July 2006).

[3] D. Shu et al., *Proc. 8th Int. Conf. X-ray Microscopy, IPAP Conf. Series 7*, 56–58, (July 2006).

[4] U.S. Patent granted No. 6,607,840, D. Shu, T. S. Toellner, and E. E. Alp, (2003).

[5] U.S. Patent granted No. 6,984,335, D. Shu, T. S. Toellner, and E. E. Alp, (2006).

[6] D. Shu and J. Maser, *Proc. 23th ASPE Annual Meeting and 12th ICPE*, 23–26, (Oct. 2008).

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A61

Mechanical Design of an MLL Multidimensional Alignment System with Nanometer-scale 2-D Focusing

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Recent developments in hard x-ray focusing on the nanometer scale with linear multilayer Laue lenses (MLLs) as reported by H. C. Kang, et al. have demonstrated a promising new x-ray optic for focusing hard x-rays in a few nanometers. Using tilted partial MLL structures, a one-dimensional focus as small as 30 nm with efficiencies up to 44% has been performed with 19.5-keV synchrotron radiation [1]. The high efficiencies should make it practical to produce a point focus using two MLLs in a crossed configuration [2]. This challenging technical approach needs a precision multidimensional alignment apparatus.

In this paper we present a novel mechanical design for a multidimensional alignment apparatus, which can achieve a two-dimensional hard x-ray focusing in the nanometer scale with two MLLs in a crossed configuration. The novelty of this new mechanical design is the positioning stability and compactness of its unique nested structure. Experimental test results are also presented [3].

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

[2] J. Maser et al., *Proc. SPIE-Int. Soc. Opt. Eng.*, 5539, 185 (2004).

[3] D. Shu, H. Yan, and J. Maser, to be published in *Nucl. Instrum. and Methods A, Proc. SRI-2008*, Saskatoon, June 10-13 (2008).

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A62

A Hardware-assisted Real-time XPCS Data Reduction System

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We describe recent progress on a system that will perform real-time or post-acquisition autocorrelations of large-volume (streaming) multispeckle x-ray photon correlation spectroscopy (XPCS) data sets. The system consists of a fast (≥30 Hz) direct detection CCD (~1 megapixel) interfaced to a frame grabber with a considerable on-board memory and a large field-programmable gate array. The frame grabber and host computer are connected to a computational cluster capable of reducing the data in real time. The CCD camera streams data to the frame grabber that performs dark subtraction, thresholding, and eventually mild compression before streaming data to the cluster where time

autocorrelations are performed. The CCD camera and frame grabber are controlled via areaDetector software while a custom Matlab GUI application manages the entire process via EPICS channel access and web service access to a cluster-based service. Ongoing tests demonstrate that the system will be capable of time autocorrelating rapidly streaming XPCS data in real time. Our efforts will advance the general user program at 8-ID. In addition, the framework that we have developed will facilitate other efforts at rapid high-volume data reduction and the adoption of even faster XPCS detectors on the near horizon.

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A63

Nanoradian Angular Stabilization of X-ray Optical Components

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X-ray free-electon laser oscillator (XFELO) has been recently proposed [1]. Angles of Bragg mirrors of the XFELO optical cavity must be continuously adjusted to compensate instabilities and maximize the output intensity. An angular stability of about 10 nrad is required [2]. To approach this goal, a feedback loop based on a null-detection principle was designed and tested using a high-energy-resolution monochromator (HRM) and a high-heat-load monochromator. Angular stability of about 50 nrad has been demonstrated for diffracting crystals of the HRM.

[1] K. Kim, Y. Shvyd'ko, and S. Reiche, *Phys. Rev. Lett.*, **100**, 244802, (2008).

[2] K. Kim and Y. Shvyd'ko, *Phys. Rev. STAB*, **12**, 030703, (2009).

A64

High-energy-resolution TES Microcalorimeter

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Superconducting transition edge sensor (TES) microcalorimeter for x-rays has been a topic of active research. New results have been published continuously, especially on the front of photons count rate. A microcalorimeter pixel consists of a metal absorber and a TES. A single x-ray photon raises the temperature of the thermally isolated absorber and TES. The TES, which is operated under voltage bias with strong negative electrothermal feedback at a cryogenic temperature, reads the small temperature change of the absorber with a reduced time constant, thus the absorbed x-ray energy. A single-pixel TES microcalorimeter, which is generally a few hundred micrometers by a few hundred micrometers, has an energy resolution of a few eV and a time constant of a few hundred microseconds. Integrated into large arrays of pixels, with superconducting quantum interference devices (SQUIDs) for readout, TES–microcalorimeters can have ~1000 counts per millimeter square for x-rays between 6 keV and 12 keV, ~ 4000 counts per millimeter square below 2 keV. The total calorimeter area can be made to be a few tens of millimeter squares with the current technology. The resulting detector will enable new and exciting spectroscopic experiments in the fields of material, environmental, biological, and medical science that were previously hard to conduct or even not possible. In collaborations with NIST, we have been developing TES microcalorimeters and related technologies at Argonne. In this poster, we present our TES microcalorimeter configuration, readout SQUID electronics, current status of development, test results, and future plans.

A65

Fast CCD Detector Collaboration between ANL and LBNL (Status and Preliminary Measurements)

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This poster will describe the status of the ongoing Fast CCD detector collaboration between the Advanced Photon Source at Argonne National Laboratory (ANL) and the Advanced Light Source at Lawrence Berkeley National Laboratory (LBNL).

The detector uses a custom LBNL-designed CCD with 480 × 480 pixels and 96 outputs, giving a nearly column parallel and therefore very fast (200 fps) readout. LBNL designed the hybrid board with the CCD and front-end electronics while ANL developed the back-end readout electronics and data acquisition system. In January, LBNL delivered their half of the detector and we assembled the detector at ANL. We are in the process of optimizing and characterizing the detector. The poster will present the detector status and preliminary measurements along with plans for future CCD devices.

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A66

Nanotomography at 2-BM, Advanced Photon Source

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Synchrotron-radiation-based microcomputed tomography (SR-μCT) is a powerful tool to obtain three-dimensional structure information in material science/engineering and biology. Combined with visible light microscope lenses, SR-μCT has micron-level resolution. Although the resolution of SR-μCT is significantly improved compared to medical CT, higher spatial resolution is highly desired in many material science/engineering and biology researches. In a recent development of nanotomography project at beamline 2-BM of the Advanced Photon Source at Argonne National Laboratory, we have demonstrated large field of view (50 μm) high-resolution (150 nm) nanotomography. With the optimization of x-ray optics, we expect to implement 100-μm field of view and 100-nm resolution nanotomography in the near future.

A67

Recent Development of Hard X-ray Transmission Microscopy at the 32-ID Beamline at the APS and Applications

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[4] Xradia Inc. 5052 Commercial Circle, Concord, CA 94520
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The high-energy, full-field transmission x-ray microscopy (TXM) at beamline 32-ID of the Advanced Photon Source has made successful achievements in both instrumentation capabilities and scientific applications. We demonstrated the world's top first-order Rayleigh resolution below 30 nm in the hard x-ray regime using home-made Fresnel zone plates. We were able to significantly improve the sensitivity, for example, to nanoparticles used for biological staining by optimizing Zernike phase contrast. We have extended the energy capability up to 20 keV, which allows us to perform *in situ*/real time measurements on thick, dense materials. Due to its high-energy, high-resolution, and high-contrast capability, the TXM has opened up new and exciting scientific applications for materials systems that are too opaque for soft x-rays or electrons, with the scientific aim for real-time and/or *in situ* investigation of three-dimensional nanoscale structures. We will present the instrumental details and the applications including real-time nanoscale imaging of dendrite growth during Cu electro-chemical deposition process.

A68

Enhanced USAXS Capabilities for Solution-mediated Nanoscale Processing, Nanostructural Materials Imaging, and High-spatial-resolved Gradient Microstructure Characterization

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Under PUP agreement 59 with the National Institute of Standards and Technology, the ultra-small-angle x-ray scattering (USAXS) instrument at Advanced Photon Source XOR sector 32-ID has been continually upgraded over the past two years to achieve new levels of performance that are needed for addressing several emerging issues in materials microstructure characterization. The need to improve dispersion quality by aligning and length-sorting dilute nanowire and carbon nanotube suspensions in an extensional flow geometry has required improved high-spatial-resolved gradient microstructure characterization (over the full nanometer-to-micrometer length scale) in dilute suspensions of elongated nanoparticles. This has required improved signal to noise and an improved understanding of the requirements for linear intensity calibration over the full USAXS Q and intensity ranges. The theoretical basis of USAXS imaging has been comprehensively established, and new USAXS optics have been developed to provide combined USAXS/USAXS imaging in the next-generation USAXS instrument design. Design requirements and performance parameters for studies of high-spatial-resolved (few micrometer level) gradient microstructure characterization have been established, including those for precision sample stage motion, linearity of intensity calibration, correction for multiple scattering, and anomalous USAXS measurements at multiple x-ray absorption edges. Finally, recent experiments show promise for future parallel x-ray photon correlation spectroscopy/USAXS measurements of structure and dynamics in new materials.

A69

Time Delay of Decay of Laser-excited Electrons/Holes in Ge

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A laser pump/x-ray absorption fine structure (XAFS) probe study of Ge using the high efficiency facility at PNC/XOR-CAT at sector 20 has discovered some surprising results of how the excited electrons/holes (e/h) decay. The 250-femtosecond pulse laser is triggered at the frequency of the APS ring so as to use the x-rays of one pulse of the 24-singlet mode at 100% efficiency. The higher efficiency use of the ring's x-rays (about two orders more efficient than usual) allowed the measurement of the XAFS spectra at a large number of delay times between laser pump and x-ray probe and at different laser powers. The XAFS data determined the time dependence of the relative distances and their vibration amplitudes of the first and second Ge neighbors. In the diamond structure of Ge, the first neighbors are the basis atoms and the second neighbors the lattice constant distances. The laser pulse excites coherent long wavelength optical phonons essentially instantaneously, which excites only the vibrations of the first neighbor [1] while to excite the second neighbor requires decay of the excited e/h. The surprising result is that this decay time is roughly proportional to the laser power at small powers, saturating at about 10–12 ps at higher powers. This proportionality to the power is contrary to what is expected by anharmonic phonon-phonon coupling. It is proposed that the excited e/h initially clump together into metastable droplets, and only after the decay of the droplets will the second neighbor vibration be excited by e/h-incoherent phonon coupling. As more charge clumps together with higher density of excited e/h their binding energy is increased, delaying their decay.

[1] T. Pfieifr, W. Kutt, H. Kurz, and R. Scholz, *Phys. Rev. Lett.*, **22**, (1992).

A70

Determination of the Shape and Dimensions of Interconnected Nanoparticle Aggregate Structures in Phase-segregated Microstructures

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Ultra-small-angle x-ray scattering (USAXS) and stereology measurements were used to determine the dimensions of nanoparticle aggregate structures in polymer-matrix composite systems that form percolating phase-segregated networks. The samples investigated contained polymethylmethacrylate (PMMA) as the matrix phase and indium tin oxide (ITO) or carbon black (CB) nanoparticles as the filler phase. These specimens have been shown to percolate at compositions with <0.5 vol.% filler [1–4]. USAXS imaging [2] and transmission optical microscopy [1, 3, 4] were previously used to demonstrate the presence of the percolating networks in the opaque CB-filled PMMA composites and the transparent ITO-filled PMMA composites, respectively. In this paper, we report on the analysis of USAXS intensity curves used for determining the diameter of the filler aggregate-wire structures. Percolation does not occur until the nanoparticles aggregate into three-dimensionally interconnected micron sized wires. Therefore, the unified rod model was applied to model the USAXS scattering data below and near the percolation threshold. The USAXS information was combined with stereological measurements of the edge lengths of the deformed PMMA particles to estimate the volume fraction of fillers necessary to promote percolating networks in these materials. Additionally, it is shown that at compositions above the percolation threshold, a transition from interconnected one-dimensional wires into interconnected two-dimensional sheets occurs.

[1] R. Ou, S. Gupta, C. A. Parker, and R. A. Gerhardt, "Fabrication and Electrical Conductivity of PMMA/CB composites: Comparison between an ordered carbon black-nanowire segregated structure and a randomly dispersed carbon black nanostructure," *J. Phys. Chem. B,* 110[**45**], 22362–22370, (2006).

[2] L. E. Levine, G. G. Long, R. A. Gerhardt, R. Ou, J. Ilavsky, and C. A. Parker, "Self-Assembly of Carbon Black into Nanowires that Form an Interconnected 3-Dimensional Micro-network," *Applied Phys. Lett.*, **90**, 014101, (2007).

[3] C. J. Capozzi and R. A. Gerhardt, "Novel Percolation Mechanism in PMMA matrix composites containing segregated ITO nanowire networks," *Adv .Functional Matls.*, **17**, 2515–2521, (2007).

[4] C. J. Capozzi, Zhi Li, R. A. Gerhardt, and R. J. Samuels, "Impedance Spectroscopy and Optical Properties of polymethylmethacrylate/indium tin oxide Nanocomposites with 3-dimensional Voronoi Microstructures," *J. Applied Physics*, **104**(11), 114902/1–114902/10, (2008).

A71

Symmetry Analysis and Universality of Charge-transfer Excitations in Undoped Cuprates from Cu K-edge RIXS

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The use of new experimental probes such as resonant inelastic x-ray scattering (RIXS) can be expected to lead to important advances in our understanding of strongly correlated electron systems. RIXS has been extensively developed at the Advanced Photon Source to study the momentum and energy dependence of electronic excitations. Here we present two distinct experimental advances based on the study of undoped cuprates which can be viewed as model Mott insulators. First, we demonstrate the presence of outgoing photon polarization effects at the Cu K-edge of Nd_2CuO_4 which, surprisingly, validate the use of conventional Raman selection rules, and we find that the RIXS spectrum is rich in excitations of various symmetries. Second, we show evidence of universality among the momentum-dependent charge-transfer excitations of Nd_2CuO_4, La_2CuO_4, and $Sr_2CuO_2C_{l2}$. The observed universal features are compared to theoretical models, demonstrating how electron correlations are crucial to the physics of the CuO_2 plane. The new insights presented here can be expected to help guide future RIXS studies.

A72

Tomographic Study of Dealloying and Coarsening Behavior of Nanoporous Gold by Transmission X-ray Microscope

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Nanoporous gold has been widely studied in terms of its novel mechanical, optical, structural, and chemical properties as well as its application such as sensors and mechanical actuator, surface enhanced Raman scattering, catalysts, electrodes of fuel cells, etc. Nanoporous gold can be fabricated by dealloying silver-gold alloy and its structure and properties can be tuned through dealloying and coarsening processes, which are chemical and heat treatment, respectively. To fully understand the dealloying and coarsening kinetic behavior in the complex interconnected nanoporous structure, three-dimension and time-resolved imaging technique is critical. Here we present imaging and tomography on the structural evolution during dealloying and coarsening of nanoporous gold using transmission x-ray microscope. A dealloying front was observed as the interface between silver-gold and nanoporous gold. And the time-dependent coarsening behavior has been quantified for different temperatures. X-ray imaging technique has played a critical rule in this research for its non-destructive and large-volume imaging capability in three-dimension. These results are significant for further application on nanoporous gold while all properties are related to structure and are also critical for fundamental science in terms of the kinetics and diffusion behavior in nanoporous materials.

A73

Long-period X-ray Standing Wave Studies of Self-assembled Nanoscale Dielectrics

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Organic thin-film transistors (OTFTs) are envisioned as the successors for classical thin-film transistors in novel electronics applications requiring large coverage area, structural flexibility, low temperature processing, and low cost. While significant research has been directed towards the semiconductor component of the OTFT, there has been relatively little research on the transistor's dielectric, which is critical to the device's electronic behavior. Recently, however, self-assembled nanoscale dielectric (SAND) multilayers composed of alpha,omega difunctionalized hydrocarbon chains, octachlorotrisiloxane capping layers, and highly polarizable "push-pull" stilbazolium dielectric layers have shown promise in organic dielectric applications. In this study, the motion and position of the heavy halide counteranion under both static and applied fields is of interest. The behavior of the counteranion is closely coupled to that of the rest of the molecule, and its behavior conveys information about the fundamental electronic properties of the dielectric itself. Understanding the counteranion behavior will allow for the tailoring of molecules that have high dielectric constants, low leakage currents, and high layer capacitances for application in high-performance non-conventional electronics. Here, we report initial findings of long-period x-ray standing wave and x-ray reflectivity studies of SAND films with varying z-offsets as under zero-field conditions as proof of concept. To better understand the dielectric's behavior in device applications, future experiments will be performed under applied electric fields in order to allow for *in situ* monitoring of counteranion behavior.

A74

Atomic-scale View of Redox of WO_x Supported on α-Fe_2O_3 (001)

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Ultrathin metal-oxide layers deposited onto oxide surfaces have wide applications in catalysis and chemical sensing. Supported tungsten (W) oxides are among these. If the atomic-scale geometrical and electronic surface structure of WO_x could be predicted, this would impact our understanding of numerous chemical processes. As a model catalytic system, atomic layer deposition (ALD)-grown WO_x on hematite (0001) was used for finding the positions of W with respect to the support lattice and its sensitivity to the reduction-oxidation (redox) cycle. Atomic force microscopy

is used to study the surface morphology changes. X-ray fluorescence and x-ray standing waves (XSW) are used in combination to determine the geometric structure changes during the redox reaction. The XSW results for 1/3 ML W show that W cations on the surface occupy different positions in the oxidized and reduced states. The ALD as-deposited W shows strong correlation vertically but no correlation laterally. Oxidization causes the W to be ordered and they occupy some special adsorption sites. However, in the reduced phase, W cations change their adsorption sites. Atomic density maps created from *in situ* XSW measurements give us direct information for the W cations surface site location. Finally, x-ray photoelectron spectroscopy is used to correlate the W oxidation state(s) with the above redox induced structural changes. A model is proposed to explain the reversible geometrical/electronic structure changes during this redox reaction.

A75

XAFS Studies of ZnO-based Spintronics Materials

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ZnO doped with transition metals is a promising candidate for a room temperature diluted magnetic semiconductor (DMS). While room temperature magnetism has been observed in doped ZnO by several groups, there is still much debate over whether these represent true DMS behavior. Magnetic nanoprecipitates or secondary phases are potential complicating factors. Such phases and precipitates can be difficult to detect in thin epitaxial films, the form in which many of these materials are made. In this paper we present as examples x-ray absorption fine structure data for Mn and Co doped ZnO thin films. For the Mn doped ZnO, the Mn is found to be Mn(II) and to reside in the tetrahedral Zn sites. There is no evidence for secondary phases or metallic nanoparticles. For Co doped ZnO the situation is more complicated. Many of the samples showed evidence for metallic nanoparticles. The nature of these nanoparticles depends on the preparation conditions. For films prepared with high Co concentrations and an Al co-dopant, the nanoparticles were close packed (hcp or fcc) Co metal. For another set of films, which were annealed in Zn vapor to induce magnetism, the metallic particles resembled CoZn. Both Co and CoZn are magnetic and can contribute a spurious magnetic signal unrelated to a true DMS signal.

A76

Phase-contrast Imaging of Superalloys Subject to High Cycle Fatigue at Extreme Temperatures

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Nickel-base superalloys, used in critical components such as turbine blades, are subject to extreme temperatures and very high cycle fatigue. Fatigue instrumentation operating at 20 kHz can simulate the transient conditions experienced in aircraft turbines to understand fatigue-crack initiation and propagation in nickel-base superalloys. Although *in situ*, real-time, three-dimensional measurements are impractical or impossible at high temperatures and cyclic frequencies with conventional microscopy techniques, synchrotron x-ray radiography is ideal. We installed a portable ultrasonic-fatigue instrument developed at the University of Michigan at Sector 32-ID for crack initiation in dog-bone-shaped, single-crystal (René N5, CMSX-4) and polycrystalline (Haynes 282) superalloys. Samples were held with mean tensile loads of 40–50 MPa and cycled in tension (R = 0.1) at 20.1 kHz. A micro-torch heated samples between 550°C and 1000°C for high-temperature studies. X-ray radiographs taken every thousand cycles over tens of millions of cycles showed micron-by-micron crack growth and oxides expelled from the crack opening. In single-crystal superalloys, cracks propagated along {111} planes at room temperature, with non-crystallographic crack propagation becoming more prevalent as temperatures increased. Diffraction contrast revealed dislocations accumulating 100–200 μm ahead of the crack front. In polycrystalline specimens, intergranular fatigue-crack growth was prevalent at lower temperatures, while transgranular crack growth occurred at higher temperatures.

A77

Structural Response of a Ferroelectric/Dielectric Multilayer to Applied Electric Fields

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X-ray scattering has the potential to resolve the structural piezoelectric distortion of individual components of a multilayer composed of repeating ferroelectric and dielectric layers. Combining the x-ray microdiffraction and time-resolved techniques, we resolved the strain developed by the multilayer in response to triangle-wave of electric fields. Theoretical modeling of the multilayer diffraction pattern provides a quantitative estimate of the distortions of the ferroelectric and dielectric components. Our model fits the changes of wavevector and intensities of the superlattice reflections of the multilayer. A $BaTiO_3/CaTiO_3$ multilayer consisting of 80 repetitions of four $BaTiO_3$ atomic layers and two $CaTiO_3$ atomic layers produces an average strain of 0.6 % under 1.25 MV/cm with a piezoelectric coefficient d_{33} value of about 42 pm/V. Both $BaTiO_3$ and $CaTiO_3$ components have linear piezoelectricity, which is the signature of ferroelectric materials. Sharing equally the average strain of the multilayer, both $BaTiO_3$ and $CaTiO_3$ components have ferroelectricity at meta-stable states under electric fields. This approach uniquely allows the electromechanical response of dielectric materials in confined geometries to be determined quantitatively.

A78

Three-dimensional Reconstruction Issues of Fresnel Coherent Diffractive Imaging

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There has been enormous progress in phase retrieval from coherent diffraction patterns. Up until now, most of the work has been focused on plane-wave approach. This approach, however, causes several problems such as uniqueness and stagnation problems in the algorithm, parasitic scattering, and beam-stop problems in the experiment. To solve these problems, G.J William et al have introduced the FCDI (Fresnel coherent diffractive imaging) method, which utilizes a curve-wave approach. While the curve-wave approach can help solve the phase retrieval problem, underlying phenomena are not intuitive to understand compared with plane-wave approach. One of them is to reconstruct a three-dimensional (3D) object from FCDI data. The difficulty of 3D reconstruction in FCDI is that diffraction patterns are so sensitive to sample motion such as translation and rotation.

In this work, we showed how to recover the incident wave field, how to use holographic data, and how to compensate for sample motion in 3D reconstruction. At first, we introduced the Quiney method to reconstruct the incident wave. Furthermore, we reconstructed zone plate image in which focused defects are well shown. Additionally, we obtained 3D information from two-dimensional holographic data with some limitations. We will discuss these limitations as well as possible applications in 3D reconstruction. Finally, assuming sample trajectory during rotation is exactly known, we tried to reconstruct 3D object and showed simulated results with some future works. We believe that the curve-wave approach will be more competitive than the plane-wave approach if we can reconstruct a 3D object successfully in the near future.

A79

Double-crystal Setup for X-ray Reflectivity Measurements on Liquid Surfaces

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Poster displayed.

A80

High-energy Diffraction Microscopy

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High-energy synchrotron radiation has been demonstrated to be a very powerful tool for *in situ* structural characterization of polycrystalline bulk materials during thermo-mechanical processing. Grain- and subgrain-specific information is obtained by techniques referred to as high-energy diffraction microscopy (HEDM). The ability to probe *in situ* deep below surfaces is a unique capability that could produce results that would rival other watershed materials capabilities like electron microscopy. In the "near-field" geometry, an area detector with high spatial resolution is placed closely behind the sample and the grain orientations and grain boundary topology are measured. In the "far-field" regime, a large area detector is placed at intermediate distance behind the sample such that strain sensitivity is greatly improved but still several complete diffraction rings are detected simultaneously. The orientation, center of mass position, volume, and complete strain tensor of individually scattering domains are measured. In the "high reciprocal space mapping" configuration, an area detector is placed far behind the sample achieving sufficient reciprocal space resolution to distinguish diffraction signatures from specific subgrain dislocation structures. The status of the HEDM instrumentation at the APS 1-ID beamline is presented and illustrated by selected case studies.

A81

Why Aren't Graphite and Graphene Strongly Correlated Electron Systems?

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We have used inelastic x-ray scattering coupled with state-of-the-art inversion algorithms to image background electronic screening processes in both graphite and graphene. By "background" we mean those processes that do *not* involve scattering among low-energy states near the Fermi surface pockets, but rather those that arise from deeper bands. Screening from these bands form the "vacuum" in which low energy theories of graphite and graphene must reside.

Using a rare, single crystal of graphite, we completely parametrized the dynamic structure factor along six directions in the Basal plane, from [100] to [110], and reconstructed the density-density Green's function, $\chi(x,t)$, for this system. Further, by making the quite reasonable assumption that the coupling between sheets is weak, we are able to convert our data and reconstruct $\chi(x,t)$ for an isolated graphene sheet.

We find, among other things, that the background dielectric constant in both graphite and even suspended graphene is ~14. This is much larger than the ~2 usually supposed, for example, in theories used to describe interactions among the Dirac fermions in isolated graphene. Our results explain, among other things, the absence a divergence in the Fermi velocity near the Dirac points in graphene, which has been expected but was not observed in either photoemission studies or in scanning electron transistor measurements of the compressibility.

A82

Withdrawn.

A83

Electrochemical Deposition on Carbon Surface for Multifunctionalization

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One of the biggest challenges for carbon-based materials applications is to tailor their physicochemical properties and assemble them into suitable architectures in a cost-effective and reproducible fashion. The coating of nickel (Ni) on individual carbon microfibers and assembly of carbon nanofiber sheets is carried out by electrodeposition. Nucleation, growth, and grain stability of nickel deposits on a single microfiber were probed simultaneously by measuring the fluorescence and diffraction from exposure of the fiber to a 6-μm diameter x-ray beam at the Advanced Photon

Source using beamline 2-ID-D. By using a specially designed electrochemical cell, synchrotron microbeam techniques provide *in situ* structural and compositional characterization capabilities. The preliminary results reveal that the nucleation and grain growth of Ni on carbon surfaces strongly depend on applied over potentials: instantaneous nucleation and growth process dominate at lower over potential (-300 mV) and the nucleation and growth become more progressive at higher over potential (-950 mV). With the understanding of the nucleation and growth mechanisms of metals on the carbon surfaces, the mechanical, electrical, thermal, and catalytic behaviors of carbon fibers can be further improved. These improvements can lead to large-scale engineering applications of carbon-based materials in lightning strike prevention, electromagnetic shielding, vibration/acoustic noise reduction, reinforced composites, catalyst substrates, supercapacitors, and bio/chemical sensors.

A84

Synchrotron Powder Diffraction Simplified: An Advanced Photon Source Mail-in Program for the 11-BM High-resolution Diffractometer

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Synchrotrons have revolutionized powder diffraction. They make possible the rapid collection of data with tremendous resolution and superb signal to noise. The high penetration and data sensitivity over a wide Q range possible at high energy light sources like the APS even allows synchrotrons to make inroads into territory that previously demanded neutron scattering techniques. Despite all these advances, still relatively few researchers travel to synchrotrons to conduct powder diffraction experiments.

To help address this, the 11-BM synchrotron powder diffractometer at the Advanced Photon Source now offers a convenient mail-in program for routine structural analyses with truly world-class data. This instrument offers resolution unmatched in the U.S. ($\Delta Q/Q \sim 2 \times 10^{-4}$). With both vertical and horizontal focusing and a detection system consisting of twelve crystal analyzers, the diffractometer can collect a superb pattern suitable for Rietveld analysis in one hour or less. Users of the 11-BM rapid access program typically receive their high-resolution data within 20 days of sample receipt.

This poster will describe the current instrument and its mail-in program, and discusses how we hope to improve the capabilities of 11-BM and widen its access to a diverse user community.

More information about the diffractometer and instructions on submitting your samples to 11-BM can be found at http://11bm.xor.aps.anl.gov.

A85

Quantitative Determination of Precipitate Size and Volume Fraction in a Nickel-base Superalloy via USAXS, Resistivity, and Microscopy

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Waspaloy is a nickel base superalloy used in gas turbine engine components. This material is often subjected to long-term exposure to high temperatures, which can affect the size and distribution of the reinforcing γ ' precipitates. Waspaloy specimens were aged at 725, 800, and 875°C for times ranging from 0.5 to 263.5 hours. Ultra-small-angle x-ray scattering (USAXS) was utilized to quantify the γ ' precipitate microstructure [1]. Modeling of the USAXS spectra revealed an eventual bimodal distribution of precipitates where the primary population showed $t^{1/3}$ coarsening behavior. The microstructural information was used to generate a figure of merit of electron scattering intended to correlate the electrical properties to the precipitate microstructure. The figure of merit, FOM, consists of two terms: one related to the size and arrangement of the gamma prime precipitates and the second related to the changes in the matrix composition. The FOM shows a non-ideal but direct correlation with the electrical resistivity. Since both electrical and mechanical properties in precipitation hardened materials are dependent on the precipitate microstructure [2], it is believed that the proposed figure of merit may be used to correlate the mechanical and electrical properties. Such correlations have implications towards lifetime prediction in gas turbine engine components.

[1] J. Ilavsky, P.R. Jemian, A. J. Allen, F. Zhang, L.E. Levine, and G. G. Long, *J. App. Cryst.*, (2009).

[2] R. L. Whelchel, V. S. K. G. Kelekanjeri, and R.A. Gerhardt, *J. International Heat Treatment and Surface Engineering I*, **3**, in press (2009).

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A86

Modeling the Elastic and Creep Properties of Collagen Fibril

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Bone has very complex hierarchical structure, which provides it with excellent mechanical properties such as high strength and high toughness at a low density. A complete understanding of relationships between the hierarchical structure and properties of bone relies on unraveling the relationship at the collagen fibril level, which is the level of the fundamental building block of bone. In this presentation, we focus on the collagen fibril level of the bone and emphasize the relationship between the structure and mechanical properties of a collagen fibril. Essentially, the collagen fibril can be viewed as a nanocomposite that is composed of the weak, viscoelastic collagen matrix and the hard, elastic mineral reinforcement. We create two-phase two-dimensional and three-dimensional unit cell models to represent the structure of the collagen fibril. By using finite element method to simulate the response of this model, we discuss the dependence of the mechanical properties of the collagen fibril on the parameters defining the structure, the properties of and the interaction between two phases. We compare our modeling results with the synchrotron x-ray diffraction data obtained from the Advanced Photon Source at ANL, which provide the deformation information of both the mineral plates and the collagen matrix. The computational and experimental results match well—quantitatively for elastic properties and qualitatively for creep properties—validating our simulation approach.

A87

Fe_3O_4@TiO_2 Nanoparticles Increase Cellular Uptake and Nuclear Localization of Doxorubicin in an Ovarian Cancer Model

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Our laboratory explores the use of TiO_2 as a biocompatible building material for nanoconjugates with applications in bio-nanotechnology. TiO_2 is particularly useful due to its nanocrystalline and semiconductor properties. Below a diameter of 20 nm, TiO_2 molecules form "corner defects" at the nanoparticles surface, which allow for covalent binding to a number of bidentate-substituted ligands. At this size as well, exposure to electromagnetic radiation in excess of the band gap (3.2eV) leads to a charge separation that has been shown to be able to traverse across covalently bound molecules. Addition of a Fe_3O_4 core has conferred magnetic resonance activity for additional tracking without compromising aforementioned properties of TiO_2. This work has focused on covalently bound doxorubicin, a widely used chemotherapeutic agent.

Conventional methods of detection do not allow for direct viewing of TiO_2 nanoparticles. By using x-ray fluorescence microscopy (XFM) at the APS 2-ID-E beamline, it is possible to generate elemental maps highlighting spatial distribution of TiO_2 within a cell. Simultaneous analyses at sequential time points of Ti by XFM and doxorubicin by confocal fluorescent microscopy have shown nuclear localization of both doxorubicin and Ti in both drug-sensitive and normally drug-resistant ovarian cancer cells.

A88

Atomic-scale Structure of Biogenic Materials by Total X-ray Diffraction: A Study of Bacterial and Fungal MnO

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Biogenic materials are produced by microorganisms and are typically found in a nanophase state. As such, they are difficult to characterize structurally. In this report, we demonstrate how high-energy x-ray diffraction and atomic pair distribution function analysis can be used to determine the atomic-scale structures of MnO_x produced by bacteria and fungi. These structures are well defined, periodic, and species specific, built of MnO_6 octahedra forming birnessite-type layers and todorokite-type tunnels, respectively. The inherent structural diversity of biogenic material may offer opportunities for practical applications.

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A89

S-XANES Analysis of Sulfur Forms in Argonne Premium Coals

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Sulfur x-ray near-edge absorption spectroscopy (S-XANES) has emerged as a powerful tool for speciating sulfur forms in complex carbonaceous solids such as kerogen and coal. It is also used to collect information about chemical and thermal sulfur transformations in these materials. This technique, used in conjunction with other techniques such as x-ray photoelectron spectroscopy and ^{13}C-NMR can be used to gain valuable information about sulfur transformations without altering the samples in the process. S-XANES has been performed on Argonne Premium Coal Samples, which are widely used as standards by various coal chemistry researchers. The Premium Coal Sample Program is intended to provide the basic coal research community with the highest quality samples of a limited number of coals (eight) for basic research. Measurements of these standards have been re-performed at the Advanced Photon Source beamline 9-BM, which is optimized for low energy x-ray absorption fine structure (XAFS) (below 4 keV) and is unique to the APS facility. The data taken at the APS has been compared to the data taken in 1991 at the Stanford Synchrotron Radiation Light Source by M. Gorbaty et al [1]. New insight has been gained into the ability to directly detect the pyritic sulfur content of these samples with XAFS, which has been problematic in the past. Sulfur characterization and analysis methods using spectra from model compounds and selected sulfur data for kerogens and coals taken from the literature is discussed.

[1] G. N. George, M. L. Gorbaty, S. R. Kelemen, and M. Sansone, *Energy and Fuels*, **5**, 93, (1991).

A90

Characterization of Atomic Layer Deposition Platinum for the Model $Pt/SrTiO_3$ Photocatalyst

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Poster displayed.

A91

Implementation of Pressure Safety for Vacuum Systems at Department of Energy Accelerator Laboratories

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Poster displayed.

A92

BioCARS: A Facility for Macromolecular Crystallography at the Advanced Photon Source

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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 as National Synchrotron Resources available to the scientific community. BioCARS (Sector 14) consists of two beamlines: an insertion device beamline (14-ID-B) with dual, inline undulators (U23 and U27) and a bending magnet beamline (14-BM-C). 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, wide-angle scattering, fiber diffraction, Laue crystallography, and 100-ps 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.

A93

Field-free Molecular Alignment for Studies Using X-ray Pulses from a Synchrotron Radiation Source

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A short, intense laser pulse may be employed to create a spatially aligned molecular sample that persists after the laser pulse is over. We theoretically investigate whether this impulsive molecular alignment technique may be exploited for experiments using x-ray pulses from a third-generation synchrotron radiation facility. Using a linear rigid rotor model, the alignment dynamics of model molecular systems with systematically increasing size is calculated utilizing both a quantum density matrix formalism and a classical ensemble method. For each system, the alignment dynamics obtained for a 95-ps laser is compared to that obtained for a 10-ps laser pulse. The average degree of alignment after the laser pulse, as calculated quantum mechanically, increases with the size of the molecule. This effect is quantitatively reproduced by the classical calculations. The average degree of impulsive alignment is high enough to induce a pronounced linear dichroism in resonant x-ray absorption using the intense 100-ps x-ray pulses currently available. However, for structural studies based on elastic x-ray scattering, bright x-ray pulses with a duration of 1 ps or shorter will be required in order to make full use of impulsive molecular alignment.

A94

A Simple Cross-correlation Technique between Infrared and Hard X-ray Pulses

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We report a gas phase technique to establish the spatial and temporal overlap of ultrafast infrared laser and hard x-ray pulses. We use tunnel ionization of a closed-shell atom in the strong field at the focus of an infrared laser beam to open a distinct x-ray absorption resonance channel with a clear fluorescence signature. The technique has an intrinsic response of a few femtoseconds and is non-destructive to the two beams. It provides a step-function-like cross-correlation result. The details of the transient provide a diagnostic of the temporal overlap of the two pulses. The

technique will be very well suited for characterizations of the planned APS short-pulse source and has potential applications as a fast streaking device at x-ray free-electron laser sources. We present experimental data from measurements at the Advanced Photon Source for 24-bunch and hybrid fill operations and compare with results from streak camera measurements and results from measurements with fast metal-semiconductor-metal photodetectors.

A95

Single-shot Ultrafast Phase-contrast X-ray Imaging of High-pressure Diesel Fuel Sprays

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By taking advantage of high-intensity and high-brilliance x-ray beams available at the Advanced Photon Source, ultrafast (150 ps) propagation-based phase-enhanced imaging was developed to visualize high-pressure, high-speed diesel spray breakup process in the optically dense near-nozzle region. The sub-ns temporal and μm spatial resolution allow us to capture the morphology of the high-speed fuel sprays traveling at >500 m/s with a negligible motion blur. Both quality and quantitative information about the spray breakup can be readily obtained. In the experiment, two types of single-hole nozzles have been used, a hydroground nozzle with rounded orifice inlet and a non-ground nozzle with a sharp inlet. The fuel sprays are extremely dynamical from both injectors. In the quasi-steady state of the injection, the jet from the hydroground nozzle (to a stagnant gas of 0.1 MPa) remains as a column showing two distinctive instability regions showing surface instability waves and ligament-dominant breakup. The surface instability waves appear to be aerodynamics independent, while the downstream breakup is due to aerodynamic interaction between the jet and the ambient gas. Helium, nitrogen, and sulfur hexafluoride at 0.1-MPa pressure are used as the ambient gases to test the aerodynamic interaction. In comparison, fuel injected from a non-ground nozzle breaks up within several nozzle diameters from the nozzle exit. We speculate that the internal cavitation causes the jet to breakup. For hydroground nozzle, the surface waves exhibit as a new instability phenomenon at a condition with both high Weber and Reynolds numbers. These wave characteristics are extremely sensitive to the injection pressure, hence, the jet speed. The downstream aerodynamic breakup can be used to validate the theory proposed previously by Reitz.

A96

Dynamics of Silica Nanoparticles near the Liquid/Vapor Interface of Dibutyl Phthalate

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We present preliminary results from x-ray photon correlation spectroscopy (XPCS) studies of the dynamics at the free surface of the suspension of the 30 nm silica particles in the model organic glass former, dibutyl phthalate (DBP). The aim of the experiment was to test if enhancement in the molecular mobility near the liquid/vapor interface suggested by the XPCS capillary wave measurements of the pure DBP could be confirmed by the monitoring of the motion of the nanotracers in the vicinity of this interface. Measurements were carried out at fixed grazing incidence angle (2.5 mrad) and the diffuse scattering was collected in a time-resolved manner using a direct detection CCD detector covering a q-range from 2×10^{-3} Å^{-1} to 3×10^{-2} Å^{-1} over a window of 0.1–1000 s. Data sets were collected over a wide temperature range starting from the 295 K down to the bulk glass transition temperature of the solvent (T_g=180 K). Obtained data clearly indicates that the surface dynamics of the colloidal suspension depend strongly on both concentration and sample temperature. For the moderate concentration solution (3% by volume), the characteristic time for the particle motion exceeds 1 s below 200 K, which is in good agreement with the capillary waves data. In case of the highly concentrated suspension (15%), the dynamics freezes already at 245 K. Aggregation of the nanoparticles near the free surface, resulting in a dramatic decrease of the particles mobility, is a probable scenario explaining the concentrated solution data. The intermediate structure factor obeys a compressed exponential line shape.

A97

Beating the Missing Data Problem Via Ptychography

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Magnetic thin layers, multilayer films, and nanostructures show intriguing magnetic properties, for example interlayer exchange coupling and giant magnetoresistance. Layered magnetic thin films could be implemented into read-head technology, leading to smaller devices and increased storage density. Coherent diffractive x-ray imaging (CDXI) is a promising new experimental technique to study the underlying physics of these structures.

In CDXI, image reconstruction procedures place stringent requirements on diffraction data quality. In real-world measurements, experimental artifacts such as positional drift and errors, missing data (e.g., due to a beam stop), and shot noise are typically present. Here we demonstrate that data obtained by ptychographic methods are sufficiently overdetermined to give high-quality reconstructions even when some of these experimental artifacts are present. An unknown complex illumination function can also be successfully recovered from these measurements. Convergence as a function of illumination function overlap, signal-to-noise ratio, and amount of missing data are studied.

We discuss newly developed algorithms in context of our recent experiments on magnetic nanoscale domains in GdFe multilayer films.

A98

Microtomography Application in Paleontology

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Poster displayed.

A99

Wetting Autophobicity of Poly(4-bromostyrene) Thin Films on Poly(4-vinylpyridine) Substrates

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The thermodynamic stability and wetting behavior have been investigated in a homopolymer thin film system consisting of a poly(4-bromostyrene) (PBrS) film prepared on top of a poly(4-vinylpyridine) (P4VP) substrate. It is demonstrated using atomic force microscopy (AFM) and x-ray standing waves generated via total external reflection (TER-XSW) from an x-ray mirror that the PBrS film exhibits autophobic wetting, where due to the presence of an inflection point on the Helmholtz free energy versus thickness curve, the film is stable at small thicknesses but unstable at large thicknesses. We used AFM to capture the final dewetting morphologies of the PBrS film as a function of thickness, while the highly sensitive TER-XSW technique was used to monitor the stability of both the PBrS/air and buried PBrS/P4VP interfaces via a spatially dependent fluorescence signal from bromine markers within the PBrS layer. In the second part of the experiment, an additional poly(styrene) (PS) layer was placed on top of the PBrS layer. From this study, we were able to learn about how PS modifies the free energy curve for PBrS from the first study and how the interaction between the highly immiscible PS and P4VP gets modified by sandwiching a PBrS layer in between.

A100

Structure-dynamics-efficiency Relation in Organic Solar Cell Studied by Grazing Incidence X-ray Scattering and Ultrafast Transient Spectroscopy

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Organic photovoltaic (OPV) devices based on bulk heterojunction materials have been extensively investigated in recent years due to their potential advantages such as low cost and roll-to-roll manufacturability over the inorganic counterpart. However, applications of these OPV devices have been hindered by their low efficiency at ~5–6%, much lower than the conventional silicon-based solar cell. In order to optimize the efficiency, the structure-dynamics-efficiency correlations in the OPV materials must be fully understood. Recently, we reported a new type of low-band-gap polymer based on thienothiophene-benzodithiophene, which exhibited a record 5.6% efficiency. Using the grazing incidence x-ray scattering, we have obtained structures of polymer and PCBM morphology and packing in length scales from subangstrom to 100 nm. The structural information combined with the dynamics of exciton generation, diffusion, and splitting provides new insight into rational design of OPV materials for higher efficiency solar cells. The structure-dynamics efficiency will be discussed in detail.

A101

The Nanopositioning Working Group

Boris Deriy, Pat Den Hartog, Soon Hong Lee, Frank Lenkszus, Curt Preissner, John Quintana, Deming Shu, and Joseph Xu
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The Nanopositioning Working Group (NPWG) has recently been formed within the APS Engineering Support Division. Chaired by John Quintana, this multidisciplinary group's role is to keep the APS at the forefront of science-enabling nanopositioning related instrumentation. The NPWG's mission includes acting as a contact point for APS and external collaboration, identifying the current and future nanopositioning requirements of x-ray science at the APS, informing instrumentation scientists about our capabilities, and bringing multidisciplinary resources to bear on nanopositioning design projects. This poster serves to inform the APS community about the NPWG's engineering capabilities.

Work supported by U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-AC02-06CH11357.

C1

Nanoscale Characterization of Epitaxial $BiFeO_3$ Nanostructures Defined by FIB Lithography

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Multiferroic $BiFeO_3$ (BFO) is a room temperature ferroelectric antiferromagnet that is widely studied for its robust ferroelectric state (high polarization and Curie temperature). In this respect, recent work has been directed at the growth, fabrication, and characterization of BFO thin-film nanostructures. In the work presented here, BFO nano-capacitors were fabricated using focused ion beam (FIB) lithography on epitaxial films grown by magnetron sputter deposition on $SrRuO_3$-coated (001) $SrTiO_3$ substrates. Preliminary piezoresponse force microscopy (PFM) studies of square (500×500 nm^2) and round (502 nm in diameter) shaped capacitors revealed that square-shaped nanostructures exhibit a single variant ferroelectric domain configuration aligned along the [-11-1] direction whereas round-shaped nanostructures exhibit seven variants of domain configuration along the [-11-1], [1-1-1], [11-1], [111], [-111], [1-11], and [-1-11] directions. Moreover, local d33 piezoelectric coefficient measurements showed hysteresis loops with a strong displacement in the voltage axis (strong imprint) for the square-shaped nanostructures, while the round-shaped ones exhibited more symmetric loops. The observed shape dependence may be due to differences in electrostatic boundary conditions, symmetry in strain field, or spatial distribution of FIB-induced damage. In the present

study, nanoscale synchrotron x-ray measurements, cross-section transmission electron microscopy (TEM), and PFM were used to characterize the role of mechanical strain and FIB-induced damage on the ferroelectric domain behavior of laterally confined nanostructures. Initial results indicate that while substantial ion-induced damage (amorphization) was present in as-fabricated nanostructures, oxygen annealing can lead to significant recovery (recrystallization). X-ray diffraction measurements revealed that the FIB-defined nanostructures were almost entirely non-diffracting prior to annealing, which is consistent with TEM imaging, which showed an amorphous layer that is nearly 75% of the total BFO thickness. In contrast, synchrotron measurements on the annealed capacitors showed diffracted intensity from features as small as 200 nm. Furthermore, comparing Fe x-ray fluorescence maps and scanning electron microscope images with BFO diffracted intensity maps revealed a significant lateral depth of FIB-induced BFO amorphization, which is strongly dependent on the ion beam path during fabrication.

Use of the Center for Nanoscale Materials and work in the Materials Science Division were supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

E1

Nanoscale Structural Characterization of a Magnetic Tunnel Junction

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A magnetic tunnel junction (MTJ) is a sandwich structure composed of two ferromagnetic layers separated by a thin insulating layer such as MgO. This trilayer structure shows the tunneling magnetoresistance effect, which is used in modern magnetic field sensors in hard disk drives. The three-dimensional morphology and chemistry of the individual layers have a strong effect on the performance of the MTJ. In this work, a model CoFeB/MgO/CoFeB MTJ is investigated with high spatial resolution in transmission electron microscopy by scanning transmission electron microscopy (STEM) tomography, electron holography, and high-resolution electron microscopy (HREM). HREM images show that the MgO barrier and CoFeB layers have good crystallinity after annealing. From electron holography, the phase shift of the electron beam is used to depict the barrier layer. The measured barrier width in the annealed sample is measured as 1.7 nm from the full width at half maximum, increased from the nominal 1.2-nm thickness as deposited, and the barrier profile is very symmetric. Finally, STEM tomography clearly shows the layered structure and grains in CoFe cap layer. The interfaces on either side of the tunnel barrier are clearly rough which will play a significant role in the resulting magnetotransport properties this model MTJ.

E2

Effect of Surface Morphology on Magnetic Properties in Epitaxial $CoFe_2O_4$ Thin Films Deposited by MOCVD

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We have successfully grown epitaxial $CoFe_2O_4$ (CFO) thin films on $SrTiO_3$ single-crystal substrates using metal organic chemical vapor deposition (MOCVD). In order to understand the surface morphology and its correlation with magnetic properties, the CFO thin films were deposited at a range of deposition temperatures, T_{dep}. As T_{dep} is decreased, there is a change in surface morphology and a decrease in surface roughness of two orders of magnitude from ~22 nm to ~0.2 nm measured by atomic force microscope. Cross-sectional transmission electron microscopy (TEM) was performed at the Electron Microscopy Center. Electron diffraction patterns and high-resolution images confirm cube-on-cube epitaxial growth. Thin film microstructures at different T_{dep} are also revealed in TEM images. The microstructural changes with T_{dep} lead to a change of magnetic properties, measured by superconducting quantum interference device: compressive strain leads to an in-plane easy axis along the [100] direction in the smooth films, whereas for the rough films, the columnar grain structure results in barriers to the magnetization reversal and thus the presence of an out-of-plane easy axis. In the future, we will study magnetic domain structures and transport properties in CFO thin films and patterned structures using *in situ* scanning transmission electron microscopy techniques.

E3

Optimized Sample Preparation for Atom Probe and Electron Tomography with Dual-beam FIB

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Poster displayed.

E4

Growth Mechanism of In_2O_3/ZrO_2 Superlattice Films: Quantitative Roughness Measurements of Buried Interfaces

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Poster displayed.

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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 source of the Western Hemisphere's brightest x-ray beams for research. 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 to 7-GeV booster/injector synchrotron, 7-GeV storage ring) to produce extremely bright, tunable x-ray beams that researchers use to carry out frontier experimentation in areas of science including materials science, biology, chemistry, engineering, environmental science, geosciences, planetary 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 2.3 cm, 2.7 cm, 3.00 cm, 3.30 cm, 3.50 cm, and 5.50 cm; and a 12.8-cm-period circularly polarized undulator.

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 approximately 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 Photon Sciences. As of March 2008, the APS staff numbered around 450. In fiscal year 2008, more than 3,600 users visited the APS an aggregate total of more than 10,000 times to carry out more than 2,900 experiments and published almost 1,000 papers (as of 4.9.09).

Electron Microscopy Center

Web site: http://www.emc.anl.gov/
User contact: 630-252-4987

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.

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.

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 is used for developmental work. A synopsis of each of these areas is provided below.

IVEM-Tandem Facility

This unique facility consists of an intermediate voltage TEM (IVEM), an 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 specimen holders allow a variety of *in situ* experiments to be performed, ranging from low temperature studies using liquid He to studies at high temperatures.

Analytical Electron Microscopy (AEM) Facility

The Analytical Electron Microscopy facility consists of a variety of electron microscopes that are essential for forefront materials research programs. Major capabilities include a field-emission TEM/STEM that is optimized for medium-high resolution imaging and analytical work and a cross-beam focussed ion beam (FIB) instrument that simultaneously scans electron and ion beams. Core capabilities include an analytical TEM and high-brightness scanning electron microscopes. These resources provide microstructural analytical capabilities for a wide range of users.

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. The EMC staff continues to develop 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, and analysis together with input and output devices. Offline software is also available for spectral analysis.

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. The reviewers evaluate the scientific and technical merit of the proposal, whether the proposed approach is appropriate, the probability of success, and the appropriateness of EMC resources requested.

The type of access granted to a user is based on the nature of the proposed work, the user's 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 is designed 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 after one year, at which time they may be renewed. 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; ambient T	~0.24 nm point ~0.1 nm lattice Cs obj. ≈ 1.2 mm Cc obj. ≈ 1.2 mm probe ≈ 0.2–1 nm
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 resolutions: 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, 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, STEM, EDXS, specimen chamber pressures 10^{-5} 2660 Pa (air & water vapor standard)	5-axis motorized stage Maximum sample size: 100 x 100 mm Heating stages (≤ 1770 K) Peltier-cooled stage (248-328 K)	SEI resolutions at 30 kV: ≤ 3 nm at 1330 Pa ≤ 10 nm at 2660 Pa

DIRECTORY OF EXHIBITORS

Special thanks to TecRep Corporation, sponsor for the Opening Reception, Sunday evening, May 3, 2009.

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ACCEL IACCEL Instruments GmbH Friedrich- Friedrich-Ebert-Straße 1 51429 515514429 Bergisch Gladbach Germany Phone number: 011.49.22.04.84.25.12 Fax number: 011.49.22.04.84.25.99 URL: www.accel.de	ACCEL Instruments GmbH is a worldwide engineering and manufacturing company specializing in custom-designed equipment for research and industry in the fields of particle accelerators, synchrotron radiation instrumentation, and medical applications. We design, manufacture, assemble, install, and commission beamlines as turnkey systems. We also provide components such as undulators, wigglers, double-crystal and double-multilayer monochromators, mirror systems, slit systems, and all sorts of beam monitors as well as all experimental stations especially for crystallography and x-ray microscopy.
Advanced Control Systems Corporation 35 Corporate Park Drive Pembroke, MA 02359 USA Phone number: 1.781.829.9228 Fax number: 1.781.829.9875 URL: www.ACSMotion.com	Advanced Control Systems Corporation is a designer and manufacturer of stepping motor drivers, servo motor amplifiers, integrated driver/controllers, and stand-alone programmable machine controllers for scientific and industrial applications. The ACS Step-Pak™ Modular Motion Control System provides economical, scalable, multi-axis motion control for physics research.
Advanced Design Consulting USA, Inc. 126 Ridge Rd. Lansing, NY 14882 USA Phone number: 1.607.533.3531 Fax number: 1.607.533.3618 URL: www.adc9001.com	Advanced Design Consulting USA, Inc. (ADC) is a leading global supplier of advanced technology products and systems to the synchrotron, neutron, research, and scientific community. The company provides devices, integrated systems, and a broad array of high-precision components and instruments to commercial, academic, and government agencies worldwide.
Advanced Research Systems, Inc. 7476 Industrial Parkway Macungie, PA 18062 USA Phone number: 1.610.967.2120 Fax number: 1.610.967.2395 URL: www.arscryo.com	ARS manufactures integrated pneumatic drive closed cycle and Helitran cryostats for materials characterization. The ARS cryocoolers have been redesigned for a temperature range of sub 1.7 to 300K or 3 to 800K. With the lowest vibrations at the sample and its slim line axial geometry, it is the cryocooler of choice for XRD manipulation. Beryllium domes allow for full angular beam access.
Aerotech Inc. 101 Zeta Drive Pittsburgh, PA 15238 USA Phone number: 1.412.963.7470 Fax number: 1.412.963.7459 URL: www.aerotech.com	Aerotech direct-drive linear and rotary air-bearing stages provide unsurpassed performance for precision tomography and crystallography applications. In addition, Aerotech manufactures drives and motion controllers that perfectly complement our stages. Aerotech's Ensemble stand-alone motion controller is compatible with the EPICS set of software tools and applications, making it ideal for use in synchrotron and general laboratory facilities.
Alan Burrill Technical Sales 8129 W. Lincoln Avenue West Allis, WI 53219 USA Phone number: 1.414.327.9055 Fax number: 1.414.327.8131 URL: www.aburrilltechsales.com	Alan Burrill Techical Sales is a manufacturer's representative firm that specializes in vaccum, thin film, and surface science related instruments, hardware, pumps, and systems.
ALIO Industries 11919 W. I-70 Frontage Road N. # 119 Wheat Ridge, CO 80033 USA Phone number: 1.303.339.7500 Fax number: 1.303.339.7501 URL: www.alioindustries.com	ALIO is an innovator in nanotechnology motion systems. ALIO's designs exceed current standards of precision product designs for automation technology. Holding two patents for the Parallel 6 Axis Hexapod and the Parallel 3 Axis Tripod, as well as several patent pendings for nano Z stages and planar air bearing systems, ALIO has set the pace for nano precision design and systems.
AMREL/AMERICAN RELIANCE, INC. 3445 Fletcher Ave. El Monte, CA 91731 USA Phone number: 1.626.443.6818 Fax number: 1.626.443.8600 URL: www.amrel.com	AMREL offers the widest selection of American designed and manufactured power supplies and electronic loads. Linear supplies are available up to 1200W and switch-mode models range from 1.2kW to 900kW. Additionally, with a GSA contract, Amrel power supplies are the lowest cost per watt for Argonne. AMREL's electronic loads can range to 100kW and higher.
Area Detector Systems Corp. 12550 Stowe Drive Poway, CA 92064 USA Phone number: 1.858.486.0444 Fax number: 1.858.486.0722 URL: www.adsc-xray.com	Area Detector Systems is a world leader in CCD array detectors used in macromolecular crystallography. We are pleased to announce the introduction of several pixal array detector products for both MX and non-MX applications. ADSC offers detectors using Atmel CCDs in several sizes: our 2 × 2 array detector, the Q210r (210 mm sq.); and our 3 × 3 array detector, the Q315r (315 mm sq.). ADSC is pleased to announce a 4 × 4 detector, the Q420r, which at 420 mm sq. will be the largest CCD detector commercially available. ADSC offers a high sensitivity 2 × 2 array detector using Kodak Blue+ CCDs with a large 270 mm square area.

attocube systems AG Königinstr.11a RGB 80539 Munich Germany Phone number: 011.49.89.2877809.0 Fax number: 011.49.89.2877809.19 URL: www.attocube.com	attocube systems provides ultra-high precision spatial positioning systems and scanning probe microscopes such as AFM, CFM, SNOM, and STM. These systems are particularly suitable for extreme environmental conditions such as cryogenic temperatures, high magnetic fields and ultra-high vacuum environments and have enabled pioneering investigations and developments in science and industry.
Blake Industries, Inc. 660 Jerusalem Road Scotch Plains, NJ 07076 USA Phone number: 1.908.233.7240 Fax number: 1.908.233.1354	Blake Industries is a leader in supplying high-precision x-ray and neutron instrumentation to research labs, university, and synchrotron beamlines worldwide. We are the exclusive distributors for Huber instruments in the U.S., Mexico, and Canada with multiple installations at all the North American synchrotrons and have been supplying x-ray users for over 40 years.
Bruker AXS Inc. 5465 East Cheryl Parkway Madison, WI 53711 USA Phone number: 1.608.276.3000 Fax number: 1.608.276.3006 URL: www.bruker-axs.com	Bruker AXS provides advanced x-ray solutions for chemistry, life, and material sciences. A wide range of single-module CCD systems is available in the PLATINUM series of detectors, which feature the newest 4K CCD chip with lower noise, faster readout, and highest sensitivity. Nanotechnology research systems include a range of powder diffraction and single-crystal solutions.
Canberra 800 Research Parkway Meriden, CT 06450 USA Phone number: 1.203.639.2148 Fax number: 1.203.235.1347 URL: www.canberra.com	CANBERRA, an AREVA Company, is the worldwide leader in nuclear measurements. With over 40 years of experience in this field, we bring solutions to our customers, not just products or services. The key to our success lies in our ability to identify the right customers and understand their needs in order to bring them value-adding and innovative solutions. In addition, we strive to meet all of the commitments that we make to our customers. CANBERRA provides "Measurement Solutions for Safety and Security" to the following markets/applications: • Nuclear Security and Safeguards • Labs & Fuel Cycle • Nuclear Power Plants
Carl Zeiss Laser Optics Carl-Zeiss-Strasse 22 73447 Oberkochen Germany Phone number: 011.49.7364.20.8975 Fax number: 011.49.7364.20.2967 URL: www.zeiss.de/laseroptics	Carl Zeiss has a long-lasting expertise in the field of optical manufacturing and material processing for reflecting and refracting optical systems. For the synchrotron community, we focus our commitment on optical mirrors of the highest quality and a variety of geometries. Custom made Zeiss mirrors ranging from high precission flats to freeform surfaces with high complexity support flexibility and excellence at beamlines throughout the world.
CATIAD Mechanical Engineering 30 W. Fay Avenue Addison, IL 60101 USA Phone number: 1.630.543.6186 Fax number: 1.630.543.6258 URL: www.catiad.com	CATIAD is an engineering and consulting firm specializing in mechatronic design services. We offer design and full system integration solutions for a wide variety of applications. Our concentration is in precision automation, medical equipment, linear motor stages, direct drive rotary stages, machine tools, robotics, construction equipment, and wind energy.
CMA/Flodyne/Hydradyne 1000 Muirfield Dr. Hanover Park, IL 60133 USA Phone number: 1.630.563.3600 Fax number: 1.630.563.3850 URL: www.cmafh.com	Full service automation distributor offering electric motion, electric controls, pneumatic, and hydraulic product lines including complete aluminum framing and guarding for all applications.
CVI Melles Griot 200 Dorado Place SE Albuquerque, NM 87123 USA Phone number: 1.505.296.9541 Fax number: 1.505.298.9908 URL: www.cvimellesgriot.com	CVI Melles Griot is excited to be introducing 100s of new products this year! • Pick up our Volume 1, Issue 1 of "All Things Photonic"s our New 2009 Catalog • Learn about our new laser products • Learn about our new exciting filter products that will save you money, time and improve your system performance Visit us at cvimellesgriot.com to get the latest information on products and capabilities to save you money, time, and energy.
FMB Oxford Limited Unit 1 Ferry Mills Osney Mead Oxford OX2 0ES United Kingdom Phone number: 011.44 1865.320300 Fax number: 011.44 1865.320301 URL: www.fmb-oxford.com	FMB is recognized as a leading supplier of instrumentation to the scientific community. With over 18 years of experience in the global synchrotron industry, we have built a product range extending from storage ring vacuum systems to complete turnkey beamlines with an installed base exceeding $100M. Our core competencies are the project management, design, final assembly, test, installation, and commissioning of beamline systems and beamline components, specifically hard x-ray monochromators and mirror systems. We market a wide range of custom detectors and diagnostics to complement our beamline offerings.

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Mager Scientific, Inc. P. O. Box 160 Dexter, MI 48130 USA Phone number: 1.734.426.3885 Fax number: 1.734.426.3987 URL: www.magersci.com	Mager Scientific is the Midwest distributor for the Leica Microsystems' line of SEM and TEM sample preparation instrumentation. We are your comprehensive supplier for sputter coaters, high-vacuum coaters, cryo SEM stages, TEM sample prep, surface preparation, and high-pressure freezing technics. Please consider Mager Scientific for your SEM, TEM, and cryo sample preparation needs.
MDC Vacuum Products, LLC 23842 Cabot Blvd. Hayward, CA 94545-1661 USA Phone number: 1.510.265.3500 Fax number: 1.510.887.0626 URL: www.mdcvacuum.com	MDC Vacuum Products, LLC stocks thousands of off-the-shelf components for high and ultra-high vacuum applications. Our product line consists of flanges and fittings, valves, roughing hardware, vacuum measurement, viewports, electrical and fluid feedthroughs, motion and manipulation instruments, thin film electron-beam evaporation systems, and surface science chambers. MDC also offers customized components, chambers, and systems and also offers gas and chemical delivery systems services and components.
MICOS USA 15375 Barranca Parkway G-101 Irvine, CA 92168 USA Phone number: 1.949.480.0538 ext. 701 Fax number: 1.949.480.0538 URL: www.micosusa.com	MICOS supplies micro- and nano-positioning equipment with a focus on the reserach market. We offer a full range of precision positioners and control electronics as well as complete system integration services. Additionally, MICOS is known as the premier supplier for vacuum to 10-10 torr, clean room, and extreme temperature applications.
Midwest Vacuum, Inc. 201 E. Ogden Ave. Suite 15 Hinsdale, IL 60521 USA Phone number: 1.630.323.5399 Fax number: 1.630.323.2142 URL: www.midwestvacuum.com	Midwest Vacuum, Inc. is a leading supplier of vacuum to UHV components and systems. Our specialty is UHV chambers and all types of vacuum pumps and instrumentation such as RGA and vacuum gauges. We represent some of the leading companies in these fields.
Minarik Corporation 1261 Wiley Road Suite A Schaumburg, IL 60173 USA Phone number: 1.847.273.9050 Fax number: 1.847.273.9060 URL: www.minarik.com	Minarik is a leading solution provider of automation, instrumentation, and motion control products and systems for the research laboratories and the synchrotron user community. As a premier motion control provider, Minarik offers high-precision nanopositioning systems capable of operating in demanding environments such as UHV (10-10 torr), clean room, radiation, RF, and/or non-magnetic applications. Minarik represents some of the leading companies in the motion control and automation industry such as: Nanomotion (ceramic servo motors and systems), Danaher Motion (actuators, stages, gearboxes, stepper motors, and amplifiers), IBEX (linear and rotary stages and systems), Applied Motion Products (stepper motors and amplifiers), Animatics (integrated stepper and servomotors), ACS Motion (multi-axis motion controller), WAGO (distributed I/O), Panasonic (PLCs, safety and light curtains), IDEC (power supplies), CiCoil (flexible flat cable assemblies), CGI (vacuum rated gearboxes), and Item (structural aluminumpProfile and assemblies). Minarik is your one source for automation and motion control needs. For more information on Minarik call 1-888-Minarik (646-2745).
Newport Corporation 1791 Deere Avenue Irvine, CA 92606 USA Phone number: 1.800.222.6440 URL: www.newport.com	Newport is a premier global resource for customers that need to make, manage, and measure light. Its Spectra-Physics division is a global leader and innovator in a broad spectrum of lasers used in various bio-analytical applications. The Oriel product line is recognized as a leader for white light sources and spectroscopic instrumentation. We have unique, integrated expertise and knowledge in both laser and white light sources, in optics and optical mechanical positioning, and detection and instrumentation. This allows us to create innovative products and complete sub assembly light engine systems that integrate easily to deliver solutions that make, manage and measure light—all from one source.
Nor-Cal Products 1967 S. Oregon Street Yreka, CA 96097 USA Phone number: 1.530.842.4457 Fax number: 1.530.842.9130 URL: www.n-c.com	Since 1962 Nor-Cal Products has manufactured high- and ultra-high vacuum components for semiconductor, compound semiconductor, thin film, surface analysis, and various research applications. Nor-Cal has earned a reputation worldwide for quality components and is ISO 9000-2001 registered. Our custom vacuum chambers are unequalled in performance and appearance. In-house electropolishing and special materials are available. Entire systems can be supplied complete with sample transfer and manipulation devices, stand, pumps, gauges, and deposition monitors. Standard components include flanges, fittings, viewports, feedthroughs, and flexhose; isolation and pressure control valves; thermal products; molecular sieve, particulate, and cold traps; thin film components; and pressure gauges and manipulators.

nPoint, Inc. 1617 Sherman Ave. Madison, WI 53704 USA Phone number: 1.608.310.8770 Fax number: 1.608.310.8774 URL: www.npoint.com	nPoint, Inc. manufactures ultra-precision motion and control devices for nano-scale research and manufacturing. Our products include a series of nanopositioning systems and closed-loop AFM scanners. The nPoint products enable rapid, precise, and repeatable motion and are used in applications ranging from life science to semiconductor industry.
Oerlikon Leybold Vacuum 5700 Mellon Road Export, PA 15632 USA Phone number: 1.724.327.5700 Fax number: 1.724.325.3452 URL: www.oerlikon.com	Oerlikon Leybold Vacuum is a leader in providing vacuum pumps, accessories, and services to all industries requiring vacuum. These include turbomolecular pumps, cryo pumps, oil lubricated and oil-free forevacuum pumps, gauges, leak detector, valves, fittings, and a full line of various coating tools. Oerlikon Leybold Vacuum also provides Engineering and Applications support, plus services for all brands of vacuum pumps.
Omicron NanoTechnology USA, LLC 14850 Scenic Heights Road Suite 140 Eden Prairie, MN 55344 USA Phone number: 1.952.345.5240 Fax number: 1.952.294.8043 URL: www.omicron-instruments.com	
Pfeiffer Vacuum 24 Trafalgar Square Nashua, NH 03063 USA Phone number: 1.603.578.6500 Fax number: 1.603.578.6550 URL: www.pfeiffer-vacuum.com	Pfeiffer Vacuum is the world's largest producer of turbomolecular pumps and has over 100 years of vacuum experience. We are your single source for vacuum products and we offer a complete line of turbomolecular pumps, dry pumps, rotary vane pumps, roots pumps, mass spectrometers, residual gas analyzers, helium leak detectors, gauges, and vacuum hardware.
Phytron, Inc. 600 Blair Park Rd. Williston, VT 05495 USA Phone number: 1.802.872.1600 Fax number: 1.802.872.0311 URL: www.phytron.com	Established in 1947, Phytron is a leading international manufacturer of stepper motors and positioning systems for use in extreme environments such as vacuum, radiation, cryogenic conditions, and space. Our products are widely used in synchrotron labs. We also make special stepper drivers to adapt to the standard controllers and cabling used at most synchrotron labs.
PI (Physik Instrumente) LP 16 Albert Street Auburn, MA 01501 USA Phone number: 1.508.832.3456 Fax number: 1.508.832.0506 URL: www.pi-usa.us	PI (Physik Instrumente) LP is an ISO9001 certified global leader in piezoelectric motion control and nanopositioning solutions. Our products include the following: vacuum-compatible, non-magnetic, auto-locking ceramic actuators and motors; linear motors and multi-axis alignment systems; parallel-kinematic systems; ultrafast XY piezo scanners/steering-mirrors for image stabilization; sensor dithering and bio-imaging; smart actuators; ultrasonic linear motors; NEXLINE® award-winning high-force piezoceramic linear motors; piezo ceramic assemblies; vacuum-compatible PICMA® ceramic encapsulated multilayer piezoelectric actuators; piezo stages; photonics alignment systems; high-power piezo amps and drivers with energy recovery; advanced hexapod 6D optics alignment systems; capacitive sensors; advanced digital piezo controllers (with improved settling, speed, and linearity); and tools for nanometrology, nanotechnology, ultra-high-resolution microscopy, interferometry, test and measurement, and optics and mask alignment. New products include NEXACT® N-380 ceramic linear actuator/motor and the M-810 Hexapod miniature 6-axis optics alignment/collimation system.
Princeton Instruments 3660 Quakerbridge Road Trenton, NJ 08619 USA Phone number: 1.609.587.9797 Fax number: 1.609.587.1970 URL: www.princetoninstruments.com	Princeton Instruments' x-ray business unit, the leading provider of scientific x-ray imaging and spectroscopy systems, introduces NEW high-sensitivity, high-resolution, and high-speed x-ray systems--the Quad-RO: 4320, the PIXIS-XF:2048, and the Nano-XF:11000. These systems can very easily be optimized with custom phosphors for x-rays between < 5 KeV and > 50 KeV for x-ray diffraction, micro-computer tomography, streak tube readout, and other applications. Princeton Instruments also offers direct detection systems--the PIXIS-XO and the PI-MTE--for detection of x-rays between ~ 30 eV and 20 KeV and used for applications including coherent x-ray imaging, x-ray holography, and soft x-ray diffraction.
Pro-Dex, Oregon Micro Systems 15201 NW Greenbrier Pkwy, B-1 Beaverton, OR 97006 USA Phone number: 1.503.629.8081 Fax number: 1.503.629.0688 URL: www.Pro-Dex.com	For over 25 years, Pro-Dex, Oregon Micro Systems has consistently proven to be an ideal supplier of multi-axis motion control products. OMS not only produces excellent motion controls--customization and integration are core competencies that fulfill unique requirements faster and with better overall results than typical off-the-shelf solutions provide.

Quality Cryogenics 425 Gennett Drive Jasper, GA 30143 USA Phone number: 1.800.966.6167 Fax number: 1.706.692.6350 URL: www.VJPipe.com	Quality Cryogenics is a manufacturer of vacuum insulated piping and flex hoses for transfer of cryogenic liquids (liquid nitrogen, LN2). We also manufacture other cryogenic equipment and accesories. We are the primary suppier for the LN2 distribution system and branch lines at APS.
Rayonix, L.L.C. 1880 Oak Avenue Suite 120 Evanston, IL 60201 USA Phone number: 1.847.869.1548 Fax number: 1.847.869.1587 URL: www.rayonix.com	Rayonix, L.L.C. has a long history of developing state-of-the-art area detectors for x-ray diffraction. The current products of Rayonix include a single-chip CCD detector, the SX165 (updated version of the popular MarCCD), the SX200 (a large format single-chip detector), and various multi-chip CCD detectors: the MX225, MX300, and MX325. Rayonix recently developed the new high-efficiency MX HE (High Efficiency) series, the first mosaic detectors built with thinned back-illuminated CCD chips. These highly sensitive detectors are especially suited to SAXS and micro-crystal diffraction. Our upcoming product is the MX HS (High Speed) detector, a high-frame-rate mosaic CCD detector utilizing frame transfer CCDs. The MX HS detector is ideal for high throughput synchrotron protein crystallography and time-resolved x-ray diffraction. We also distribute Marresearch GmbH products in North America including the Mar345 Image Plate, MarDTB goniometer, and MarCSC cryogenic sample changer.
SPECS USA Corp. 182 Howard St. # 150 San Francisco, CA 94105-1611 USA Phone number: 1.415.397.7327 Fax number: 1.415.397.7328 URL: www.specsus.com	SPECS USA offers SPECS, CreaTec, and BESTEC products in the USA. We provide systems for surface analysis in UHV for XPS, UPS, AES, ISS, STM/AFM, LEEM/PEEM, LEED, SIMS, SNMS, and HREELS and components such as sources for sample preparation, deposition (k-cell and e-beam), excitation, and charge neutralization. We also provide analyzers, x-ray and UV-monochromators, and research microscopes like LEEM, STM, and LT-STM. Another focus is custom systems combining MBE with spectroscopic and microscopic options. Finally, through BESTEC, we offer custom solutions for synchrotron beamlines such as monochromators, apertures, translation stages and end stations.
Steinmeyer, Inc. 56 Middlesex Turnpike Suite 200 Burlington, MA 01803 USA Phone number: 1.781.273.6220 Fax number: 1.781.273.6602 URL: www.steinmeyer.com	At Steinmeyer, we are dedicated to the design and engineering of precision linear and rotary stages. These compact products are manufactured by our FMD division located in Dresden, Germany and boast some of the lowest runout accuracies available while still using mechanical linear bearings. Some of our stages even have motors and controllers built in for space saving solutions! Steinmeyer has also been manufacturing precision ball screws since 1965 and has established a global reputation for reliability and performance.
Synergy Vacuum, Inc. P.O. Box 2084 2400 E Main St. #F Montrose, CO 81402 USA Phone number: 1.970.209.5802 Fax number: 1.970.240.9229 URL: www.synergyvacuum.com	SYNERGY VACUUM - Original ANEST IWATA Oil-Free Scroll Pumps – including the NEW small and compact ISP-50, new DVSL 100C & 501B high vapor pumping capacity oil-free scroll pumps, plus two oil-free pump/blower packages, turbopPump and RGA carts, and a new lineup of diaphragm pumps. As always, total support – our customizable OEM service and maintenance.
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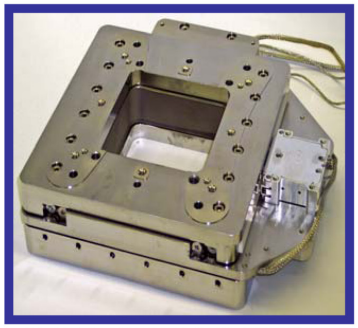
TecRep Corporation 1919 S. Highland Avenue, 330-A Lombard, IL 60148 USA Phone number: 1.630.627.9110 Fax number: 1.630.396.7012 URL: www.tec-rep.com	TecRep is a manufacturer's representative specializing in hardware and software tools utilized in engineering and technical disciplines. Products include electronic test and measurement instrumentation, AC and DC power supplies, power analyzers and energy management tools, data acquisition hardware, electromagnetic compatibility (EMC) test solutions, optical and telecommunications instrumentation, rack-mount enclosures, and accessories.
Thermionics Vacuum Products 231 Otto Street Port Townsend, WA 98368 USA Phone number: 1.800.962.2310 Ext. 126 Fax number: 1.360.385.6617 URL: www.thermionics.com	Founded in1958, Thermionics Laboratoy, Inc. listens to customers and responds with innovative designs and solutions to ordinary and extraordinary challenges. We are a company that finds solutions where others do not. Performance, quality, reliability, and customer service are our highest priority.

Trinos Vacuum Systems Inc. 525 W. Monroe St. Suite 2360 Chicago, IL 60661 USA Phone number: 1.206.203.6310 Fax number: 1.206.203.6311 URL: www.trinos.com	Trinos Vacuum Systems designs and manufacturers vacuum chambers and components for high- and ultra-high vacuum applications. We stock over 4,500 different products and our inventory includes over 50,000 items. Any item can be customized to meet your unique requirements.
Vacuum One 717 N. Ravenswood Suite 240 Chicago, IL 60613 USA Phone number: 1.773.244.3102 Fax number: 1.773.244.3975 URL: www.vacuumone.com	Vacuum One is a Midwest sales representative firm based in Chicago. Our philosophy is to represent vacuum industry leaders and offer a total vacuum solution to our customers. We represent Edwards Vacuum, Inficon, MDC Vacuum Products, Insulator Seal Incorporated, CTI-Cryogenics, Granville-Phillips, Polycold, Gamma Vacuum, SAES Getters, KLA-Tencor, Toho Technology, Advanced Research Systems, and Intlvac.
Varian Inc. 121 Hartwell Avenue Lexington, MA 02421 USA Phone number: 1.800.882.7426 Fax number: 1.781.860.5405 URL: www.varianinc.com/vacuum	Varian Inc. is a world wide leader in providing vacuum solutions. Product offerings include primary, high and ultra-high vacuum pumps, gauges, valves and fittings, as well as leak detection for all applications. Varian offers unique expertise in applications, support, and system design to integrate these superior components into optimized vacuum solutions.
WIENER Plein & Baus Corp. 300 E. Auburn Ave. Springfield, OH 45505 USA Phone number: 1.937.324.2420 Fax number: 1.937.324.2425 URL: www.wiener-us.com	WIENER Plein & Baus, Ltd. provides a full line of electronics for accelerator, experiment control, and read-out. Our product line covers the following vendors and products for the North American market: • WIENER (www.wiener-d.com / Germany): low-voltage power supplies and power supply systems, powered crates for NIM, CAMAC, VME, VME64x, VXS and VXI / custom spec., CAMAC and VME controller and modules. • ISEG (www.iseg-hv.de/vcms.php/home/en / Germany): high voltage supplies, desktop units and modules (NIM, CAMAC, VME), computer controlled multi-channel high-voltage systems. • HYTEC (www.hytec-electronics.co.uk / UK): CAMAC, VME and VE64x modules, IP carrier and IP modules (controller, analog & digital I/O, counter, interfaces, …) with EPICS support. • MESYTEC (www.mesytec.com / Germany): analog electronics for detector read out, data acquisition systems. WIENER NIM, CAMAC, VME / VME64x, VXS, and VXI crates are modular constructed to allow easy replacement of parts like fan tray or power supply. They are combining superior designed mechanic chassis with high quality, microprocessor controlled, low-noise power supplies and a high level of integrated diagnostic and monitoring. The newest feature is the Ethernet interface for remote monitoring and control. In a joint venture between WIENER and ISEG, we provide the new high-density, multi-channel low- and high-voltage power supply system MPOD. A new family of high performance controllers for VME and CAMAC with USB2 interface completes our line of interface modules.
XIA LLC 31057 Genstar Rd. Hayward, CA 94544 USA Phone number: 1.510.401.5760 Fax number: 1.510.401.5761 URL: www.xia.com	XIA LLC (formerly X-ray Instrumentation Associates) produces advanced x-ray and gamma-ray detector electronics and related instruments including high-rate multi-channel digital signal processing electronics for synchrotron and other research applications.

True Nano™ Vacuum Motion Systems

Standard Products for HV (10^{-7} TORR) or UHV (10^{-10} TORR)
Extremely Low Out-Gassing

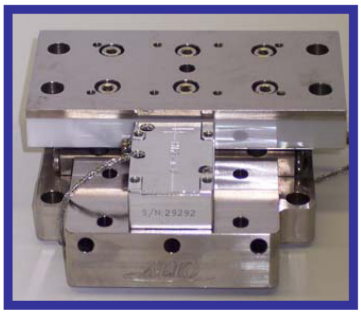
AI-HEX-HR2-HV



AI-HR4-15000E-150-XY-HV

Synchrotron Applications

AI-HEX-HR2-SS-HV



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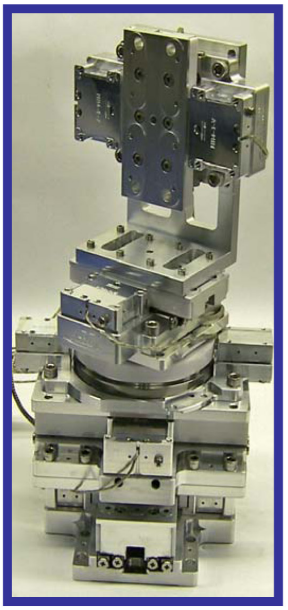
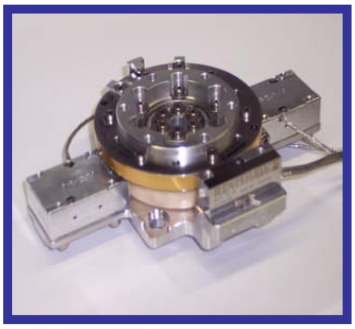


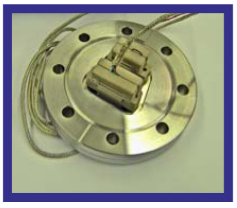
6-Axis HV System



AI-40R-D-HV

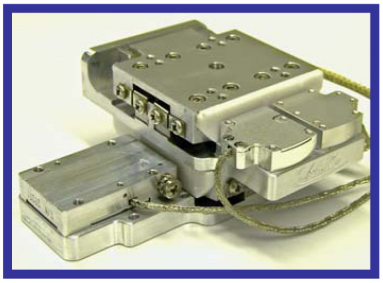
Semiconductor Applications

E-Beam Applications



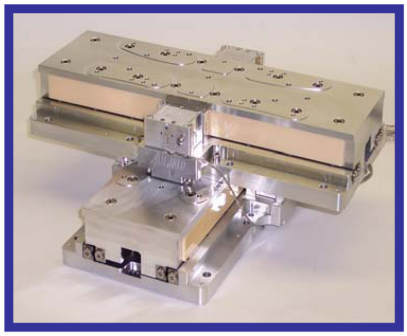
Vacuum Connectors

World Renown Vacuum Systems Expertise



Miniature HV Goni Stack

ALIO INDUSTRIES



AI-D8-20000E-HV XY Stack

GENERAL INFORMATION

Practical Matters

Locations

General sessions will be held in the lecture hall on the first floor of the APS conference center, Bldg. 402. Vendor exhibits will be in the center's lower level and the atrium on the main level. The poster sessions will be held in Building 437 (accessible from the experiment hall). Workshop locations are listed on the Comprehensive Program and posted at the registration desk. A map of the APS and conference center is provided at the end of this program book.

Meals

The conference fee includes coffee breaks each day, the vendor reception, and the poster session reception. Buffet lunches will be served Monday through Wednesday in the tent on the lower-level conference center patio. If you pre-ordered lunches on your registration form, you will receive your tickets when you receive your meeting materials. (A limited number of extra lunch tickets are available for purchase at the registration desk.) The conference banquet will be held Tuesday, May 7, at at the Argonne Guest House. Banquet tickets are nonrefundable after the stated deadline. The Argonne cafeteria and Argonne Guest House restaurant are both open for lunch; the Guest House restaurant is also open for dinner on Monday and Wednesday. A list of nearby restaurants is available on the APS home page (www.aps.anl.gov) under the side heading Visitor Information.

Telephones and Messages

Pay telephones are located in the atrium of Bldg. 401 (the APS central laboratory/office building) and on the lower level of Bldg. 402 (the APS conference center). Messages for you can be left at the registration desk; the telephone numbers there are 630.252.9580 and 630.252.9581. The messages will be posted on a bulletin board by the entrance to the lecture hall. If you need to send or receive a fax, a fax machine is located in Bldg. 401, B1154 (the APS User Office). The number of this machine is 630.252.9250.

Transportation

Conference staff can make limousine reservations for you during the meeting.

ATM

An automated teller machine is located in Bldg. 233, behind the Argonne cafeteria. This machine accepts the following cards: American Express, Discover/Novus, The Exchange, Master Card, Plus, Visa, and 24 Access.

Computer Access

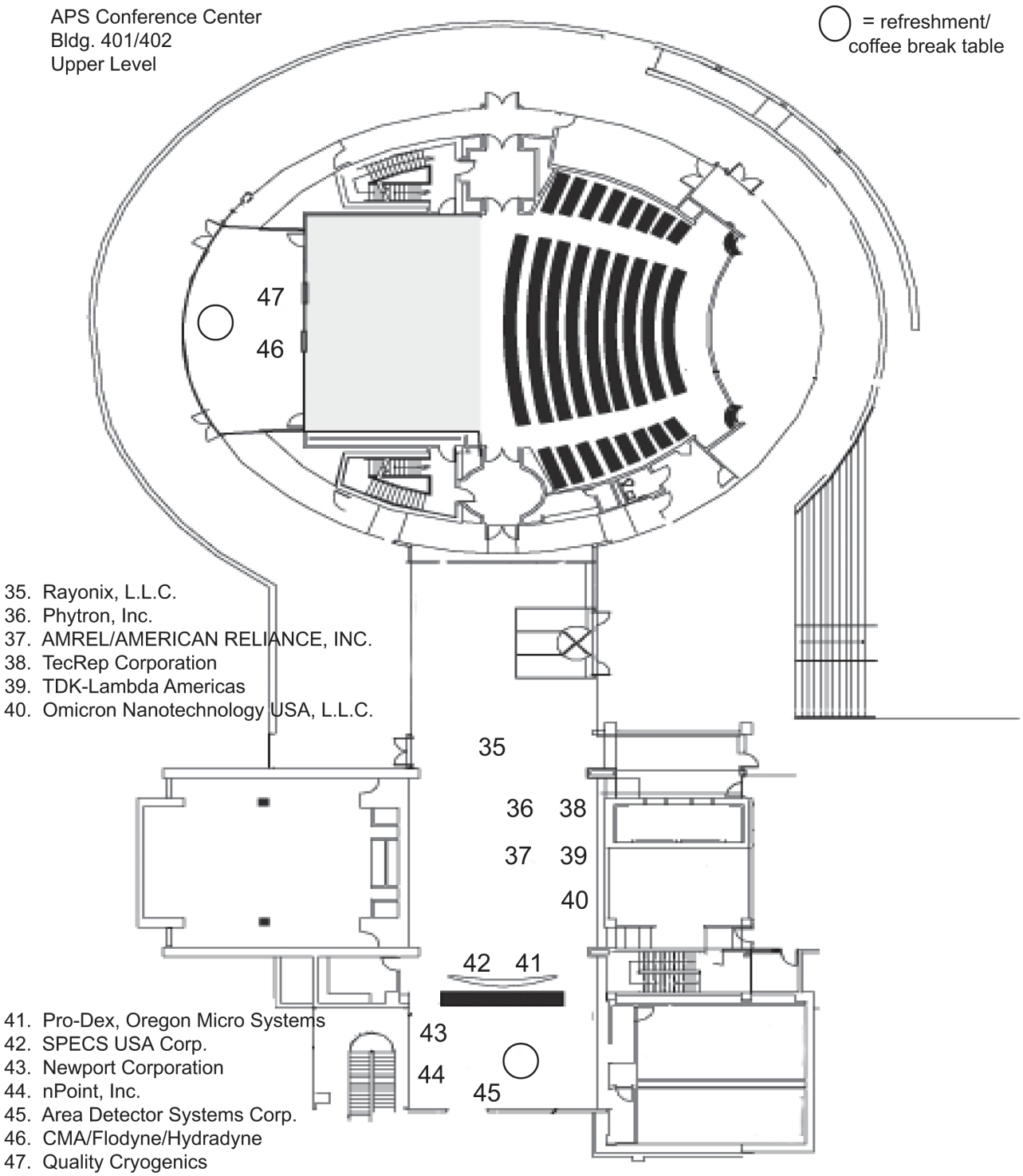
Public computer terminals are available in the Bldg. 401 atrium behind the silver wall. Wireless access is also available in the Conference Center.

To use your laptop computer on the APS wireless networks, complete the following steps:

1. Open the wireless connection on your computer (either the 401 or 402 networks).
2. Read and accept the Argonne internet access policy, which will appear as a Web page on your desktop. After you click "accept," a registration Web page will appear.
3. The registration web page asks you for the following information:
   a. First and last name
   b. E-mail address
   c. Building and room where you will be located *(use 402 conference center)*
   d. Phone number where you can be reached on site *(use 630-252-9090)*
   e. Name of person you are visiting or conference you are attending
   f. Home institution
   g. Do you need to send e-mail directly to an off-site server?
   h. What is the duration of this registration? *(You'll be given choices.)*

If you have problems, please contact someone in the APS User Office (B1154, located immediately off the Conference Center atrium).

Vendor Locations – 2009 Users Week
Argonne National Laboratory

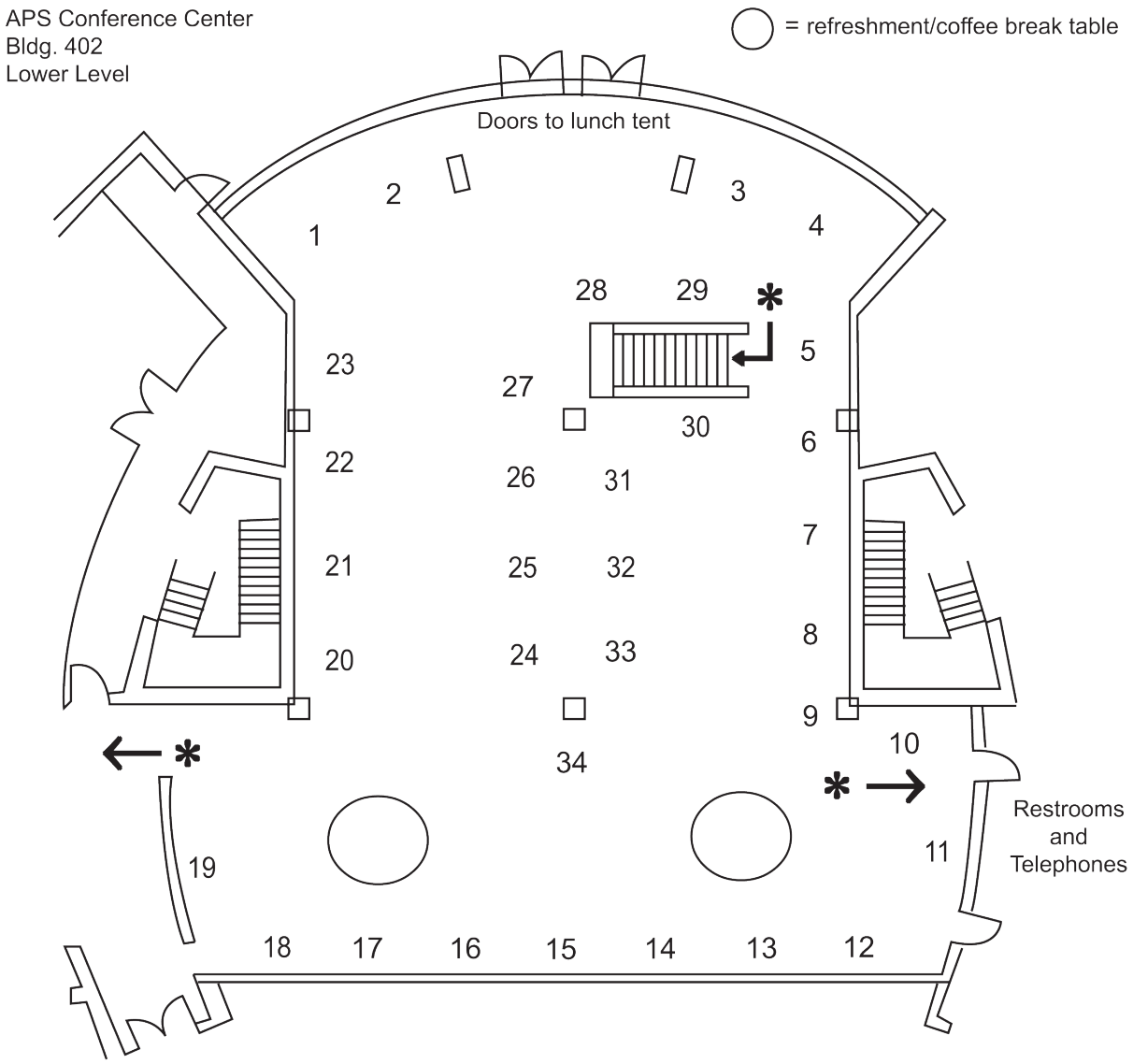


35. Rayonix, L.L.C.
36. Phytron, Inc.
37. AMREL/AMERICAN RELIANCE, INC.
38. TecRep Corporation
39. TDK-Lambda Americas
40. Omicron Nanotechnology USA, L.L.C.

41. Pro-Dex, Oregon Micro Systems
42. SPECS USA Corp.
43. Newport Corporation
44. nPoint, Inc.
45. Area Detector Systems Corp.
46. CMA/Flodyne/Hydradyne
47. Quality Cryogenics

Vendor Locations – 2009 Users Week
Argonne National Laboratory

APS Conference Center
Bldg. 402
Lower Level



1. ACCEL Instruments GmbH
2. Pfeiffer Vacuum
3. MICOS USA
4. Mager Scientific, Inc.
5. Blake Industries, Inc.
6. WIENER, Plein & Baus, Ltd.
7. XIA LLC
8. Aerotech, Inc.
9. Bruker AXS, Inc.
10. Thermionics Vacuum Products
11. CVI Melles Griot
12. FMB Oxford Limited
13. Trinos Vacuum Systems Inc.
14. Advanced Control Systems Corp.
15. Oerlikon Leybold Vacuum
16. attocube systems AG
17. Alan Burrill Technical Sales
18. Synergy Vacuum, Inc.
19. ALIO Industries
20. Midwest Vacuum, Inc.
21. Nor-Cal Products, Inc.
22. Princeton Instruments
23. Canberra
24. Steinmeyer, Inc.
25. CATIAD Mechanical Engineering
26. Johnsen Ultravac Inc.
27. Advanced Research Systems, Inc.
28. Advanced Design Consulting USA, Inc.
29. Carl Zeiss Laser Optics
30. MDC Vacuum Products, LLC
31. Vacuum One
32. PI (Physik Instrumente) LP
33. Varian, Inc.
34. Minarik Corporation

* UPSTAIRS/ATRIUM EXHIBITS PLUS REFRESHMENT TABLES

SCHEDULE AT A GLANCE

2009 Users Week at Argonne National Laboratory

	Sunday, May 3	Monday, May 4	Tuesday, May 5	Wednesday, May 6
Morning		**Registration** 7:00 – 5:00 401 Atrium	**Registration** 7:30 – 5:00 401 Atrium	**Registration** 7:30 – 5:00 401 Atrium
		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
		Opening Session 8:15 – 12:00 402 Lecture Hall • *APS Update* • *Congessional Report* • *DOE Perspective* • *Compton Award* • *CNM Update* • *EMC Update*	**APS Renewal Focus, Pt. 1: Science** 8:00 – 11:45 402 Lecture Hall • *Dynamics of Life (8:10)* • *Materials for Energy Applications (8:50)* • *Materials under Pressure (9:25)* • *Chemistry in Action (10:30)* • *User Visions (11:10)*	**WK2 - Optics for the Future** 8:40 – 12:15 402 Lecture Hall
				WK4 High-Speed Imaging Opportunities with X-rays 8:55 – 11:55 (& PM) 402 Room E1100/E1200
				WK5 Imaging Structural Hierarchy in Biological Systems 9:00 – 12:00 (& PM) 401 Room A1100
				WK6 Synchrotron Radiation in Chemical Science 8:55 – 11:45 (& PM) 401 Room A5000
				WK7 High Pressure Synchrotron Science: Future Directions (Auxiliary) 8:30 – 12:30 (& PM) 326 Auditorium
Noon		Lunch 11:45 – 1:30 Tent	Lunch 11:45 – 1:30 Tent	**APSUO St. Com.** 12:00 – 1:00 401 Fifth Floor Lunch 11:45 – 1:30 Tent
Afternoon	**Exhibitor Set-up** 11:00 - 3:00 402 Gallery, 401 Atrium	**Science Highlights** 1:00 – 3:30 402 Lecture Hall • *Argonne Strategic Directions* • *Science Talks* • *Invited Student Talk*	**APS Renewal Focus, Pt. 2: Techniques** 1:00 – 5:10 402 Lecture Hall • *Imaging (1:00)* • *Microscopy (1:35)* • *Optics (2:10)* • *Detectors (3:15)* • *Short-pulse science (3:50)* • *User Visions (4:30)*	**WK1 Detectors for the Future** 1:30 – 5:00 402 Lecture Hall
				WK3 NextGen Nanopositioning 1:30 – 5:00 440 (CNM) Room 105/106
				WK4 High-Speed Imaging Opportunities with X-rays 1:20 – 5:00 402 Room E1100/E1200
				WK5 Imaging Structural Hierarchy in Biological Systems 1:30 – 5:00 401 Room A1100
				WK6 Synchrotron Radiation in Chemical Science 1:15 – 5:15 401 Room A5000
				WK7 High Pressure Synchrotron Science: Future Directions (Auxiliary) 2:15 – 6:00 326 Auditorium
		APS Renewal Status Report 3:30 – 5:00 402 Lecture Hall	**APS Renewal Discussion** 5:10 – 5:45 402 Lecture Hall	**Workshop Wrapup Reports** 5:15 – 5:45 402 Lecture Hall
Evening	**Registration** 5:00 - 7:00 401 Atrium	**Poster Session and Reception** 5:00 – 7:00 Bldg. 437	**Banquet** 6:00 – 9:00 Argonne Guest House	
	Exhibitor Expo and Reception 5:00 – 7:00 401 Atrium, 402 Gallery	**Partner User Council** 7:00 – 8:00 Argonne Guest House		

Argonne NATIONAL LABORATORY

Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439

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