2010 APS/EMC Users Meeting

Argonne National Laboratory

May 3–5, 2010

Argonne NATIONAL LABORATORY

Celebrating 20 Years of Groundbreaking Science

Program and Abstracts

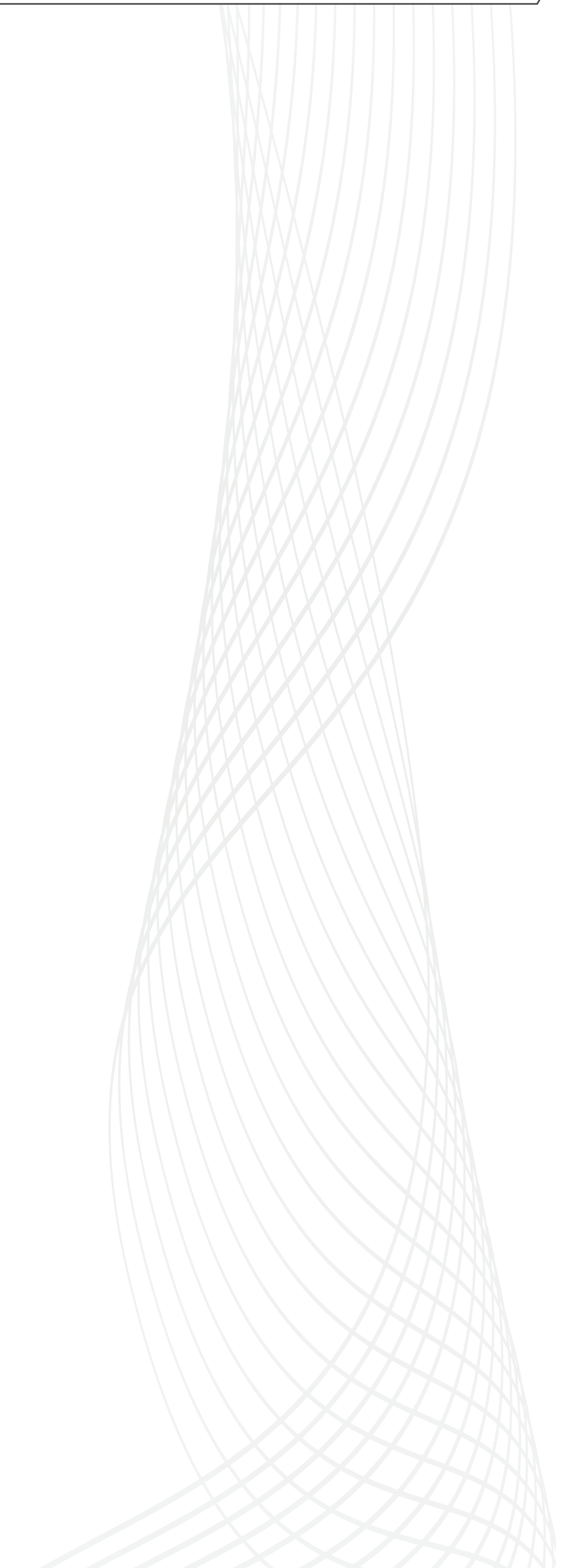
Cover Photo

Breaking ground for the Advanced Photon Source on June 4, 1990, were, from left, David Nelson, U.S. Department of Energy; David Moncton, Argonne Associate Laboratory Director for APS; Hilary Rauch, DOE; Argonne Director Alan Schriesheim; Rep. Harris Fawell, R-Ill; and Rep. Lynn Martin, R-Ill. The six spades were joined by a section of aluminum vacuum chamber from the APS.

Then and Now

The future site of the APS as it looked in May 1990 just before the groundbreaking (top), and as it looked nearly twenty years later in May 2009 (bottom). To compare the photos, note the water tower in the middle distance and the small complex of buildings in the foreground.

PROGRAM &
ABSTRACTS



May 3-5, 2010

Advanced Photon Source
Argonne National Laboratory
Argonne, Illinois USA

Celebrating 20 Years of Groundbreaking Science

User Facilities at Argonne National Laboratory

User Contacts

Advanced Photon Source
http://www.aps.anl.gov
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apsuser@aps.anl.gov

Argonne Leadership Computing Facility
http://www.alcf.anl.gov
630-252-0929

Argonne Tandem Linac Accelerator System
http://www.phy.anl.gov/atlas
630-252-4044

Center for Nanoscale Materials
http://nano.anl.gov
630-252-6952
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Electron Microscopy Center
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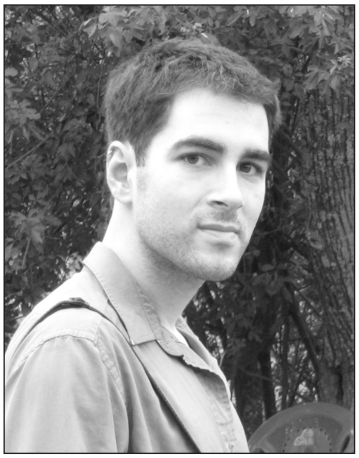

2010 Rosalind Franklin Young Investigator Award

Rafael Jaramillo
Harvard University

The Advanced Photon Source (APS) Users Organization has named Rafael Jaramillo as the recipient of the 2010 Rosalind Franklin Young Investigator Award. Jaramillo is recognized for furthering understanding of itinerant magnetism and for contributions to the study of quantum matter at high pressure using synchrotron X-ray diffraction.

The award recognizes an important technical or scientific accomplishment by a young investigator that depended on, or is beneficial to, the APS.

Jaramillo received a B.S. in applied physics from Cornell University and a Ph.D. in physics from The University of Chicago, where he was a member of Thomas Rosenbaum's research group. The work recognized in this award resulted from an extensive collaboration between Jaramillo and Yejun Feng, who is now a member of the APS staff in the Magnetic Materials Group, X-ray Sciences Division. Jaramillo is currently a Ziff Environmental Fellow at the Harvard University Center for the Environment and the Harvard School of Engineering and Applied Sciences, where he is developing advanced materials for solar energy conversion.

Jaramillo's work has focused on itinerant magnetism, an ephemeral form of magnetism that arises from interactions among conduction electrons in a metal, and specifically on the question of how magnetism emerges from disorder. Previous work had hinted that it would be possible to completely suppress the magnetism in chromium metal by applying very high pressures at temperatures near absolute zero. The result would be a phase transition between magnetism and disorder. However, unlike well-understood phase transitions that are driven by thermal energy (for example, ice melting into water), this phase transition would be governed by quantum mechanical agitation of the conduction electrons. If accessible, this transition would be a prime example of quantum criticality.

Getting to the quantum critical point, however, was a daunting challenge. The phase transition would be signaled by subtle changes in the X-ray diffraction pattern as the electron spins changed from ordered to disordered—changes so subtle that picking them up would be like trying to hear a pin drop in the middle of a football game. Moreover, these subtle changes would have to be recorded from miniature crystals that were maintained intact while being squeezed at pressures up to 10 GPa (100,000 atmospheres) at temperatures near absolute zero. But Jaramillo and colleagues did just that, and by measuring signals more than one billion times weaker than the standard diffraction pattern they were able to show that chromium does undergo a quantum phase transition at high pressure and low temperature. Of even greater interest to physicists, there appears to be no limit on how closely such an experiment can approach the quantum phase transition. Chromium therefore provides a unique example of a "naked quantum singularity." The report of quantum criticality in chromium appeared in the May 21, 2009, issue of *Nature* (**459**, 405, 2009). An overview of the phenomena involved is given in an APS research highlight that coincided with the publication of the *Nature* paper (May 21, 2009).

The award recognizes Jaramillo's achievement in integrating three techniques to achieve the required sensitivity: low-temperature studies, high-pressure diamond anvil cells, and high-resolution diffraction at beamline 4-ID-D. After several years spent wrangling these techniques together, the team was able to resolve both charge density waves and spin density waves (*nine* orders of magnitude weaker than the Bragg peak), at extreme pressures and low temperatures. This work created a new toolbox for measuring condensed matter systems by bringing high-pressure techniques to a level of precision comparable to long-established techniques such as applied magnetic fields and variable temperature. As a result, a new set of questions have become open to study, and the technique they developed is now being applied at the APS to other condensed matter systems.

To achieve this remarkable sensitivity, "we evolved a series of technical approaches that relied on the state-of-the-art diffractometer at sector 4, the low-temperature experience in our group, and high-pressure experience at APS," Jaramillo said. "We were starting from a good base of expertise on diffraction at high pressure at the APS, and we were really grateful for the advice we got from around the ring. We did a lot of little things, like when we wanted to get from 8 K to 5 K. Each one doesn't sound like much but at the end of the day we had a new technique." After years of "sustained meticulousness" and a lot of sleepless nights, Jaramillo is especially gratified that the technique has proved more broadly useful; several users at sector 4 are already working to apply these methods to their systems. "We were chasing the quantum phase transition in chromium, but it's really nice to see there were other things waiting to be cracked." The work also gratified his own impulse to understand the relationship between order and disorder, Jaramillo said: "That's the motivating interest behind most of condensed matter physics, and this project answered it in a very satisfying way."

Publications

R. Jaramillo, Y. Feng, T. F. Rosenbaum, "Diffraction line-shapes, Fermi surface nesting, and quantum criticlaity in antiferromagnetic chromium at high pressure," *J. App. Phys*. **107**, in press.

Y. Feng, R. Jaramillo, J. Wang, Y. Reng, T. F. Rosenbaum, "High-pressure techniques for condensed matter physics at low temperature," *Rev. Sci. Instrum.* **81**, 041301 (2010).

R. Jaramillo, Y. Feng, J. C. Lang, Z. Islam, G. Srajer, P. B. Littlewood, D. B. McWahn, T. F. Rosenbaum, "Breakdown of the Bardeen-Cooper-Schrieffer ground state at a quantum phase transition," *Nature* **459**, 405 (2009).

R. Jaramillo, Y. Feng, J. C. Lang, Z. Islam, G. Srajer, H. M. Ronnow, P. B. Littlewood, T. F. Rosenbaum, "Chromium at high pressures: Weak coupling and strong fluctuations in an itinerant antiferromagnet," *Phys. Rev. B* **77**, 184418 (2008).

Y. Feng, R. Jaramillo, G. Srjaer, J. C. Lang, Z. Islam, M. S. Somayazulu, O. G. Shpyrko, J. J. Pluth, H. K. Mao, E. D. Isaacs, G. Aeppli, T. F. Rosenbaum, "Pressure-tuned spin and charge ordering in an itinerant antiferromagnet," *Phys. Rev. Lett. 99*, 137201 (2007).

APS Research Highlight

"Using High Pressure to Reveal Quantum Criticality in an Elemental Antiferromagnet," May 21, 2009, http://www.anl.gov/Media_Center/News/2009/news090618.html.

COMPREHENSIVE
PROGRAM

Monday, May 3

Exhibits:	8:00 – 5:00 *Bldg. 402, Gallery; Bldg. 401, Atrium*
Registration:	7:00 – 5:00 *Bldg. 401, Atrium*
Lunch:	12:00 – 1:30 *In the tent*

Job Fair

A lunchtime talk will focus on postdoctoral opportunities at Argonne (Monday, 12:15 – 1:00 in Room A1100; feel free to bring your lunch). The web page for the Argonne postdoctoral program is http://www.dep.anl.gov/postdocs/. In addition, an Argonne human resources representative will be available each day at the registration desk and at the poster session to discuss career opportunities at the Advanced Photon Source.

Opening Session — Bldg. 402 Lecture Hall

8:50 – 9:00	Paul Fuoss, Moderator (Argonne National Laboratory), APSUO Chair *Welcome*
9:00 – 9:05	Mark Peters (Deputy Director for Programs, Argonne National Laboratory) *Welcome from the Laboratory*
9:05 – 9:35	Harriet Kung (Associate Director, Office of Basic Energy Sciences, DOE Office of Science) *Office of Science Perspective*
9:35 – 10:00	Douglas Platz (Legislative Assistant to U.S. Congressman Bill Foster) *Congressional Report*
10:00 – 10:30	Murray Gibson (Director, APS) *Update on the Advanced Photon Source*
10:30 – 11:00	Coffee Break Gallery and Atrium
11:00 – 11:15	Dean Miller (Director, EMC) *Update on the Electron Microscopy Center*
11:15 – 11:30	Amanda Petford-Long (Director, CNM) *Update on the Center for Nanoscale Materials*

11:30 – 12:00	KEYNOTE PERSPECTIVE Gopal Shenoy (APS, Argonne National Laboratory) *Conceiving and Realizing APS: A Symphony of Fresh Vision and Extraordinary Challenges*
12:15 – 1:00	POSTDOC LUNCH LECTURE (Bldg. 401, Rm. A1100) Giselle Sandì-Tapia (Argonne National Laboratory) *Postdoctoral Opportunities at Argonne National Laboratory* *(OK to bring lunch to the meeting room)*
12:00 – 1:30	Lunch Tent

Plenary Session — Bldg. 402 Lecture Hall

1:25 – 1:30	Dave Tiede, Moderator (Argonne National Laboratory), APSUO Vice-Chair *Welcome*
1:30 – 1:50	AWARD RECIPIENT: Student Invited Talk Student Winner TBD
1:50 – 2:30	KEYNOTE SCIENCE Wayne Hendrickson (Columbia University) *Synchrotron Crystallography in Biological Discovery*
2:30 – 3:00	Coffee Break Gallery and Atrium
3:00 – 3:40	KEYNOTE SCIENCE J. Kent Blasie (University of Pennsylvania) *Interfacial Structure of Biological and Biomimetic Films*
3:40 – 4:20	KEYNOTE SCIENCE John Parise (Stony Brook University) *Understanding the Composition and Structure of Crystallographically Challenged Materials*
4:20 – 5:00	UPGRADE Dennis Mills (APS, Argonne National Laboratory) *APS Upgrade Summary*
5:00 – 7:00	APS poster session & reception APS Upgrade posters Bldg. 401, Rm. B1200 (former library)
7:30 – 9:00	APS Partner User Council Meeting & Dinner Argonne Guest House

Tuesday, May 4

Exhibits:	8:00 – 5:00 *Bldg. 402, Gallery; Bldg. 401, Atrium*
Registration:	8:00 – 5:00 *Bldg. 401, Atrium*
Lunch:	12:00 – 1:30 *In the tent*

APS Science Session I — Bldg. 402 Lecture Hall

8:50 – 9:00	Alec Sandy, Moderator (APS, Argonne National Laboratory) *Welcome and Announcements*
9:00 – 9:30	AWARD RECIPIENT: DOE Early Career Award Antonino Miceli (APS, Argonne National Laboratory) *My Perspective on Future X-ray Detectors*
9:30 – 10:00	USER SCIENCE HIGHLIGHT: Biomembranes Ka Yee Lee (The University of Chicago) *Beyond Wrinkles: Stress Relaxation in Lipid Monolayers and Other Elastic Thin Films*
10:00 – 10:30	UPGRADE SCIENCE: Ultrafast Dynamics David Reis (SLAC and Stanford University) *Science Opportunities with Short-Pulse X-rays*
10:30 – 11:00	Coffee Break Gallery and Atrium
11:00 – 11:30	UPGRADE SCIENCE: Imaging and Coherence Ross Harder (Argonne National Laboratory) *Coherent X-ray Diffraction Imaging of Strain on the Nanoscale*
11:30 – 12:00	AWARD RECIPIENT Rafael Jaramillo (Harvard University) *Magnets, Metals, and Insulators: Electronic Order at High Pressure*
12:00 – 1:30	Lunch Tent

APS Science Session II — Bldg. 402 Lecture Hall (Parallel Session)

1:25 – 1:30	Nadia Leyarovska, Moderator (APS, Argonne National Laboratory) *Welcome and Announcements*
1:30 – 2:00	UPGRADE SCIENCE: High-Resolution Spectroscopy Gerald Seidler (University of Washington) *X-ray Spectroscopies and Hard Problems in Energy Science: One Size Doesn't Fit All*
2:00 – 2:30	UPGRADE SCIENCE: Extreme Conditions Malcolm Guthrie (Carnegie Institution of Science) *Extreme Environments, Multiple Length Scales*
2:30 – 3:00	UPGRADE SCIENCE: Interfaces in Complex Systems Roy Clarke (University of Michigan) *Mapping Epitaxial Interfaces*
3:00 – 3:30	Coffee Break Gallery and Atrium
3:30 – 4:00	UPGRADE SCIENCE: Proteins to Organisms Maxim Boyanov (Argonne National Laboratory) *X-ray Biogeochemistry: Elucidating Bacteria-Mineral-Contaminant Interactions at the Molecular Scale*
4:00 – 4:30	USER SCIENCE HIGHLIGHT: Microbeam Protein Crystallography Robert Fischetti (GM/CA-CAT, Argonne National Laboratory) *Macromolecular Crystallography and Observations of Reduced Radiation Damage with Micro-beams*
4:30 – 5:00	APSUO "Town Hall Meeting" Moderator: Paul Fuoss, Argonne National Laboratory
6:00 – 9:00	Banquet Argonne Guest House

EMC Science Session — Bldg. 402, Rm. E1100/E1200 (Parallel Session)

In situ Electron Microscopy and the Potential and Use of Aberration-corrected TEMs

1:25 – 1:30	Jennifer Mawdsley, Moderator (Argonne National Laboratory) *Welcome and Announcement*
1:30 – 2:00	James Ciston (Brookhaven National Laboratory) *Catalysis in an Aberration-free Environment: Atomic Resolution Studies of* in situ *Water Splitting*
2:00 – 2:30	Paulo Ferreira (University of Texas–Austin) *Are Dislocations Possible in Nanoparticles?*
2:30 – 3:00	Renu Sharma (NIST) In situ *Evaluation of the Factors Controlling Carbon Nanotube Synthesis*
3:00 – 3:15	Coffee Break Gallery and Atrium
3:15 – 3:45	Frances Ross (IBM T.J. Watson Research Center) *Step Flow and Interface Formation during the Growth of Heterojunction Nanowires*
3:45 – 4:15	Nigel Browning (University of California, Davis & Lawrence Livermore National Laboratory) *Dynamic Transmission Electron Microscopy (DTEM)*
4:15 – 4:45	Andrew Minor (Lawrence Berkeley National Laboratory & University of California, Berkeley) *Quantitative Nanomechanical Testing in a TEM*
4:45 – 5:15	EMC Users Group Meeting and Elections
6:00 – 9:00	Banquet Argonne Guest House

Wednesday, May 5

8:00 – 5:00	Exhibits *Bldg. 402 Gallery, Bldg. 401 Atrium*
8:00 – 1:00	Registration *Bldg. 401 Atrium*
12:00 – 1:30	Lunch *Tent*
12:00 – 1:00	APSUO Steering Committee Lunch *Bldg. 401, Fifth Floor Gallery*

Workshops

	AM Session	AM Break	PM Session	PM Break
WK1 **Application of Advanced X-ray Techniques to Industrial Research**	**8:50 – 12:00** *Bldg. 402, Lecture Hall*	10:15 –10:30	**1:30 – 4:30** *Bldg. 402, Lecture Hall*	3:00 – 3:15
WK2 **Beamline 2.0: The Fully Integrated Instrument**	**8:30 – 12:00** *Bldg. 402, Rm. E1100/ E1200*	10:00 – 10:30	**1:30 – 5:30** *Bldg. 402, Rm. E1100/ E1200*	3:30 – 4:00
WK3 **Linking Structure and Magnetism in Novel Materials: Future Opportunities at an Upgraded APS**	**8:45 – 12:15** *Bldg. 440, Rm. A105/ A106*	10:10 – 10:30	**1:30 – 5:30** *Bldg. 440, Rm. A105/ A106*	3:15 – 3:45
WK4 **Science Opportunities with an X-ray Free Electron Laser Oscillator**	**8:30 – 12:30** *Bldg. 401, Rm. A1100*	10:25 –10:45		
WK5 **Biomolecular Assemblies as Materials Synthesis Templates: From Molecular Fundamentals to Energy and Environmental Sustainability Applications**	**8:40 – 12:25** *Bldg. 401, Rm. A5000*	10:30 – 10:45		
WK6 **Actinide Research at the APS: Successes and Prospects**			**1:00 – 5:20** *Bldg. 401, Rm. A1100*	3:05 – 3:20
WK7 **Are Phase-Contrast and Diffraction Imaging/Microscopy Ready for Biology and Medicine?**			**1:30 – 4:45** *Bldg. 401, Rm. A5000*	3:05 – 3:30

GENERAL SESSION
ABSTRACTS

Monday, May 3

APS

Interfacial Structure of Biological and Biomimetic Films

J. Kent Blasie[1], Venkata Krishnan[1], Jaseung Koo[1], Ruili Zhang[1], Jing Liu[1], Andrey Tronin[1], Joseph Strzalka[2], Ivan Kuzmenko[2], Thomas Gog[2] and Chian Liu[2]

[1] Department of Chemistry, University of Pennsylvania, Philadelphia, PA 19104, USA
[2] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Interferometric techniques provide substantial advantages in the study of the profile structures of ultrathin organic and bio-organic films at interfaces by specular X-ray and neutron reflectivity. Employing a *multilayer* reference structure provides not only for a unique solution to the phase problem in the derivation of the scattering-length density profile across the interface, but also for a substantial improvement in both the spatial resolution and sensitivity to details in this profile structure. Artificial proteins, engineered to exhibit specific biological or abiological functionality, as well as natural proteins with their evolved functionality, can be vectorially oriented in an extended 2D ensemble at liquid-gas interfaces if they possess sufficient amphiphilic nature, or at solid-liquid interfaces if they possess some chemical specificity for the solid surface. Extending X-ray reflectivity to higher X-ray energies, enhanced by such interferometric techniques, allows investigation of the "steady-state" profile structures of the proteins at the solid-liquid interface as a function of the electrochemical potential across the interface. Undulator-based sources, cylindrically bent reference structures, and fast time-framing detectors further allow "time-resolved" structural studies of such proteins in response to a relevant excitation. Examples illustrating these recent developments address key problems in both materials device applications and membrane biophysics.

Support: Grants (JKB) from the DOE, NSF & NIH

APS

Understanding the Composition and Structure of Crystallographically Challenged Materials

J. B. Parise

Stony Brook University, Stony Brook NY 11794, USA

Amorphous or poorly crystalline materials are ubiquitous in the environment, where they are important actors in several chemical cycles, including H_2O, S, CO_2, etc. Technologically, the utility of amorphous materials ranges from applications such as glass formation, for nuclear waste disposal for example, to biologically mediated nanoparticle formation and cryobiological research on organ preservation. The atomic arrangements in these materials, and the changes these arrangements undergo with changing environmental conditions, are key to understanding fundamental issues such as how such materials are stabilized in or destabilized from their nanocrystalline form. The derivation of useful models for atomic arrangements using the pair distribution function (PDF) approach are now well established; this maturity is the result of burgeoning capabilities at high-energy X-ray scattering beamlines and the judicious use of isotopically substituted materials at neutron sources. Further, there is growing realization that *in situ* studies are essential to ensuring the structures determined from PDF are useful and that metastable intermediate states are captured.

Several topical examples will serve to illustrate the need for complementary techniques to supplement PDF data. For example, a growing body of evidence confirms a single-phase model (SPM) suffices to explain the atomic arrangement in inorganically and biologically derived 2- and 6-line varieties of ferrihydrite, an important iron oxide hydroxide in environmental remediation applications. The 50 years of effort expended on studying this material produced inconsistent results, probably due to variations in preparation, and confusion due to over-fitting information-poor diffraction and X-ray absorption data with overly complex models. Careful large-batch synthesis of hydrogenous and deuterated samples and the use of several analytical probes, including IR, thermal analysis, and X-ray and neutron scattering on synthetic and natural samples now provide a consistent picture: (1) there is no need to fit the data with a three-phase model; and (2) models claiming water contents of 10% or more are at odds with combined *in situ* X-ray diffraction–differential scanning calorimetry (DSC) data and *ex situ* thermogravimetric analysis–DSC data.

X-ray measurements performed on glasses and aerodynamically levitated melt droplets show evidence of structural heterogeneities in the liquid state and the transitions in the glass state under compression. Changes in the PDF upon cooling or compression are enhanced using diffraction isosbestic points (DIPs) and the complementary use of ultrasonic techniques to reveal "hidden" transitions. For example, molecular dynamics simulations of $CaO-SiO_2$ melts and observations of DIPs indicate an underlying structure associated with the polymerization of CaO_6 units. In the high temperature liquid, DIPs indicate the increase in edge shared Ca octahedra is linear, however as the T_g is approached the magnitude of the structural changes increases rapidly.

Acknowledgments: The genesis and further development of the research described grew out of discussions and collaboration with colleagues at APS-11-ID-C (C. Benmore, S. Antao, R. Weber), 11-ID-B (P. Chupas, K. Chapman), and 1-ID-C (P. Lee, S. Shastri), and at Stony Brook (R. Reeder, R. Harrington, W. Xu, F. M. Michel), Temple (D. B. Hausner, S. Debnath, D. R. Strongin), Birmingham (J. Hriljac), and Montana (C. Jolley, T. Douglas).

Tuesday, May 4 — Morning

APS

My Perspective on Future X-ray Detectors

Antonino Miceli

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

I will give a brief overview of the current state of X-ray detectors for synchrotron research. I will discuss several new detectors that will be commercially available in the next two years, including the next-generation Pilatus XFS and the analog pixel array detector from ADSC. Several new detector architectures will be discussed that have the potential to provide higher spatial resolution and efficiencies at higher energies (>20 keV). Finally, I will discuss the superconducting detectors project at the APS.

APS

Coherent X-ray Diffraction Imaging of Strain on the Nanoscale

R. Harder[1], M. Newton[3], L. Beitra[2], S.J. Leake[4], I.K. Robinson[2]

[1] Argonne National Laboratory, Argonne, IL, USA
[2] University College London, London, UK
[3] Surrey University, Surrey, UK
[4] Paul Scherrer Institut, Villigen, Switzerland

In nanoscience, the bulk concepts of lattices and crystal defects must be reconsidered in order to explain why nanomaterials have new and exciting properties. The current state of the art in coherent x-ray diffraction (CXD) instrumentation resides at the APS and is capable of imaging the total lattice deformation of micrometer- and sub-micrometer-sized crystals with high spatial resolution. By measuring the coherently scattered photons in the vicinity of Bragg peaks of the samples, and computationally inverting the intensities to an image, we gain this powerful capability. As a result, the entire strain tensor of these tiny crystals can be determined in three dimensions, which then opens the door to understanding their, often unique, physical and electronic properties.

The APS upgrade will enable beautiful new measurements on samples inaccessible to CXD imaging today. The current limitation of studying small, compact objects can be removed with the implementation of the newest scanning coherent diffraction microscopy techniques.

In this talk, I will highlight recent results from micrometer-sized zinc oxide crystals and sub-micrometer-sized gold crystals and also discuss future capabilities afforded by an upgraded CXD instrument at the APS.

APS

Magnets, Metals and Insulators: Electronic Order at High Pressure

Rafael Jaramillo
Harvard University, Cambridge, MA 02138, USA

X-ray diffraction was originally developed to probe the distributions of ions in condensed matter; the most famous such measurement remains Rosalind Franklin's photo 51. Synchrotrons provide the sensitivity to probe not only the distribution of ions, but also of conduction electrons and magnetic moments as they organize within magnets, metals, and insulators. I will discuss measurements of the charge and spin ordering in the elemental antiferromagnet Cr as the magnetism is suppressed towards the quantum critical point with pressure in a diamond anvil cell. This experiment resolves a long-standing question in the field of itinerant magnetism, establishes an important model system for studying quantum criticality, and expands the possibilities for probing quantum matter at high pressure using x-ray diffraction.

Tuesday, May 4 — Afternoon, APS

APS

X-ray Spectroscopies and Hard Problems in Energy Science: One Size Doesn't Fit All

Gerald Seidler
University of Washington, Seattle, WA 98195, USA

"*No one spectroscopy, or spectrometer, serves all purposes.*" While few would argue with this statement, it is worthwhile to discuss two broad realizations of this observation in energy sciences research. First, a deep understanding of the electronic and chemical complexity of many of the most relevant or promising materials for sustainable energy applications requires the coordinated use of multiple spectroscopic methods. Whether one is investigating the charge transfer in novel battery electrodes, the chemical content of multiphase fuel cell materials, the local coordination or chemistry in catalysts, or the nature of chemical bonding in actinide compounds, an integration of results from several synchrotron radiation techniques is frequently necessary. This need for integration raises new opportunities and new difficulties in experiment design, data analysis, and comparison with theory. Second, many experiments of both fundamental and applied interest in energy sciences require sophisticated support apparatus to achieve the desired sample conditions; this includes, for example, laser pump/probe methods, *in situ* film growth, extremes in temperature or magnetic field, and electrochemical, chemical, or high-pressure chambers. With growing frequency, compatibility issues arise between the constraints imposed by the equipment needed to achieve the desired sample conditions and the constraints presently required by the available X-ray technique, instrument, or end-station. To illustrate the above issues, I'll discuss recent APS-based studies and instrument development projects that serve to broaden the impact and applicability of X-ray spectroscopic methods in several branches of energy sciences.

APS
Extreme Environments, Multiple Length Scales

Malcolm Guthrie
Carnegie Institution for Science, Washington, DC 20005, USA

Our modern world is shaped greatly by a wide range of materials with diverse properties and uses. At a fundamental level, our ability to create new useful materials depends on a detailed understanding of how atomic level processes propagate to longer length scales and how these determine material properties. Subjecting matter to extreme conditions is important because a single, closed system can be driven through multiple states with radically different properties. For example, under pressure, elemental oxygen first solidifies and then progresses through a series of insulating states, before finally metallizing at around 100 GPa; under combined pressures and temperatures, graphitic carbon can be driven from a soft conductor into diamond, which is both insulating and the hardest material known to man. When coupled with extreme environment capabilities, synchrotron beams are ideally suited to reveal the changes in structure, dynamics, and magnetic properties that accompany these transitions.

Of particular significance are the hard X-rays available at sources such as the APS, which can readily penetrate complex sample-environment set-ups. With renewal of the APS, a whole new range of experimental environments and probes will become accessible. In this talk, some of the more dramatic advances will be highlighted, with particular attention focused on future possibilities for structural characterization that bridges length scales from atomic, through nanometer, to micrometer.

APS
Mapping Epitaxial Interfaces

Roy Clarke
University of Michigan Applied Physics Program, Ann Arbor, MI 48109, USA

Epitaxial heterostructures constitute a large fraction of the materials systems used in current optoelectronics technology. As device dimensions continue to shrink to the nanoscale, atomic interfaces play an increasingly dominant role in their characteristics and performance. Moreover, new classes of devices are envisioned based on novel phenomena emerging from the complex ionic and electronic rearrangements occurring at interfaces. Energy harvesting, quantum information processing, and smart sensors are but a few of the possible applications. An essential requirement for harnessing these transformative developments is to provide accurate and detailed maps of the structure, chemical composition, and strain at epitaxial interfaces prepared by various deposition methods, including molecular beam epitaxy, metallorganic chemical vapor deposition, focused ion beam and pulsed laser deposition. This presentation will describe some of the exciting science drivers on the APS-U horizon, in the context of the proposed X-ray Interface Science (XIS) sector. Examples include the use of direct methods for achieving sub-Ångstrom resolution maps of complex oxide interfaces, quantum-dot tailoring, and the prospects for *in situ*, real-time X-ray interface microscopy. Such experiments will allow us to understand how local structure can give rise to novel macroscopic properties. Ongoing developments in X-ray optics, pixel area detectors, and real-space and real-time techniques will converge to realize extremely interesting science opportunities at the new XIS facility.

Supported by DOE Basic Energy Sciences Contract DE-FG02-06ER46273 and the University of Michigan Energy Frontiers Research Center.

APS

X-ray Biogeochemistry: Elucidating Bacteria–Mineral–Contaminant Interactions at the Molecular Scale

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The selection of waste repository sites and the design of remediation strategies require a good understanding of radionuclide contaminant transport in subsurface environments. Macroscopic parameters of interest such as concentration profiles or propagation rates are ultimately controlled by the molecular interactions between the dissolved ions and the mineral and/or biological phases along the flow path. In addition to passive processes such as surface sorption, the solubility of contaminants is strongly influenced by redox transformations caused by metabolizing bacteria or their products. The interplay between biotic and abiotic factors creates a complex reaction network that is actively being studied with the goal of building a predictive computational model.

This presentation will review several important molecular-scale processes that control uranium solubility in the subsurface and present case studies where X-ray absorption fine structure (XAFS) spectroscopy and X-ray fluorescence (XRF) nano-spectroscopy at the Advanced Photon Source were instrumental in providing the insight necessary for understanding observed contaminant behavior. In one such study of uranium reduction by bacteria, XRF spectroscopy at submicron spatial resolution was able to show co-localization of uranium precipitates and biological material in the extracellular space, implicating outer-membrane proteins (cytochromes) in the extracellular reduction of uranium. In another study, X-ray absorption near-edge spectroscopy (XANES) at submicron spatial resolution was able to show heterogeneity in the valence state of uranium relative to the position of single bacteria on a thin iron oxide film. In a third study of U(VI) reduction, we show how a combination of biotic and abiotic experiments provides insight on mineral nucleation near organic surfaces and leads to the discovery of unexpected U(IV) phases that may have very different transport properties than the usually assumed uraninite phase. These studies highlight the importance of molecular scale information in understanding macroscale processes and the need for improvements in the techniques used to access this information.

Tuesday, May 4 — Afternoon, EMC

In situ Electron Microscopy and the Potential and Use of Aberration-corrected TEMs

EMC

Catalysis in an Aberration-free Environment: Atomic Resolution Studies of *in situ* Water Splitting

James Ciston[1], Yimei Zhu[1,2], Jose Rodriguez[3], and Christian Jooss[4]

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[2] Department of Condensed Matter Physics, Brookhaven National Laboratory, Upton, NY 11973, USA
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We have successfully imaged the Cu precipitates in $Ce_{1-x}Cu_xO_2$ during an H_2 annealing cycle. The $Ce_{1-x}Cu_xO_2$ system has immediate applications in the low temperature steam reforming of hydrogen from hydrocarbon fuels via the water-gas shift reaction ($CO + H_2O \rightarrow H_2 + CO_2$). These are the first results obtained from the FEI Titan environmental transmission electron microscope (TEM) at Brookhaven National Laboratory, which is capable of atomic resolution at pressures up to 10 torr and temperatures of up to 700°C. We have found the precipitates to be 10–15 nm in size, which is 5–10 times larger than anticipated. During the subsequent oxygen anneal, it was found that the Cu precipitates do not return to the bulk $Ce_{1-x}Cu_xO_2$ phase in solid solution as suggested by prior synchrotron diffraction and extended X-ray absorption fine structure (EXAFS) results, but instead form an amorphous oxide at the surface of the particles. We will also present *in situ* data from the $Pr_xCa_{1-x}MnO_3$ system in a water vapor environment, which is among the most active oxygen evolution catalysts for electrochemical water splitting. Using our *in situ* imaging capabilities, we have identified which regions of the catalyst particles are most active for oxygen evolution and have found that some phases exhibit almost no activity.

EMC

Are Dislocations Possible in Nanoparticles?

C.E. Carlton and P.J. Ferreira

Materials Science and Engineering, University of Texas at Austin, Austin, TX 78712, USA

The deformation behavior of nanoscale metals continues to be an exciting area for materials research. However, in the case of single-crystal 0D nanoscale metals, no deformation experiments, to our knowledge, have been performed at the nanoscale. The one experiment closest to the nanoscale was an *in situ* TEM compression of ~200 nm Si nanoparticles. However, the particle tested was too large to extract relevant information at the nanoscale and the mechanical deformation of Si is also expected to be different from that of metals. For nanoparticles it is claimed there is a conspicuous lack of dislocations, regardless of the materials processing history, even after significant deformation. Therefore, it has been suggested that dislocations cannot exist or do not play a role on the deformation of 0D nanomaterials.

To address this issue of the role played by dislocations in the deformation of 0D nanomaterials, nanoparticles with diameters less than 20 nm were compressed *in situ* under phase-contrast in a transmission electron microscope (TEM). Two phase-contrast TEM experiments were done, one in a conventional TEM and the other in an aberration-corrected TEM. Evidence for nucleation of dislocations and dislocation motion was observed during *in situ* TEM nanoindentation; upon unloading, however, dislocations were no longer visible. A new model for explaining dislocation instability is introduced. In this model we consider the change in Gibbs free energy of an edge dislocation as it moves through the nanoparticle towards the surface. The nanoindentation experiments seem to confirm the model proposed.

EMC

In situ Evaluation of the Factors Controlling Carbon Nanotube Synthesis

Renu Sharma

National Institute of Science and Technology, Gaithersburg, MD 20899-6203, USA

Carbon nanotubes (CNTs) are being investigated for a wide variety of applications ranging from biotechnology to nanotechnology. Tremendous progress has been made in understanding their formation, with a view toward large-scale synthesis and building CNT-based devices. Generally CNTs are synthesized by catalytic chemical vapor deposition (C-CVD) using transition metals such as Ni, Fe, and Co as the catalysts and hydrocarbons as the carbon source. During the last five years, *in situ* observations using environmental transmission microscopy (ETEM) has played a crucial role in revealing atomic level structural transformations occurring during their nucleation and growth. We have combined *in situ* and *ex situ* measurements to demonstrate the importance of temperature and precursor pressure in controlling the structure and morphology of the CNTs. We have also used the column of the ETEM for site-specific deposition of Fe catalyst particles and revealed the atomic-level structural transformations occurring during nucleation and growth of CNTs.

We have recently observed increased activity in tubular structure formation for samples with low amounts of Au (~20% nominal composition). Atomic resolution videos reveal that the graphene layers (the essential topological form of carbon for tubular growth) form at facets of the crystalline catalyst particles. Analysis (both *in situ* and *ex situ*) of the composition of the catalyst particles, the effect of temperature and pressure on the morphology, and the structural mechanism for the formation of various morphologies observed will be presented.

EMC

Step Flow and Interface Formation during the Growth of Heterojunction Nanowires

Frances M. Ross

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

Controlled fabrication of semiconductor nanowires that contain heterojunctions is the key to exciting electronic applications that make use of quantum dots and barriers. *In situ* transmission electron microscopy (TEM) provides a unique window into observing, and perhaps controlling, the formation of these complex nanostructures. We have therefore used *in situ* TEM to observe the growth of Si and Ge nanowires, and Si and Ge on III-V nanowires, through the vapor-liquid-solid and vapor-solid-solid mechanisms. Movies recorded during growth allow us to quantify wire nucleation, determine catalyst stability, measure nanowire surface structure and growth kinetics, and see the fascinating dynamic nature of the catalyst during heterostructure formation. We find that certain solid catalysts can be used to form compositionally abrupt Si/Ge and Si/SiGe interfaces, and we quantify the atomic-level step flow processes that take place during interface formation. We compare Si/Ge interfaces with group IV/III-V interfaces and discuss the implications of interface control for improved Si nanowire-based electronic devices.

EMC

Dynamic Transmission Electron Microscopy (DTEM)

N.D. Browning[1,2,3], M.A. Bonds[2], G.H. Campbell,[1] J.E. Evans,[1,3] K.L. Jungjohann[2], J. McKeown,[1] T.B. LaGrange[1], B.W. Reed[1], and M. Santala[1]

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[2] Department of Chemical Engineering and Materials Science, University of California-Davis, Davis, CA 95616, USA
[3] Department of Molecular and Cellular Biology, University of California-Davis, Davis, CA 95616, USA

A dynamic transmission electron microscope (DTEM) has been developed at Lawrence Livermore National Laboratory (LLNL) that permits dynamic phenomena in materials systems to be observed with both high spatial (~1 nm or better) and high temporal (~1 μs or faster) resolution. The high temporal resolution is achieved by using a short-pulse laser to create the pulse of electrons through photoemission. This pulse of electrons is propagated down the microscope column in the same way as in a conventional high-resolution TEM. To synchronize this pulse of electrons with a particular dynamic event, a second laser is used to "drive" the sample a defined time interval prior to the arrival of the laser pulse. An important aspect of this DTEM is that one pulse of electrons (a typical 10 ns pulse contains ~10^8 electrons) is used to form the whole image, allowing irreversible transitions and cumulative phenomena such as nucleation and growth to be studied directly in the microscope. In this presentation, a summary of the development of the DTEM will be described. The potential improvements in spatial and temporal resolution that can be expected through the implementation of upgrades to the lasers, electron optics, and detectors will also be discussed, along with the impact of new *in situ* gas and liquid stages to study catalysts and biological systems.

Development of the DTEM at LLNL was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under Contract DE-AC52-07NA27344. Development of in-situ stages for the DTEM at UC-Davis was supported by DOE NNSA-SSAA grant number DE-FG52-06NA26213 and NIH grant number RR025032-01.

EMC

Quantitative Nanomechanical Testing in a TEM

Andrew M. Minor[1,2]

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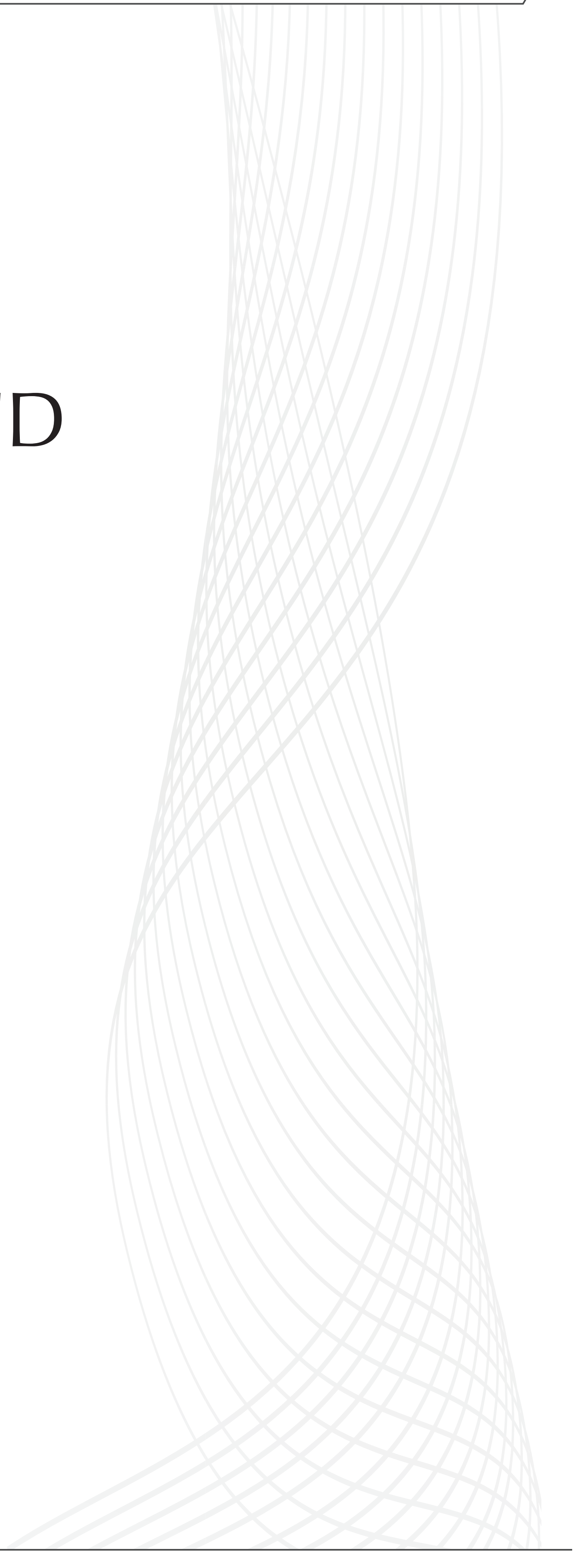
Recent progress in both *in situ* and *ex situ* small-scale mechanical testing methods has greatly improved our understanding of mechanical size effects in volumes from a few nanometers to a few microns. Besides the important results related to the effect of size on the strength of small structures, the ability to systematically measure the mechanical properties of small volumes through mechanical probing allows us to test samples that cannot easily be processed in bulk form, such as a specific grain boundary or a single crystal. In the case of individual nanostructures, the need to address the nanostructure in a direct manner is even more acute, and *in situ* TEM in many cases makes this possible. This talk will demonstrate how individual nanostructures and individual microstructural features can be tested quantitatively inside a TEM with different loading schemes such as indentation, compression, and tension. In addition, the promise of *in situ* nanomechanical testing in an aberration-free environment will also be addressed, with a discussion of the opportunities and challenges involving quantitative *in situ* mechanical testing at high resolution.

WORKSHOP **AGENDAS** AND **ABSTRACTS**



Workshop 1

Application of Advanced X-ray Techniques to Industrial Research

Building 402, Lecture Hall

Organizers: Steve Heald, Dean Haeffner, Jin Wang, Randall Winans
(APS, Argonne National Laboratory)

Synchrotron methods have long been applied to industrial problems. Foremost among these have been standard diffraction and XAFS methods. In recent years a number of more advanced methods have been developed that could have application to industrial problems. These include high speed imaging, X-ray Raman and other advanced spectroscopies, and new techniques using high energy X-rays. Such methods can be applied to such diverse problems as in-situ studies of batteries and catalysts, advanced materials for energy applications, and imaging of fuel sprays in internal combustion engines. These capabilities may not be as widely appreciated in the industrial community as the more standard techniques. This workshop will survey the current and future applications of some of these newer techniques. It will also look at how the APS upgrade could enhance opportunities for industrial research.

8:50 – 9:00	Steve Heald (APS, Argonne National Laboratory) *Introductions and logistics*
9:00 – 9:15	Dennis Mills (APS, Argonne National Laboratory) *Introduction to Industrial Research at the APS*
9:15 – 9:45	Shelly Kelly (EXAFS Analysis) *Resonant X-ray Emission Spectroscopy Measurements of Co-Containing Catalysts*
9:45 – 10:15	Douglas K. McCarty (Chevron ETC) *The Defective Structure of Excess-Ca Dolomite*
10:15 – 10:30	Coffee break
10:30 – 11:00	Angus Wilkinson (Georgia Institute of Technology) *Real-time X-ray Diffraction Studies of Oil Well Cement Hydration under Down Hole Conditions*
11:00 – 11:30	Brian Landes (Dow) In situ *and Time-resolved X-ray Scattering Studies: Bridging Molecular Architecture and Material Performance*
11:30 – 12:00	Joseph Ziegelbauer (GM) *Synchrotron-based Fuel Cell Research at General Motors*
12:00 – 1:30	Lunch

1:30 – 2:00	Conal Murray (IBM) *Peering under the Hood: Investigating Next-generation Microelectronic Materials*
2:00 – 2:30	Gaurav Jain (Medtronic, Inc.) *Building Batteries with Extended Longevity for Implantable Medical Devices*
2:30 – 3:00	Yan Gao (GE) *Application of Synchrotron Techniques to Materials Research at GE Global Research*
3:00 – 3:15	Coffee break
3:15 – 3:45	Scott Parrish (GM) *Application of Optical Diagnostics for Spray Characterization*
3:45 – 4:15	Trudy Bolin *S-XANES Analysis of Sulfur Forms in Coals and Kerogens*
4:15 – 4:30	Susan Strasser (APS, Argonne National Laboratory) *Access to the APS: General User Proposals, Rapid Access, Mail-in Powder Diffraction, and Proprietary Work*
4:30	Informal beamline tours and discussions with beamline scientists

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WK1

Resonant X-ray Emission Spectroscopy Measurements of Co-Containing Catalysts

Shelly D. Kelly[1], Simon R. Bare[2], and Gerald T. Seidler[3]

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Cobalt-molybdenum catalysts designed for the selective hydrotreating of fluid catalytic cracked (FCC) gasoline produce ultra-low sulfur gasoline by removing sulfur while minimizing octane loss and olefin saturation. Nonselective FCC gasoline hydrotreaters used in the past to remove sulfur also saturate olefins, thus creating paraffins, which have lower octane values. Hence, nonselective FCC gasoline hydrotreaters remove sulfur but also significantly reduce product octane values. Maximizing octane retention adds significant value to the FCC gasoline portion of the motor gasoline pool. Although Co-Mo hydrotreating catalysts have been studied for many years, much remains to be learned about the active catalytic site, and there is demand for better performing hydrotreating catalysts. Indeed, even the active site within this catalyst is still being debated. During the sulfidation of the catalyst, the X-ray absorption near edge structure (XANES) spectra of Co and Mo evolve from the oxide to the sulfided form. Untangling the spectra of the actively changing species from the spectra of the species that do not change (bulk oxide components) is difficult. We have used resonant X-ray emission spectroscopy (XES) to probe Co in these samples in the oxidized and sulfided state to elucidate the Co species that is evolving with activity. Our investigations have determined unique X-ray fluorescence shifts for the active and inactive phases, such that the spectrometer can be used to obtain information about each phase. By comparing samples during different stages of sulfidation and comparing catalysts with different activity, the mechanistic properties can be inferred with the help of many other microscopies and bulk chemistry measurements. A new X-ray spectrometer is uniquely suited to provide the Co XES spectra. We believe there is a wealth of information that many scientific groups could realize by using resonant XES at the Advanced Photon Source.

WK1

The Defective Structure of Excess-Ca Dolomite

Douglas K. McCarty
Chevron ETC, 3901 Briarpark, Houston, TX 77063, USA

Diffraction features observed for non-stoichiometric excess-Ca dolomite samples from Eocene rocks of the Floridan aquifer and from Permian rocks of New Mexico demonstrate the presence of defect structures. The X-ray diffraction patterns recorded using synchrotron and Cu Kα radiation contain two types of reflections in the form of sharp, narrow Bragg lines and diffuse maxima. The intensity of the Bragg reflections decrease more than expected with increasing 2θ angle, while the intensity of the diffuse maxima increases as a consequence of the structural modifications arising from compositional heterogeneity. The observed diffraction features are analyzed based on the theory of X-ray diffraction in nonideal crystals.

It is shown that both the Bragg and diffuse reflections belong to a single defective structure. It was assumed that the dolomite microcrystals in the samples contain structural distortions as a result of compositional heterogeneity in which high-excess-Ca dolomite microcrystals contain small domains of low excess Ca. These domains create long-range atomic displacements. A modified Rietveld method was used to refine the average structures of both the high-excess-Ca and low-excess-Ca components of these samples. It was found that the A sites in both structures are occupied by Ca, whereas the excess Ca is located in the B site. The refined interatomic distances for the average structure are consistent with site occupancies and ionic radii. The findings are related to previous transmission electron microscopy studies showing the existence of defective domains. A possible physical model of the defective structure of the studied excess-Ca dolomite samples is discussed.

Keywords: Dolomite, excess calcium dolomite, Rietveld refinement, crystal chemistry, X-ray diffraction

WK1

Real-time X-ray Diffraction Studies of Oil Well Cement Hydration under Down Hole Conditions

Angus P. Wilkinson[1], Andrew C. Jupe[1], and Gary P. Funkshouser[2]
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Portland cement is used in large quantities to “grout” oil wells. It is placed between the metal liner and the borehole walls to support the liner and prevent fluid migration outside the liner. The cement has to perform at high temperatures and pressures and in chemically aggressive environments. To develop a better understanding of the hydration of oil well cement, powder diffraction has been used to study both hydration pathways and hydration kinetics, in the presence and absence of additives, at pressures of up to ~1 kbar and temperatures of up to 200°C. Powder diffraction has also been combined with simultaneous ultrasound reflection measurements so that the reaction chemistry, observed by diffraction, can be correlated with the development of mechanical properties.

WK1

Synchrotron-based Fuel Cell Research at General Motors

Joseph M. Ziegelbauer

Electrochemical Energy Research Lab, General Motors Company, Warren, MI 48090, USA

Over the past decade, General Motors has pursued the goal of getting fuel-cell-based passenger vehicles on the roads. This goal is exemplified by the Project Driveway program: More than100 fuel-cell-powered Chevy Equinox crossovers have accumulated more than a million miles of real-life testing in select markets. However, to attain cost-competitiveness with modern internal-combustion-powered vehicles, the amount of precious metals in the cathode catalyst must be reduced while maintaining acceptable levels of performance and durability. GM employs a variety of methods to study next-generation materials, including high-resolution transmission electron microscopy, X-ray diffraction, electrochemistry, and so on. However, clear assessment of the advantages and disadvantages of new materials requires a technique that offers atomic resolution under *in situ* conditions. This presentation will provide an overview of how *in situ* X-ray absorption spectroscopy analysis of catalyst and support technologies contributes to these goals.

WK1

Peering Under the Hood: Investigating Next-generation Microelectronic Materials

Conal E. Murray

I.B.M. T.J. Watson Research Center, Yorktown Heights, NY 10598, USA

The drive toward smaller device dimensions and greater densities in high-performance CMOS (complementary metal–oxide–semiconductor) technology has led to the adoption of both novel designs and materials to achieve better performance. The challenges associated with incorporating these improvements present new opportunities for characterization of microelectronic technology, particularly at a submicron resolution. After a survey of existing techniques commonly employed in semiconductor manufacturing, examples will be given that demonstrate the needs for both highly localized and spatially averaged measurements. One such example involves strain engineering, which can improve carrier mobility but requires an understanding of the mechanical response of the device as a result of adjacent stressor structures. Synchrotron-based X-ray microbeam methods provide the capability to directly map the strain generated within silicon-on-insulator channels and their surrounding environment. A comparison of experimental data to mechanical modeling confirms that strain fields emanating from stressor features can extend large distances, leading to significant overlap in current designs. Another example is the detection of stress gradients generated at capping layer/metallization interfaces, where voiding phenomena such as electromigration can occur. *In situ* investigations of these buried interfaces can provide key information about conductor longevity and reliability.

WK1

Building Batteries with Extended Longevity for Implantable Medical Devices

Gaurav Jain

Medtronic, Inc., Brooklyn Center, MN 55430, USA

Implanted medical devices serve patients in a range of therapy areas, such as cardiac rhythm management, spinal cord stimulation for pain management, deep brain stimulation for movement disorders, and sacral nerve stimulation for bladder control. A persistent challenge facing this industry is the need to extend device longevity while offering smaller devices with more complex features. Focusing on batteries for implanted devices, this talk will present approaches for designing, testing, and modeling performance for device lifetimes exceeding a decade. An important part of this process is gaining a fundamental understanding of underlying ageing mechanisms. Specific examples will be cited from state-of-the-art primary and rechargeable battery technologies where mechanistic understanding has been crucial for advancements. Areas where use of advanced characterization techniques may be beneficial to meet the future goals of the industry will be discussed.

WK1

S-XANES Analysis of Sulfur Forms in Coals and Kerogens

Trudy Bolin

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

Sulfur X-ray near edge absorption spectroscopy (S-XANES) has emerged as a powerful tool for speciating sulfur forms in such complex carbonaceous solids as kerogen and coal. S-XANES is also used to collect information about chemical and thermal transformations of sulfur in these materials. This technique, used in conjunction with other techniques, such as X-ray photo-electron spectroscopy and ^{13}C NMR can be used to gain valuable information about sulfur transformations without altering the samples in the process [1]. S-XANES has been performedon 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 (eight) of coals for basic research [3]. Measurements of these standards have been re-performed at the Advanced Photon Source beamline 9-BM. This beamline is optimized for low-energy X-ray absorption fine structure (XAFS) spectroscopy (below 4 keV) and is unique to the APS facility. The data taken at APS has been compared to data taken in 1991 at the Stanford Synchrotron Radiation Light Source by M. Gorbaty et al. [2]. 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. Kelemen, S. R.; Afeworki, M.; Gorbaty, M. L.; Sansone, M.; Kwiatek, P. J.; Walters, C. C.; Freund, H.; Siskin, M.; Bence, A. E.; Curry, D. J.; Solum, M.; Pugmire, R. J.; Vandenbroucke, M.; Leblond, M,; Behar, F., Energy and Fuels, 2007, 21, 1548-1461.
2. George, G. N.; Gorbaty, M. L.; Kelemen, S. R.; Sansone, M, Energy and Fuels 1991 5, 93.
3. Vorres, K. S. et al., Energy Fuels, 1990, 4 (5), pp 420–426.

Workshop 2

Beamline 2.0: The Fully Integrated Instrument

Building 402, Rm. E1100/E1200

Organizers:	Lahsen Assoufid (APS, Argonne National Laboratory), Dean R. Haeffner (APS), Peter J. Eng (The University of Chicago), and John P. Quintana (APS)

The potential of an APS upgrade gives the community the opportunity to reexamine the paradigm on which beamlines are designed and integrated. The workshop will examine storage ring beamlines from the standpoint of complete integrated instruments from the particle beam through the final experimental setups. Future directions in state-of-the-art synchrotron radiation instrumentation technology will be explored. Speakers will present their individual topics within the framework of what is required of a completely integrated beamline instrument.

Session 1

8:30 – 9:00	Peter Eng (Center for Advanced Radiation Sources, The University of Chicago) *Beamline 2.0: The Fully Integrated Instrument*
9:00 – 9:30	Michael Borland (Accelerator Systems Division, Argonne National Laboratory) *Accelerator Considerations and the APS Upgrade*
9:30 – 10:00	Efim Gluskin (Accelerator Systems Division, Argonne National Laboratory) *Undulators at the APS: Current State and Future Plans*
10:00 – 10:30	Coffee Break

Session 2

10:30 – 11:00	Jean Vilain (Newport Corporation) *Fine, Fast, Coordinated Motion and Its Control*
11:00 – 11:30	Jon Kelly (Instrument Design Technology, UK) *Technical Challenges of Monochromator Design*
11:30 – 12:00	Michael Landry (California Institute of Technology) *LIGO: The Laser Interferometer Gravitational Wave Observatory*
12:00 – 1:30	Lunch

Session 3

1:30 – 2:00	Chris Jacobsen (X-ray Science Division, Argonne National Laboratory) *Integrated Microscopes: Challenges from Optics to Nanopositioning to Cryo*
2:00 – 2:30	Ivan Vartaniants (HASYLAB, Deutsches Elektronen-Synchrotron) *Coherence Properties of Hard X-ray Synchrotron Sources and X-ray Free Electron Lasers*

2:30 – 3:00 Lahsen Assoufid (X-ray Science Division, Argonne National Laboratory)
Optics and Metrology Development at the APS

3:00 – 3:30 Kazuto Yamauchi (Osaka University)
A Novel On-site Wavefront Correction Method for Ultimate Focusing of Hard X-Rays

3:30 – 4:00 Coffee Break

Session 4

4:00 – 4:30 Jean Susini (Instrumentation Services and Development Division, European Synchrotron Radiation Facility)
The ESRF Upgrade Programme: Challenges for Instrumentation

4:30 – 5:00 Nino Micelli (X-ray Science Division, Argonne National Laboratory)
Beyond the APS Detector Pool: Detector Systems Evaluation, Characterization, and Integration

5:00 – 5:30 Mark Rivers (Center for Advanced Radiation Sources, The University of Chicago)
High-Performance Data Acquisition and Beamline Control: Current Capabilities and Future Needs

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WK2

Accelerator Considerations and the APS Upgrade

Michael Borland

Accelerator Systems Division, Argonne National Laboratory, Argonne, IL 60439, USA

We review the properties of accelerators that are relevant to beamline design, in particular, emittance, lattice functions, and energy spread. We discuss the differences between ideal and nonideal conditions, as well as some special, limited-availability customizations. Properties of possible lattices for the APS upgrade are also reviewed.

WK2

Undulators at the APS: Current State and Future Plans

Efim Gluskin

Accelerator Systems Division, Argonne National Laboratory, Argonne, IL 60439, USA

Design criteria and tradeoffs for APS undulators will be presented including the impact on beamline designs. Current undulators will be reviewed and future plans discussed.

WK2

LIGO: The Laser Interferometer Gravitational Wave Observatory

M. Landry for the LIGO Scientific Collaboration

California Insitute of Technology, Pasadena, CA 91125, USA

The Laser Interferometer Gravitational Wave Observatory (LIGO) endeavours to detect gravitational waves from astrophysical sources. The observatory comprises three kilometer-scale interferometers at two distinct sites: one in Richland, Washington, the other in Livingston, Louisiana. In this talk we will give an overview the LIGO science goals and then describe some technologies that LIGO may share with light sources, such as the large-scale vacuum

enclosure; pristine optics and cleanliness requirements; the myriad feedback and feed-forward controls that maintain the detectors at their respective operating points; and issues originating in the physical environment, such as tides, earthquakes, and ever-present anthropogenic noise sources.

WK2

Integrated Microscopes: Challenges from Optics to Nanopositioning to Cryo

Chris Jacobsen

X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

So here you are, a poor graduate student trying to get high-resolution images and element maps from some cultured cells. You have to rapidly freeze your cells, check their quality, get them into an endstation, find the right region and then scan it at high precision, and hope that you have a reliably small focus on the sample. You have to collect fluorescence data and see that you don't have major elemental contaminants, and you have to collect ultrastructural data so you know where the metals are. And you're here to do this for the first time, yet you expect to leave at the end of the week with publishable data. Your only hope? To have an integrated microscope, designed as a whole rather than assembled by bolting together some micropositioning stages, designed in conjunction with a sample preparation and evaluation lab, and equipped with control software that does not present you with twenty EPICS devices.

WK2

Coherence Properties of Hard X-ray Synchrotron Sources and X-ray Free Electron Lasers

I.A. Vartaniants

Deutsches Electronen-Synchrotron (DESY), D-22607 Hamburg, Germany

A general theoretical approach based on the results of statistical optics is used for the analysis of the transverse coherence properties of third-generation hard X-ray synchrotron sources and X-ray free-electron lasers (XFELs). Correlation properties of the wavefields are calculated at different distances from an equivalent Gaussian Schell-model source. This model is used to describe coherence properties of a typical undulator source at the synchrotron storage ring. In the case of XFEL sources the decomposition of the statistical fields into a sum of independently propagating transverse modes is used for the analysis of the coherence properties of these new sources. A detailed calculation is performed for the parameters of a typical hard X-ray undulator planned at the European XFEL. It is demonstrated that only a few modes contribute significantly to the total radiation field of that source. Recent results on application of these coherent beams for coherent X-ray diffraction imaging will be demonstrated.

WK2

A Novel On-site Wavefront Correction Method for Ultimate Focusing of Hard X-Rays

Kazuto Yamauchi

Osaka University, Osaka 565-0871, Japan

We developed precision fabrication and measurement methods to realize nano-focusing mirror devices for synchrotron radiation hard X-rays [1]-[5]. The fabricated KB mirrors were tested at the 1-km-long beamline (BL29-XUL) of SPring-8 and confirmed to provide nearly diffraction-limited focusing with a spot size less than 30 nm for 15 keV X-rays [6].

In a recent research project, we achieved 7 nm focusing of 20 keV X-rays by using Pt/C multilayer mirrors with an on-site wavefront phase measurement and correction method [7]-[10], [12]. The on-site phase-error measurement is based on a phase-retrieval method using precisely measured intensity profiles near the beam waist. A bendable mirror is placed upstream of the focusing mirror to compensate for the wavefront error due to the imperfection of the focusing devices. Details of the method [11] will be presented with the latest results achieved in collaborative research with RIKEN and JASRI of SPring-8.

This research was partially supported by Grant-in-Aid for the Specially Promoted Research (No. 18002009), "Promotion of X-ray Free Electron Laser Research" and the Global COE Program "Center of Excellence for Atomically Controlled Fabrication Technology" from the Ministry of Education, Culture, Sports, Science and Technology (MEXT).

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WK2

The ESRF Upgrade Programme: Challenges for Instrumentation

Jean Susini

Instrumentation Services and Development Division, European Synchrotron Radiation Facility, 38043 Grenoble, France

As at any other synchrotron facility, instrumentation and the associated enabling technologies underpin every aspect of scientific activity at the ESRF. With the Upgrade Programme already well underway, it was recognized that the overall performance of future synchrotron beam lines depends critically on innovative and successful X-ray instrumentation R&D programs. Furthermore, ensuring optimum designs for beam lines implies taking an integral approach to their specification, design, and implementation. A new multidisciplinary strategy is needed when striving to achieve these goals.

It was also recognized early on that our organizational structure was not optimal for facing the many challenges inherent to our beam line modernization objectives. This recognition led to the creation of the Instrumentation Services and Development Division in 2009. This division brings together all in-house experts to create synergies between the various engineering areas involved in tackling new, multidisciplinary projects of integrated instrumentation.

After a brief overview of the different projects of the Upgrade Programme, some instrumentation challenges will be described, followed by discussion of how we propose to address these issues.

WK2

Beyond the APS Detector Pool: Detector Systems Evaluation, Characterization, and Integration

Antonino Miceli

X-ray Sciences Division, Argonne National Laboratory, Argonne, IL 60439, USA

The APS Detector Pool began operation in April 2003. It has grown from a library of detectors into a centralized knowledge base about detectors for the APS community. In this talk, we will discuss plans to the expand the scope of the Detector Pool beyond simply providing commercial detectors to the beamlines, including the expansion of the exploration of new detector technologies from industry to meet the needs of the APS community. We will discuss the evolution of the Detector Pool towards a systems-level group that works closely with the beamlines to systematically analyze, characterize, and calibrate various detectors to improve data quality and throughput. In addition, we will discuss detector-related activities planned for the Optics and Detectors Beamline at 6-BM.

WK2

High-Performance Data Acquisition and Beamline Control: Current Capabilities and Future Needs

M.L. Rivers

Center for Advanced Radiation Sources, University of Chicago, Argonne, IL 60439, USA

Operating synchrotron beamlines efficiently demands minimizing the overheads and delays during data acquisition. Modern detectors are capable of very high data collection rates but are often run at lower rates than the beamline X-ray photon flux would allow because of inappropriate data collection schemes. As X-ray sources, beamline optics, and detectors continue to improve, this mismatch will only get worse.

This talk will discuss current solutions to on-the-fly data collection that minimize the overheads, so that data collection speeds are limited only by the X-ray flux. These solutions include complex coordinated motions and continuous detector acquisition. Examples from X-ray diffraction with the Pilatus detector and X-ray spectroscopy with the XIA xMAP will be presented. With the Pilatus, we are currently able to collect 200 frames per second while moving a 6-circle diffractometer in a complex nonlinear trajectory. With the XIA xMAP, we can stream 4,000 spectra per second from a quad silicon drift diode detector (1,000 pixels per second) with collection coordinated with sample stage motion. Both of these techniques fundamentally change the type of experiments that can be performed.

In the future, an integrated beamline will require even tighter and more complex coordination than is currently possible. For example, continuous data acquisition will need to accommodate continuous motion of the undulator gap, monochromator, and sample. The hardware and software requirements for such experiments will be discussed.

Workshop 3

Linking Structure and Magnetism in Novel Materials: Future Opportunities at an Upgraded APS

Building 440, Rm. 105/106 (Center for Nanoscale Materials)

Organizers: J.W. Freeland (APS, Argonne National Laboratory) and A. Enders (University of Nebraska)

Magnetism is a fundamental property that underpins much of our basic understanding of the functionality of many systems. Since magnetism arises from cooperative interactions between atoms, it is not only dependent on the electronic state of a local atom but also how it is connected to the rest of the system (i.e., its structure). Understanding this interrelationship is key to unraveling forefront questions in magnetism, such as how magnetic order evolves with reduced dimensionality and how it responds to external stimuli. Of equal importance is the interrelationship with other properties (e.g., metallicity, superconductivity, ferroelectricity, and so on) and how we can rationally control the state of a material through its structural, electronic, and magnetic interactions. Synchrotron radiation provides a unique suite of tools (spectroscopy and diffraction) that are crucial to our understanding of this link between structure and magnetism. This workshop will focus on how the planned upgrade of Advanced Photon Source will allow us to go well beyond our current capabilities by enabling new types of measurement. Topics of interest are controlling magnetism with external stimuli (pressure, light, magnetic fields), the evolution of the magnetic state with dimensionality (i.e., nanomagnetism), and probing structural response to changes in magnetic state.

8:45 – 9:00	John Freeland (APS, Argonne National Laboratory) and Axel Enders (University of Nebraska) *Welcome*
9:00 – 9:35	Jacques Chakhalian (University of Arkansas) *Strongly Interacting Electrons at Complex Oxide Interfaces: From Growth to Physics*
9:35 – 10:10	Chris Palmstrøm (University of California at Santa Barbara) *Epitaxial Magnetic Metal/Semiconductor Heterostructure Interfaces*
10:10 – 10:30	Coffee Break
10:30 – 11:05	Tiffany Santos (Center for Nanoscale Materials, Argonne National Laboratory) *Modulation Doping of Ferromagnetism in* $LaMnO_3/SrMnO_3$ *Superlattices by Digital Synthesis*
11:05 – 11:40	James M. Rondinelli (University of California at Santa Barbara) *Magnetostructural Coupling in Oxides: Insights from First Principles*
11:40 – 12:15	Jan Honolka (Max Planck Institute for Solid State Physics, Stuttgart) *Tuning Magnetic Properties through Coordination: From 2D Metal-Organic Networks to Metal Nanostructures*
12:15 – 1:30	Lunch

1:30 – 2:05	John Schlueter (Argonne National Laboratory) *Correlating Structure and Magnetism in Coordination Polymers under Extreme Conditions of Pressure and Magnetic Field*
2:05 – 2:40	Kevin Moore (Lawrence Livermore National Laboratory) *Understanding Magnetic Stabilization of Metallic Crystals through the Actinide Element Curium*
2:40 – 3:15	Manuel Angst (Institut für Festkörperforschung, JCNS, and JARA-FIT Forschungszentrum Jülich, Germany) *Interplay of Spin, Charge, and Structural Degrees of Freedom in Magnetoelectric* $LuFe_2O_4$
3:15 – 3:45	Coffee Break
3:45 – 4:20	Jacob P. C. Ruff (Department of Physics, McMaster University) *Magnetoelastics of a Spin Liquid: X-ray Single-crystal Diffraction Studies of* $Tb_2Ti_2O_7$ *in Pulsed Magnetic Fields*
4:20 – 4:55	Jiun-Haw Chu (Stanford University) *Evidence for an Electron Nematic Phase Transition in Underdoped Iron Pnictide Superconductors*
4:55 – 5:30	Wrap-up/Discussion

WK3

Strongly Interacting Electrons at Complex Oxide Interfaces: From Growth to Physics

Jacques Chakhalian
Physics Department, University of Arkansas, Fayettetville, AR 72701, USA

Complex oxides are a class of materials containing a variety of competing strong interactions that create a subtle balance to define the lowest energy state, which leads to a wide variety of interesting properties (e.g., superconductivity, magnetism, and so on). By utilizing the bulk properties of these materials as a starting point, interfaces between different classes of oxides offer an opportunity to break the symmetry present in the bulk and alter the local environment. Utilizing advances in oxide growth, one can now combine materials with distinctly different and even competing orders to create new materials in the form of heterostructures. The broken symmetry, strain, and altered environments at the interfaces then provide an avenue to manipulate this subtle balance and perhaps even create new phases.

The next big step in understanding these fascinating phases, however, requires detailed studies of the heterostructure properties *in situ* and often during growth. Here I will touch on several recent examples to illustrate how a powerful combination of X-ray probes and advanced growth techniques offer the ability to probe interface properties to gain unique insight into the underlying physics.

WK3

Epitaxial Magnetic Metal/Semiconductor Heterostructure Interfaces

Q.O. Hu[1], M. Hashimoto[1], J.M. LeBeau[2], E. S. Garlid[3], B.D. Schultz[1], S. Stemmer[3], P. A. Crowell[3], and C.J. Palmstrøm[1,2]

[1] Electrical and Computer Engineering, University of California, Santa Barbara, CA 93106, USA
[2] Materials, University of California, Santa Barbara, CA 93106, USA
[3] School of Physics and Astronomy, University of Minnesota, Minneapolis, MN 55455, USA

The spin-dependent transport across ferromagnet/semiconductor interfaces depends critically on their structure and electronic properties. Interfacial reactions, the formation of nonmagnetic interlayers, and conductivity mismatch have been attributed to a low efficiency of spin injection. A range of ferromagnetic elemental and metallic compound/compound semiconductor tunneling contacts for spin injection have been developed. The contacts have been characterized by a variety of methods: *In situ* methods included scanning tunneling microscopy (STEM), X-ray photo-electron spectroscopy (XPS), reflection high-energy electron diffraction (RHEED), and low-energy electron diffraction (LEED); *ex situ* methods included X-ray diffraction (XRD), Rutherford backscattering spectrometry (RBS), high angle annular dark field STEM (STEM-HAADF), magnetotransport, and magnetic characterization combined with molecular beam expitaxy (MBE) growth.

In the case of Fe_3Ga/GaAs(001) interfaces, the interface reconstruction was found to depend on the GaAs(001) surface reconstruction and the Fe_3Ga growth conditions. The efficiency of the spin-polarized current injected from the ferromagnetic contact has been determined by measuring the electroluminescence polarization of the light emitted from $Ga_{1-x}Al_xAs$ light-emitting diodes as a function of applied magnetic field and temperature. Interfacial reactions during MBE growth and post-growth anneal, as well as the band structure of the semiconductor device, were found to have a dramatic influence on the measured spin injection, including sign reversal. Lateral spin-transport devices with an epitaxial ferromagnetic metal source and drain tunnel barrier contacts have been fabricated. These devices have allowed demonstration of electrical detection and the bias dependence of spin-polarized electron injection and accumulation at the contacts.

This presentation will emphasize the progress in the understanding of the atomic-scale structure of epitaxial magnetic metal/III-V semiconductor heterostructures interfaces and the influence this structure on the magnetic and spin transport properties.

WK3

Modulation Doping of Ferromagnetism in $LaMnO_3/SrMnO_3$ Superlattices by Digital Synthesis

Tiffany Santos

Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439, USA

The perovskite manganite $La_{1-x}Sr_xMnO_3$ has a rich magnetic, structural, and electronic phase diagram, exhibiting ferromagnetism for La-rich compositions and antiferromagnetism for Sr-rich compositions. The ferromagnetic/antiferromagnetic phase transition occurs at x=0.5 and is accompanied by a structural transition and change in orbital occupancy. We have investigated the role of cation order and lattice strain in nucleating the ferromagnetic or antiferromagnetic state. Using ozone-assisted molecular beam epitaxy, we have synthesized fully epitaxial superlattices of $LaMnO_3$ and $SrMnO_3$, designed to be equivalent in composition to random alloys of $La_{1-x}Sr_xMnO_3$ in the vicinity of x=0.5. In our digital synthesis method, whereby we interleave single unit-cell layers of $LaMnO_3$ and $SrMnO_3$ layers, we are able to tune between the ferromagnetic and antiferromagnetic metallic states by inserting an extra $LaMnO_3$ or $SrMnO_3$ layer, respectively.

By using neutron diffraction, we have verified the A-type antiferromagnetic spin structure and measured a Néel temperature of 300K, which is an enormous enhancement of 160K over the value for bulk (unstrained crystal) $La_{0.5}Sr_{0.5}MnO_3$. However, this material is on the verge of ferromagnetism. We find that inserting an additional single unit-cell layer of $LaMnO_3$ into the superlattice, and thereby delta-doping a layer of electrons, causes a significant increase of the net magnetic moment while still retaining the A-type spin structure. Our polarized neutron reflectometry experiments revealed a highly modulated moment commensurate with the structural periodicity of the superlattices, with higher moment in the region of the extra $LaMnO_3$ layer. Thus, introducing a single La dopant layer results in a localized enhancement of ferromagnetic double exchange along the c-axis and a canted moment in an otherwise antiferromagnetic structure. The polarized neutron reflectometry measurements determined the length scale over which these delta-doped charges extend normal to the interfaces.

WK3

Magnetostructural Coupling in Oxides: Insights from First Principles

James M. Rondinelli and Nicola A. Spaldin

Materials Department, University of California, Santa Barbara, CA 93106-5050, USA

Perovskite-structured transition metal oxides with ABO_3 stoichiometry display a myriad of useful phenomena, such as metal-insulator transitions, ferroelectricity, and superconductivity. These properties are due to their correlated electrons—the behavior of an electron depends explicitly on the location of all the others—and structural diversity. All oxides in this class, however, share a common flexible corner-connected oxygen octahedral network which makes their structural phase transitions dependent on temperature, pressure, and epitaxial strain. As a result of such structural changes, the allowed coupling between the magnetic and lattice degrees of freedom is determined from the modified crystal symmetry; the strength of the coupling is related to the angular modifications in the exchange pathways among the transitional metal oxygen bonds. In this talk, I illustrate how magnetostructural coupling in bulk perovskite oxides and heterostructures offers a new landscape for tailoring functionality. I describe how we combine first-principles (parameter-free) density functional theory (DFT) calculations with group theoretical tools and crystal chemistry to identify microscopic spin–lattice couplings by rational design. I conclude by outlining some open questions for engineering magnetostructural couplings and how high-resolution experimental probes could assist in identifying new control routes for these interactions.

WK3

Tuning Magnetic Properties through Coordination: From 2D Metal-Organic Networks to Metal Nanostructures

Jan Honolka

Max Planck Institute for Solid State Research, 70569 Stuttgart, Germany

X-ray magnetic circular dichroism (XMCD) spectroscopy is a powerful technique to examine magnetic nanostructures on surfaces. It gives element-specific, quantitative values of spin and orbital magnetic moments and their anisotropies. Using this technique we demonstrate the effect of local coordination on magnetic properties for very different systems, such as 2D metal-organic networks on surfaces, monolayer surface alloys, and quasifree metallic clusters on decoupling xenon matrices.

Metal-organic networks where single 3*d* metal atoms are forced into a 2D regular array by molecule spacers mark the limit of non-ferromagnetic systems where the magnetic properties of each magnetic center is driven by ligand field effects [1]. Lateral coordination with the molecular ligands yields unsaturated yet stable coordination bonds, which enable chemical modification of the electronic and magnetic properties of the 3*d* atoms independently from the substrate. The easy magnetization direction of the 3*d* center can be switched by oxygen adsorption, thus opening a way to control the magnetic anisotropy in supramolecular layers akin to that used in metallic thin films.

Bringing 3*d* transition metals in close proximity to heavy 4*d*/5*d* elements is another route to tune magnetic properties. The heavy elements introduce strong spin-orbit coupling effects to the systems under study, which—depending on the atomic coordination—can lead to exceptionally large magnetic anisotropies, e.g., in atomically thin $Fe_{50}Pt_{50}$ surface alloys grown on Pt(997) [2], but also to complex Dzyaloshinskii-Moriya driven magnetic phases. Furthermore, 4*d*/5*d* element systems on their own are of fundamental interest because of their borderline magnetic behavior in the smallest cluster size regime, where ferromagnetism is observed in the quasifree limit and orbital and spin moments are determined by finite size effects.

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WK3

Correlating Structure and Magnetism in Coordination Polymers under Extreme Conditions of Pressure and Magnetic Field

John A. Schlueter

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

The reversible control of magnetic properties through the application of an external stimulus presents a contemporary challenge in materials research. Crystalline magnetic coordination polymers are attractive because the low energy scale of their spin exchange interactions and the “softness” of their crystal structures enable one to employ stimuli such as pressure or magnetic field to control their physical and structural properties. In this regard, we have chosen to study hydrogen-bonded systems. In the $[Cu(pyz)_2(HF_2)]BF_4$ (pyz = pyrazine) system, we have identified a magnetoelastic response in which the pyz rings distort in the presence of large magnetic fields. We have also studied the related $CuF_2(H_2O)_2(pyz)$ system under the influence of pressure. Here we find that the Jahn-Teller axis of the $CuF_2O_2N_2$ octahedra switches reversibly from the N-Cu-N ($P < 0.9$ GPa) to the O-Cu-O ($0.9 \text{ GPa} < P < 3.1$ GPa) to the F-Cu-F ($P > 3.1$ GPa) bonds; that is, the magnetic orbital plane switches from the CuF_2O_2 to the CuF_2N_2 to the CuO_2N_2 plane. This sequential orbital reordering induced by pressure is unprecedented and leads to a profound change in the magnetic properties: The magnetic structure changes from two-dimensional to one-dimensional above 0.9 GPa. Prospects for understanding these complex materials through advanced X-ray techniques will be discussed.

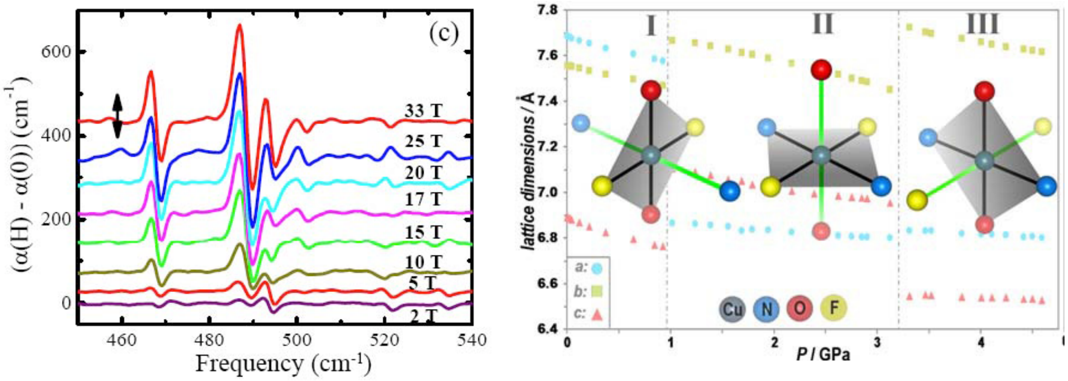

Figure. In the $[Cu(pyz)_2(HF_2)]BF_4$ system, exposure to magnetic fields lead to a distortion of the molecular pyz linkages (left). The $CuF_2(pyrazine)(H_2O)_2$ coordination polymer shows a predictable sequential reorientation of the electronic orbitals under applied pressure (right).

WK3

Understanding Magnetic Stabilization of Metallic Crystals through the Actinide Element Curium

Kevin Moore

Lawrence Livermore National Laboratory, Livermore, CA 94550, USA

The atomic crystal structure of some metals is a result of the element's own intrinsic magnetism: that is, spin and/or orbital polarization of bonding electrons dictates atomic geometry. The actinide metal curium (Cm) was recently shown to have a crystal structure stabilized by magnetism (Heathmen et al., Science 2005). Using electron energy-loss spectroscopy in a transmission electron microscope, synchrotron-radiation-based X-ray absorption spectroscopy, and many-electron spectral simulations, we explain why this occurs. We then use this understanding combined with density-functional theory to show why magnetic stabilization of metallic crystal structures occurs throughout the periodic table. Future research will be discussed that entails high pressure and X-ray magnetic circular dichroism measurements using the actinide $M_{4,5}$ edge. Such work will reveal new insight into the delocalized–localized transition of the 5f electronic states that occurs near Pu in the actinide series and illuminate how the transition influences crystal structure.

WK3

Interplay of Spin, Charge, and Structural Degrees of Freedom in Magnetoelectric $LuFe_2O_4$

Manuel Angst

Institut für Festkörperforschung, JCNS, and JARA-FIT Forschungszentrum Jülich, 52425 Jülich, Germany

Recently, $LuFe_2O_4$ has been attracting attention as a potential multiferroic with the novel mechanism of ferroelectricity due to charge order and significant magnetoelectric coupling. Extraordinarily large magnetic coercivity also contributed to interest in this material. On a microscopic level, both magnetoelectric coupling and the large coercivity are connected to a complex interplay of spin, charge, and structural degrees of freedom. The elucidation of this interplay primarily through diffraction experiments will be presented. Combining macroscopic magnetization measurements with X-ray resonant magnetic scattering and neutron diffraction, a complex magnetic phase diagram, including partially disordered magnetic phases, is established. To relate the different magnetic phases with structural and charge-orbital degrees of freedom, supplementary measurements of nonresonant X-ray diffraction and resonant X-ray diffraction with polarization analysis were performed. One of the phase transitions involves spin and charge as well as structural degrees of freedom, the latter being the origin of the large coercivity and other unusual magnetic features. Other transitions involve primarily one degree of freedom, with only subtle coupling effects to the other degrees of freedom being observed. The relation of these effects to the observed magnetoelectric coupling will be discussed.

WK3

Magnetoelastics of a Spin Liquid: X-ray Single-crystal Diffraction Studies of $Tb_2Ti_2O_7$ in Pulsed Magnetic Fields

Jacob P. C. Ruff

Department of Physics, McMaster University, Hamilton, ON L8S 4M1, Canada

High-resolution single-crystal X-ray diffraction measurements of the frustrated pyrochlore magnet $Tb_2Ti_2O_7$ have been performed using a novel low-temperature pulsed magnet system. This unique instrument allows a thorough characterization of structural degrees of freedom to temperatures as low as 4.4K and in applied magnetic fields as high as 30 Tesla. We show that $Tb_2Ti_2O_7$ manifests several intriguing structural effects under the application of magnetic fields, including strongly anisotropic giant magnetostriction, a restoration of local symmetry in low magnetic fields, and ultimately a structural phase transition in high magnetic fields. A treatment of spin-liquid physics in this compound based on spin degrees of freedom alone seems simplistic, as these results show that magnetoelastic coupling plays a signicant role in $Tb_2Ti_2O_7$ at low temperatures.

WK3

Evidence for an Electron Nematic Phase Transition in Underdoped Iron Pnictide Superconductors

Jiun-Haw Chu

Stanford University, Stanford, CA 94305-4045, USA

High-temperature superconductivity often emerges in the proximity of a symmetry-breaking ground state in strongly interacting electronic materials. In the case of the superconducting iron pnictides, in addition to the antiferromagnetic ground state of the parent compounds, a ubiquitous but small structural distortion breaks the crystal's C4 rotational symmetry in the underdoped part of the phase diagram. It has been proposed that this structural transition is driven by an electronic nematic order, in which the electronic system spontaneously breaks a rotational symmetry of the original Hamiltonian. Similar ideas have previously been suggested for certain underdoped cuprates, for $Sr_2Ru_3O_7$, and some specific fractional quantum Hall states. Recently, through magnetotrasnport and direct optical imaging, we have found that in-plane magnetic fields can be used to move the twin boundaries associated with structure domains in single crystals of underdoped $Ba(Fe_{1-x}Co_x)_2As_2$. In addition, by applying small amount of uniaxial pressure, we have aligned most of the twin domains in the samples, which enables us to probe the intrinsic electronic anisotropy of the symmetry breaking phase. Our observation hints at a strong magnetoelastic coupling and highly anisotropic electronic ground state in this class of materials.

Workshop 4

Science Opportunities with an X-ray Free Electron Laser Oscillator

Bldg. 401, Rm. A1100, morning

Organizers: K.-J. Kim and Yu. Shvyd'ko (APS, Argonne National Laboratory)

An X-ray free electron laser oscillator (XFELO) is a next-generation hard X-ray source, which will produce fully coherent X-ray beams of record spectral purity and average brightness in the photon energy range from about 5 to 25 keV. The characteristics of an XFELO are complementary to those of self-amplified spontaneous emission (SASE) and are briefly summarized below. The goal of the workshop is to explore the scientific opportunities that such an X-ray source could open up and thus to evaluate an XFELO as a future light source complementary to the upgraded APS.

XFELO Characteristics

- Photon energy coverage from 5 keV to 25 keV (and third harmonics)
- Tunable photon energy (5%)
- Fully coherent transversely and temporally
- High spectral purity with ~1 meV bandwidth
- Length of individual x-ray pulse ~0.1-1 ps
- Number of photons ~109 per pulse (~106–104 for third harmonics)
- Peak brightness comparable to that of SASE XFELs
- Pulse repetition rate ~1 MHz
- Time-averaged brightness is five orders of magnitude higher than that of the LCLS and three orders of magnitude higher than that of the European XFEL.

The organizers gratefully acknowledge the following sponsors: Blake Industries, Inc., and KOHZU Precision Co., Ltd.

Session 1 — Chair, Yuri Shvyd'ko

8:30 – 8:45	Kwang-Je Kim *Introduction to XFELO*
8:45 – 9:10	Paul G. Evans (University of Wisconsin-Madison) *Opportunities for Materials Science using a Fully Coherent Hard X-ray Laser*
9:10 – 9:35	Ralf Röhlsberger (Deutsches Elektronen-Synchrotron, Germany) *Perspectives for Nuclear Resonant Scattering and Spectroscopy with an XFELO*
9:35 – 10:00	Laurence Lurio (Northern Illinois University) *Prospects for New Science using X-ray Photon Correlation Spectroscopy at an XFELO*
10:00 – 10:25	Speaker to be determined *Topic: Hard x-ray photoemission spectroscopy*
10:25 – 10:45	Coffee Break

Session 2 — Chair, Kwang-Je Kim

10:45 – 11:15 Chris Jacobsen (APS, Argonne National Laboratory)
XFELO Microscopy: Exploiting Repeating Coherent Beams

11:15 – 11:40 Clement Burns (Western Michigan University)
Scientific Opportunities for High-Resolution Inelastic X-ray Scattering with an XFELO Source

11:40 – 12:05 Shin-ichi Adachi (Photon Factory, Japan)
Watching Photo-induced Dynamics with Ultrafast X-ray Structural Analysis

12:05 – 12:30 Panel discussion — Discussion leader, Linda Young (APS, Argonne National Laboratory)

Workshop 5

Biomolecular Assemblies as Materials Synthesis Templates: From Molecular Fundamentals to Energy and Environmental Sustainability Applications

Building 401, Rm. A5000, morning

Organizers: Hyunmin Yi (Tufts University), and Qian Wang (University of South Carolina)

This workshop will focus on studies in harnessing the exquisite structural and chemical features of nanobiological template materials ranging from peptide assemblies to viruses for facile synthesis of functional materials. In particular, research endeavors aimed at gaining fundamental understanding in molecular and nanoscale structures as well as emerging technologies for applications in catalytic, energy, and environmental sustainability applications will be presented.

The topic of biotemplated inorganic materials synthesis has attracted significant attention in the last decade because it offers the potential to exploit properties nature has already provided to biologically derived materials and interactions. In particular, viral and peptide assemblies have recently been employed in a variety of studies for precise control over dimensions, structures, and assembly features. Such materials also have the potential to be genetically engineered to confer and manipulate properties such as chemical functionality and size. For example, tubular (tobacco mosaic virus), filamentous (M13 bacteriophage), and icosahedral (cowpea mosaic virus) viruses have been enlisted for metal nanoparticle synthesis toward nanoelectronics, catalysis, and battery applications. Importantly, X-ray scattering techniques are emerging as versatile tools to examine delicate and dynamic nanoscale phenomena that cannot be accessed via traditional techniques. This workshop will bring together experts working on major issues in the field and applications of X-ray techniques.

Session 1 — Chair, Hyunmin Yi

8:40 – 8:45	Welcome: Hyunmin Yi
8:45 – 9:20	Gerard Wong (Department of Bioengineering, University of California, Los Angeles) *How to Grow Different Crystal Structures using the Same Virus*
9:20 – 9:55	Millicent A. Firestone (Materials Science Division, Argonne National Laboratory) *Dual Ionic and Electronic Transport in Biologically Inspired Polymers*
9:55 – 10:30	Qian Wang (Department of Chemistry and Biochemistry, University of South Carolina) *Controlled Assembly of Viral Nanoparticles for Biomaterials Development*
10:30 – 10:45	Coffee Break

Session 2 — Chair, Qian Wang

10:45 – 11:10 Hyunmin Yi (Department of Chemical and Biological Engineering, Tufts University)
Viral Templated Palladium Nanocatalysis

11:10 – 11:45 James N. Culver (Institute for Bioscience and Biotechnology Research & Department of Plant Science and Landscape Architecture, University of Maryland)
An Infectious Approach to Biofabrication

11:45 – 12:20 Matthew B. Francis (Department of Chemistry, University of California, Berkeley)
Using Synthetically Modified Proteins to Build Integrated Photocatalytic Systems

12:20 – 12:25 Closing Remarks and Announcements: Hyunmin Yi and Qian Wang

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WK5

How to Grow Different Crystal Structures using the Same Virus

Gerard C. L. Wong

Department of Bioengineering, University of California, Los Angeles, Los Angeles, CA 90095-1600, USA

We explore how one might grow a crystal structure the way one folds a protein. Not only are proteins chemically heterogeneous, they interact via a hierarchy of interactions (electrostatic, hydrophobic, H-bonding, chaperones, and so on) at different time scales. It is currently believed that this multiplicity of interactions leads to the famous funnel-shaped "free-energy landscape" with many local free-energy minima that correspond to a large number of structural states. Being able to rationally and reproducibly navigate through the free-energy funnel will open the possibility of making a broad range of new structures. FCC is the global free-energy minimized crystal structure for ensembles of essentially all spherical objects that interact via isotopic potentials. However, by using bacteriophage viruses (MS2 and Qbeta, ~28 nm diameter) interacting via two prototypical principles of attraction, we can make this free-energy minimum almost irrelevant. Not only can we reproducibly grow face-centered cubic and hexagonal close-packed structures, we can also grow tetragonal-centered structures and unanticipated layered phases. We investigate these systems using a combination of high-resolution synchrotron X-ray scattering and molecular dynamics simulations.

WK5

Dual Ionic and Electronic Transport in Biologically Inspired Polymers

Millicent A. Firestone[1], Sungwon Lee[1], Gregory Becht,[1] Byeongdu Lee[2], and Soenke Seifert[2]

[1]Materials Science and [2]X-ray Sciences Divisions, Argonne National Laboratory, Argonne, IL 60439, USA

Membrane proteins give rise to some of Nature's most intricate and important processes, ranging from metabolism to energy transduction. The ability to harness this evolutionarily optimized functionality outside the cell could lead to the development of protein-based systems useful for advancing alternative or renewable energy storage or production. The future of protein-based materials (and ultimately devices), however, requires the further development of a suitable supporting matrix that will preserve, order/orient, and control the activity of the proteins. A family of responsive soft nanostructures based upon polymerized ionic liquids (poly(ILs)) has been developed. These nanostructured chemical (polymeric) gels are formed by the spontaneous organization of a binary mixture of water and polymerizable analogues of *N*-alkylimidazolium-based ionic liquids. Photopolymerization of the preassembled IL monomers results in the formation of ductile, mechanically robust gels that have the ability to spatially localize guests within defined compartments in the anisotropic matrix. Many of the polymer architectures resemble those found in cellular environments, featuring amphiphilic bilayers separated by water channels, thus offering the possibility of

sequestering both lipophilic (e.g., membrane proteins) and water-soluble guests into the segregated domains. We have worked to incorporate components that will facilitate the interfacing and coupling of protein output (light-generated electron flow) to traditional device architectures. Specifically, we have modified these polymers in two ways so as to promote electrical communication between the encapsulated proteins and an external circuit. In the first approach, a hybrid material that organizes Au nanoparticles into columns within a hexagonally perforated, lamellar-structured poly(IL) was shown to behave as a conduction pathway or conduit. In the second approach, a thiophene moiety was incorporated into the IL monomer, yielding a nanostructured, electrically conductive polymer. Efforts are underway to examine approaches for the co-integration and electrical interfacing of the RCs in these materials.

This work was supported by the Office of Basic Energy Sciences, Division of Materials Sciences, United States Department of Energy under Contract No. DE-AC02-06CH11357 to the UChicago, LLC

WK5

Controlled Assembly of Viral Nanoparticles for Biomaterials Development

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Alignment of nanoparticle building blocks into ordered hierarchical structures is currently of key importance in colloidal and materials chemistry. Self assembly is the most effective way to build up sophisticated structures at the nano- and micrometer scale. In our study, we chose viruses and other biological particles as natural building blocks that can be tailored chemically and genetically. Compared with synthetic nanoparticles, the uniform shape and size of viruses provide highly promising possibilities for the self-assembly of nanoscale materials with hierarchical ordering. In particular, three different assembly methods have been developed and utilized in our research: interfacial assembly, convective assembly, and controlled assembly at the three-phase contact line. By using these methods, spherical and rod-like viruses can be assembled into hierarchically organized structures, which offer unique scaffolds for applications in drug delivery and tissue engineering.

WK5

Viral Templated Palladium Nanocatalysis

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Biological macro/supramolecules such as DNA, proteins, and viruses have gained substantial attention over the past two decades as templates for the synthesis of inorganic materials. A large array of nanometer-scale materials from particles to wires have been synthesized for a variety of applications in nanoelectronics, biomedical imaging, catalysis, and energy. Of particular interest are viruses, which offer attractive templating platforms because their precise dimensions and robust structures can be exploited by genetic manipulation to confer additional functionalities and material-specific adsorption sites.

Our research is mainly focused on exploiting several unique structural, chemical, and biological properties of genetically modified tobacco mosaic virus (TMV) to create templates for nanocatalyst fabrication. Specifically, TMV is a biologically derived nanotube (18 nm diameter, 300 nm length, 4 nm inner channel) that has 2,130 identical coat proteins helically wrapped around a 6.4kb single-strand genomic mRNA. Because of its safety, well-controlled dimensions, and extraordinary stability, TMV has been extensively enlisted for synthesis of nanowires and inorganic nanoparticles. In particular, we harness precisely spaced thiol functionality displayed on the outer surface of each coat protein via a small genetic modification. The modified virus permits facile and readily controllable synthesis of palladium nanoparticles.

In this presentation, we will highlight our recent progress in the fabrication of such nanostructured palladium catalysts and in catalytic reaction studies of direct relevance to environmental remediation. In particular, we are using atomic force microscopy (AFM) and grazing incidence small angle X-ray scattering (GISAXS) to characterize these nanoparticles, with a view to addressing critical challenges in nanocatalysis and to gain fundamental understanding of nanoparticle growth kinetics.

WK5

An Infectious Approach to Biofabrication

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Advances in nanotechnology offer significant improvements in a range of applications including lightweight materials with greater strength, increased energy efficiency from electronic devices, and better sensors for a range of medical and environmental uses. However, these advances require the development of systems for the design, modeling, and synthesis of nanoscale materials. Interestingly, many biological molecules function on this scale and possess unique properties that impart the ability to assume defined conformations and assemblies as well as interact with specific chemical or biological substrates. Studies in our laboratory utilize RNA plant viruses as templates for the “bottom up” self-assembly and patterning of novel nanomaterials. Such viruses represent very simple macromolecular assemblies consisting of a single molecule of nucleic acid packaged by many copies of an identical coat protein. Using molecular genetic and chemical methods we have introduced specific structural features onto the virus backbone that direct its surface assembly and functionalization with a range of inorganic materials. Virus-assembled surfaces display an ~80-fold increase in surface that significantly enhances the activities of functionalized materials. Additionally, these virus surfaces provide a robust platform for traditional “top down” production methods, including lithography, sputtering, and atomic layer deposition. The integration of “bottom up” virus assembly methods with “top down” manufacturing processes represents an integrated approach for the production of novel nanoscale materials and devices with enhanced activities.

WK5

Using Synthetically Modified Proteins to Build Integrated Photocatalytic Systems

Matthew B. Francis

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The protein capsids of viruses provide a convenient source of rigid, nanoscale scaffolds for the construction of complex multifunctional materials. These proteins can be produced in large quantities through expression in *E. coli* and can be genetically tailored to possess reactive groups for the positioning of synthetic molecules on their surfaces. Through this program, we have explored the use of two capsid-forming proteins to build light-harvesting systems and connect them to photocatalytic groups. In one example, we have developed chemical strategies to attach the rod-like protein shell of the tobacco mosaic virus to polymers, carbon nanotubes, light-harvesting chromophores, and porphyrins. As a second target, methods have been developed to append new functionality to both the external and internal surfaces of MS2 viral capsids. These spherical assemblies have been used to house chromophores that collect light and transfer the energy to catalysts located on the exterior surface. Taken together, these new scaffolds provide many new avenues for the integration of multiple functional components with defined spatial relationships. Equally important for these studies is the set of chemical strategies that has been developed to modify biomolecules with high site selectivity and yield.

Workshop 6

Actinide Research at the APS: Successes and Prospects

Building 401, Rm. A1100

Organizers: Jim Tobin (Lawrence Livermore National Laboratory) and Mark Antonio (Argonne National Laboratory)

The actinide elements are unique in many respects, from their fundamental, practical, theoretical, and environmental concerns to their impact on the worldwide energy economy. As such, actinide materials pose extreme challenges to the scientific community in terms of their chemistry, composition, and characterization. For example, their complex electronic structures result in many unusual properties that even today are not well understood, if at all. In this regard, the availability of synchrotron radiation has revolutionized actinide chemistry and materials research. The focus of this workshop will be basic actinide science and its role in resolving technical challenges posed by actinide materials. Both fundamental and applied experimental approaches, as well as theoretical modeling and computational simulations, will be part of the workshop.

Experimental results gathered at synchrotron radiation sources, both national—with highlights of activities at the APS—and international facilities, on a deep and broad selection of actinide systems will be discussed. Particular emphasis will be placed upon projecting the needs for the next generation of cutting-edge experiments with actinide elements. Issues associated with the nuclear fuel cycle, ranging from fuel development to waste disposal, particularly as it relates to environmental remediation, will be of special significance.

13.00 – 13.05	Introductory remarks: M. Antonio and J. Tobin
13.05 – 13.35	Gerald Seidler (University of Washington) *"Standardizing" Novel Actinide X-ray Spectroscopies: A Path to a More Complete Characterization of Actinide Local Electronic Structure and Chemical Bonding at the APS*
13.35 – 14.05	Rodney C. Ewing (University of Michigan) *Actinide Materials under Extreme Conditions*
14.05 – 14.35	J. Fortner (Argonne National Laboratory) *Nuclear Fuels and Related Materials*
14.35 – 15.05	H. Cynn (Lawrence Livermore National Laboratory) *Phase Identification of Actinides and Their Compounds using X-ray Diffraction at High Pressures and High Temperatures*
15.05 – 15.20	Coffee Break
15.20 – 15.50	S. Vogt (Argonne National Laboratory) *Imaging Actinides at the APS: Current and Future Possibilities*
15.50 – 16.20	C. Hennig (Forschungszentrum Dresden-Rossendorf) *Actinide Redox Speciation and Structure Analysis in Aqueous and Nonaqueous Solutions*

16.20 – 16.50 J. Catalano (Washington University)
Application of Surface X-ray Scattering to the Study of Actinide Chemistry at Environmental Interfaces

16.50 – 17.20 L. Soderholm (Argonne National Laboratory)
Determining Free Energies of Metal Complexes in Solution using X-ray Scattering

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WK6

"Standardizing" Novel Actinide X-ray Spectroscopies: A Path to a More Complete Characterization of Actinide Local Electronic Structure and Chemical Bonding at the APS

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University of Washington, Seattle, WA 98195, USA

Among the wide range of contemporary research efforts falling into the broad category of "energy science," only a small number of scientific fronts have the clear potential to influence energy production in the United States at the level of 1 TW, i.e., the appropriate order of magnitude for comparison to present U.S. energy consumption. Work on improved actinide separations chemistry, basic f-electron material synthesis, and the post- and re-processing of nuclear fuels is in this elite category. However, many important aspects of the local electronic structure of actinide materials (e.g., the nature of chemical bonding) remain elusive, or at least unexpectedly controversial. While enhanced user capabilities in actinide X-ray spectroscopies can play a key role in fundamental understanding of f-electron bonding, it is at least as important to recognize the great, pragmatic value in simply being able to rapidly and comprehensively characterize such properties on a case-by-case basis, even (or perhaps especially) in the absence of any improved first-principles understanding. In this talk, I'll summarize ongoing work at XOR-PNC aimed at transitioning presently "novel" approaches for actinide X-ray spectroscopy into methods that can be efficiently, effectively, and rapidly employed by general users of the APS. Such approaches include studies of low-energy edges of actinides and low-Z ligands by nonresonant inelastic X-ray scattering (NRIXS) or hard X-rays and also studies of actinide L-edge and M-edge X-ray absorption spectroscopy (XAS) with high enough energy resolution to strongly suppress core–hole lifetime effects, either in partial fluorescence yield XAS (L_3-edge) or in resonant X-ray emission spectroscopy ($M_{4,5}$-edges). In this latter example, our work also includes the development of X-ray emission spectrometers which are sufficiently inexpensive that they may be used as a disposable outermost containment level for highly radioactive materials.

WK6

Actinide Materials under Extreme Conditions

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During the past few years, there has been increased interest in the response of actinide-bearing materials to extreme conditions of pressure, temperature, and radiation fields. The applications span the high temperatures and pressures in the radiation fields of nuclear reactors to the formation of fission tracks in minerals deep in Earth's crust. Additionally, an understanding of the phase stability and transformation processes under these extreme conditions can be used to create a new class of materials that have novel properties or that are resistant to radiation damage.

The compressibility, phase stability, and vibrational properties of coffinite ($USiO_4$) and brannerite (UTi_2O_6) were studied by *in situ* synchrotron X-ray diffraction (XRD) and infrared (IR) measurements at high pressure up to 40 GPa. The structural behavior of brannerite is mainly characterized by a pressure-induced amorphization process, whereas in coffinite an irreversible phase transition from the zircon-type to scheelite-type structure was found to occur at 14–17 GPa [1]. However, this phase transformation in coffinite was accompanied by partial amorphization, as shown by XRD analysis. The predicted transition pressure calculated by density functional theory was in good agreement with the experimental results. The behavior of coffinite under irradiation was systematically investigated by energetic ion beam irradiation (1 MeV Kr^{2+}) and *in situ* transmission electron microscopy (TEM), which revealed a crystalline-to-amorphous transformation at a relatively low dose of ~0.27 displacements per atom (dpa) at room temperature [2].

Most recently, diamond anvil cell experiments (up to 40 GPa) have been coupled with energetic ion beams (10s of GeV) to investigate the combined effect of pressure and irradiation on $Gd_2Zr_2O_7$ pyrochlore [3]. The formation and stabilization of a new metastable high-pressure phase was observed that cannot be obtained by irradiation or pressure applied separately. TEM data and quantum-mechanical calculations suggest that these novel structural modifications are caused by the formation of nanocrystals that change the energetics of the phase transformation. This result highlights the importance of the combined use of high pressure and high-energy ion irradiation as a new means for manipulating and stabilizing novel materials to ambient conditions that otherwise could not be recovered.

1. F.X. Zhang, V. Pointeau, L.C. Shuller, D.M. Reaman, M. Lang, Z.X. Liu, J. Hu, W.R. Panero, U. Becker, C. Poinssot, and R.C. Ewing, *Structural transitions and electron transfer in coffinite, $USiO_4$, at high pressure*, Am. Mineral. **94** (2009) 916-920.
2. J. Lian, J.M. Zhang, V. Pointeau, F.X. Zhang, M. Lang, F.Y. Lu, C. Poinssot, R. C. Ewing, *Response of synthetic coffinite to energetic ion beam irradiation*, J. Nucl. Mater. **393** (2009) 481-486.
3. M. Lang, F.X. Zhang, J.M. Zhang, J.W. Wang, B. Schuster, C. Trautmann, R. Neumann, U. Becker, R.C. Ewing, *Nanoscale manipulation of the properties of solids at high pressure with relativistic heavy ions*, Nature Materials **8** (2009) 793-797.

WK6

Nuclear Fuels and Related Materials

Jeffrey A. Fortner

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Present and future nuclear fuel cycles will require an understanding of the complex chemistry of trace fission products and transuranium actinides in used nuclear fuel and related nuclear materials. Because of the unique analytical challenges that used nuclear fuel presents to the materials scientist, many of its fundamental physical and chemical properties remain poorly understood, especially on the microscopic scale. For example, important radionuclides, such as technetium and neptunium, are present in used nuclear fuel at concentrations much less than 1%, and the fuel matrix (e.g., uranium oxide), with its own complex spectroscopy, creates a formidable technical hurdle to understanding trace element microstructure. An understanding of the chemical states of radionuclides in used nuclear fuel would benefit development of technologies for fuel monitoring, fuel performance improvement and modeling, fuel reprocessing, and storage and disposal of used fuel. We have demonstrated the use of synchrotron X-ray absorption spectroscopy (XAS) to examine crystal chemical properties of actinides and fission products in specimens of used uranium oxide fuel and other radionuclide-bearing materials from the nuclear fuel cycle. Information obtained includes oxidation state, chemical bond coordination, and quantitative elemental concentration and distribution. We have also used XAS in a scanning mode to obtain X-ray spectral micrographs with resolution approaching 1 micron. A brief overview of technique will be presented, along with findings on uranium, plutonium, neptunium, technetium, and molybdenum in commercial used nuclear fuel specimens.

Research funded by the U.S. Department of Energy, Office of Civilian Radioactive Waste Management, under Contract DE-AC02-06CH11357.

WK6

Phase Identification of Actinides and Their Compounds using X-ray Diffraction at High Pressures and High Temperatures

Hyunchae Cynn

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X-ray diffraction (XRD) is an extremely valuable technique to study materials at extreme conditions, especially high pressures and high temperatures (when coupled with a diamond anvil cell). Information that can be obtained using XRD analysis includes phase identification, phase stabilities, equations of state, phase boundaries, microstructures, and crystal structures.

A newly cast Pu alloy with ~2 atomic-% Ga examined using synchrotron XRD at APS shows δ, γ, and α phases coexisting at high pressures. The XRD measurements show α→β→γ→δ transitions. Growths of β and γ phases were identified as spotty diffraction rings on the images, which may indicate microstructure behavior during nucleation. At ~650 K and ~10 kbar, the δ→β transition was observed. Structural refinement shows that the atomic coordinates for β´-Pu agree very well with the measurements of Zachariasen and Ellinger (1963). Coexistence of the δ, γ, and α phases in Pu alloys (both ~2 atomic-% Ga and ~3 atomic-% Ga) were also observed at 5~7 kbar at ambient temperature. This result is similar to a previous report by Faure et al. (2005). Phase stabilities of Pu alloys and some actinide compounds will be presented.

1. W.H. Zachariasen and F.H. Ellinger., Acta Crystallog., **16**, 369, 1963.
2. Faure et al., MRS Symposium Proceedings., **893**, 223, 2005.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory in part under Contract W-7405-Eng-48 and in part under Contract DE-AC52-07NA27344 and performed at HP-CAT (Sector 16), Advanced Photon Source, Argonne National Laboratory. HP-CAT is supported by DOE-BES, DOE-NNSA, NSF, and the W.M. Keck Foundation. APS is supported by DOE-BES, under Contract No. DE-AC02-06CH11357.

WK6

Imaging Actinides at the APS: Current and Future Possibilities

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Most specimens and materials of interest, either manmade or natural, are complex and heterogeneous, typically in three dimensions. X-ray imaging and microscopy are unique tools that permit not only visualization of specimen structure at high spatial resolution, but also, because of their significant depth penetration, the acquisition of depth information through the use of tomographic approaches. Full-field techniques typically excel in speed of data acquisition and direct visualization of specimen structure, down to the level of several tens of nanometers. Scanning probe methods tend to be slower, but they can provide richer information, including chemical sensitivity down to trace levels, and offer the ability to probe local elemental oxidation states, as well as probe reciprocal space information through microdiffraction.

We will give an overview of imaging techniques available at the APS and report on their use in several relevant applications, ranging from the visualization of trace amounts of Pu in mammalian cells to the characterization of oxide layers formed on Zr alloys. We will also provide an outlook on future instrumentation.

WK6

Actinide Redox Speciation and Structure Analysis in Aqueous and Nonaqueous Solutions

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[2] Rossendorf Beamline, European Synchrotron Radiation Facility, Grenoble, France

Actinide redox chemistry plays a crucial role in nuclear technologies such as fuel reprocessing and in aspects of nuclear waste disposal, including predicting actinide mobility in the environment. Hydrolysis and complex formation of the early actinides are strongly related to their redox behavior in aqueous and nonaqueous solutions. We are interested in understanding the complexation of actinide compounds under controlled redox conditions in the presence of inorganic oxo ligands and organic ligands with carboxylic groups.

This study is mainly focused on the use of extended X-ray absorption fine structure (EXAFS) spectroscopy in combination with other supporting methods. The information from EXAFS spectroscopy is restricted to a pair distribution function providing coordination numbers and distances of the next neighbors. Two pathways were used to relate this information to the spatial arrangement of the ligands: (i) EXAFS was combined with density functional theory (DFT) calculations, which introduce constraints of molecular characteristics [1], and (ii) individual solution species were preserved in crystal structures and determined with single crystal diffraction. EXAFS was used subsequently to quantify differences or identify agreement between the coordination of complex structures in solution and solid state [2-4]. EXAFS is furthermore not very sensitive for differentiating between individual solution species, especially not in a single sample. This problem was solved in the following manner: (i) by combining EXAFS with other more species-sensitive techniques, e.g., ultraviolet-visible spectroscopy [5], and (ii) by using sample series with broad variation of species distribution and subsequent application of statistical analysis techniques to separate the scattering contribution of individual solution species [6]. Finally, we investigated the correlation between the formal redox potential and the stability range of solution species [7].

1. Hennig et al. *The sulfate coordination of Np(IV), Np(V) and Np(VI) in aqueous solution*. Inorg. Chem. **48** (2009) 5350-5360.
2. Hennig et al. *Comparative investigation of the solution species* $[U(CO_3)_5]^{6-}$ *and the crystal structure of* $Na_6[U(CO_3)_5]\cdot 12H_2O$. Dalton Trans. **39** (2010) 3744–3750.
3. Takao et al. *First hexanuclear* U^{IV} *and* Th^{IV} *formate complexes – structure and stability range in solution*. Eur. J. Inorg. Chem. (2009) 4771-4775.
4. Hennig et al. *Coordination of a uranium(IV) monomer in aqueous solution and in solid state*. Inorg. Chem. **47** (2008) 1634-1638.
5. Hennig et al. *The relationship of monodentate and bidentate coordinated uranium(VI) sulfate in aqueous solution*. Radiochim. Acta **96** (2008) 607-611.
6. Hennig et al. *Species distribution and coordination of uranyl chloro complexes in acetonitrile*. Inorg. Chem. **47** (2008) 2987-2993.
7. Takao et al. *Complex formation and molecular structure of neptunyl(VI) and –(V) acetates*. Inorg. Chem. **48** (2009) 8803-8810.

WK6

Application of Surface X-ray Scattering to the Study of Actinide Chemistry at Environmental Interfaces

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Reactions occurring at the interface between aqueous solutions and environmental solids often control the fate and speciation of actinides in the environment. Laboratory-based studies have sought to characterize the mechanistic processes involved in actinide interfacial reactions using a variety of macroscopic and molecular-scale probes, most notably extend X-ray absorption fine structure (EXAFS) spectroscopy. However, many critical aspects of actinide interfacial reactions cannot be probed directly or unambiguously with these traditional methods. Surface X-ray scattering methods have seen application in recent years to the study of environmentally relevant actinide interfacial reactions. Recent experimental studies using surface X-ray scattering will be reviewed and outstanding questions in actinide chemistry that may be addressed using these methods will be discussed.

WK6

Determining Free Energies of Metal Complexes in Solution using X-ray Scattering

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Although it is understood that a metal ion dissolved in solution experiences correlations with both solvent molecules and other dissolved ions, quantifying these interactions has been hampered by the absence of an appropriate experimental probe. X-ray absorption fine structure (XAFS) spectroscopy has provided critical information concerning both the presence of correlations and the ions involved, information that has had an impact on modeling metal-ion solubilities, reactivities, and stabilities as a function of solution composition. Unfortunately, technical difficulties with XAFS spectroscopy limit the ability to determine near-neighbor coordination numbers to within an uncertainty of about 10%. This precision is often too low to extract the needed information about the coordination environment. In addition, the second- and higher-neighbor correlations are often inaccessible for study with this technique. We have recently begun to employ high-energy X-ray scattering (HEXS) as a more sensitive probe of metal correlations in solution [1-4]. Our initial experiments have involved dissolved 4f (lanthanide) and 5f (actinide) ions because of their high atomic number and therefore their contrast and overall contribution to the total scattering from aqueous solution. We have been able to extract metrical coordination information for the inner coordination sphere environment within an error of about 2%, and second- and higher-sphere coordination information to about 5%. Changes in metal-ion coordination as a function of anion concentration can now be probed with a precision sufficient to provide the quantitative information needed to determine thermodynamic stability constants for simple metal-ion solution reactions. For example, HEXS provides quantitative structural information on the chloride ion association with Er^{3+} in acidic solution [5],

$$Er^{3+} + Cl^{-} + ErCl^{2+} + Cl^{-} + ErCl_2^{+},$$

data that have been used to determine independent stability constants for the inner and outer sphere complexes simultaneously present in solution. Thus the new information provided by the scattering experiments can be used to directly relate the structure of complexes formed in solution with their thermodynamic stability. This capability is providing an entirely new perspective on textbook concepts such as simple inner- vs. outer-sphere complexation and their relative importance to the overall metal energetics. The results are of particular importance to theoreticians who are trying to model solution behavior while explicitly considering only near-neighbor correlations.

This work is supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Chemical Sciences, under contract DE-AC02-06CH11357.

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Workshop 7

Are Phase-Contrast and Diffraction Imaging/Microscopy Ready for Biology and Medicine?

Building 401, Rm. A5000, afternoon

Organizers:	Han Wen (National Institutes of Health), and Christoph Rose-Petruck (Brown University)

Within the past five years, phase-contrast (PC) and diffraction imaging saw rapid advance, particularly in full-field techniques, which are driven by the speed requirement of biological and medical applications. Among these are wave-propagation-based PC techniques using high spatial-coherence sources, Talbot-grating-based phase-stepping techniques, grid-based spatial harmonic techniques, and crystal-analyzer-based techniques. These approaches are distinct but also interconnected. They are now being implemented in a number of forms that include microscopy, radiography, tomography, and elastography and some have reached live-animal *in vivo* imaging. This workshop aims to facilitate exchange of the latest ideas among researchers working with the various approaches, to focus on the first biomedical applications that are on the horizon, and to highlight what needs to be developed for further expansion into biology and medicine.

1:30 – 1:35	Introduction
1:35 – 2:20	Carol Muehleman (Rush Medical College) *Diagnostic Potential of Phase-sensitive Radiography*
2:20 – 3:05	Christopher Parham (University of North Carolina) *Breast Imaging*
3:05 – 3:30	Coffee Break
3:30 – 3:55	Zhong Zhong (Brookhaven National Laboratory) *Beamline Instrumentation and Diffraction Imaging*
3:55 – 4:20	Christoph Rose-Petruck (Brown University) *High-performance Sources and Imaging*
4:20 – 4:45	Han Wen (National Institutes of Health) *Spatial Harmonic Techniques for* in vivo *Imaging*

POSTER
ABSTRACTS

U-01

The Advanced Photon Source Upgrade (APS-U)—Beamline Optics and X-ray Detector Capabilities

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Novel beamline optics and x-ray detectors will be required to fully realize the potential of the planned Advanced Photon Source Upgrade (APS-U). This poster presents the current approach envisioned by members of the APS-U Conceptual Design Report (CDR) optics and detectors teams to address these beamline instrumentation challenges. We expect that this poster will help continue the dialogue between the CDR teams and the APS user community on optics and detector needs for APS-U and the best strategy to satisfy them.

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.

U-02

Changes to the Advanced Photon Source Storage Ring as Part of the APS Upgrade

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The Advanced Photon Source (APS) is a third-generation storage-ring-based x-ray source that has been operating for more than 13 years and is enjoying a long period of stable, reliable operation. While APS is presently providing state-of-the-art performance to its large user community, we must clearly plan for improvements and upgrades to stay at the forefront scientifically. Significant improvements should be possible through upgrades of beamline optics, detectors, and end-station equipment. In this paper, we discuss the evolutionary changes that are envisioned for the storage ring itself. Among the possibilities are long straight sections, superconducting undulators, picosecond x-ray pulses, higher current, and improved beam stability.

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

U-03

Upgrade Projects in High-resolution Spectroscopy

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The APS has a history of strong and successful programs in high-resolution spectroscopy, and these will be significantly improved in the APS upgrade. In most cases the upgrade will result in dedicated beamlines with optimized sources and enhanced detectors, expanding both capabilities and availability. At sector 20, dual canted undulators will be used to provide for a dedicated beamline for an enhanced LERIX (lower-energy resolution inelastic x-ray scattering) spectrometer, giving up to 50x improvement in signal. The existing microspectroscopy station at sector 20 will be optimized for high-resolution fluorescence spectroscopy and ultra-dilute detection capabilities. Similarly sector 30 will be upgraded for full time operation of an improved HERIX (high-energy resolution inelastic x-ray) spectrometer with an optimized undulator source. The MERIX (medium-energy resolution inelastic x-ray scattering) programs at sectors 9 and 30 will be consolidated to a full-time program at 9-ID, again with an optimized undulator and upgraded spectrometer. At sector 3 the undulator will be optimized, and a new hutch added to house a super- inelastic x-ray scattering spectrometer. Finally, the only bending magnet beamline in this section, 9-BM, will be optimized for catalyst research with improved optics and a quick-scanning monochromator. We will briefly describe these upgrades, expected performance improvements, and some of the science that will be enabled by the enhancements.

U-04

The APS Upgrade: Imaging and Coherence

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The APS Upgrade will provide exciting new capabilities for imaging and coherence experiments. This poster describes the plans at present, so as to inform the user community and solicit input.

U-05

Advanced Photon Source Upgrade (APS-U)—Extreme Conditions

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Materials subjected to the extremes of pressure, temperature, magnetic fields, or electric fields can display a host of novel electronic and structural properties. Understanding how these new phases of matter are formed can frequently provide a path towards engineering new functionalities into materials at ambient conditions. Due to its high brilliance and penetrating power, synchrotron radiation provides an ideal probe for materials at extreme of phase space, since such measurements generally require small sample volumes. This poster will describe proposed beamline upgrades at the APS seeking to enhance the capabilities for studying materials under extreme environments.

U-06

APS Upgrade: Connecting Proteins to Organisms

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One of the grand challenges in the life sciences is to connect our understanding of proteins on the structural level to their function within organisms through their role in organelles, cells, organs, and beyond. Other important molecules of life such as RNA and DNA are involved in replication, carbohydrates in recognition, membrane proteins in signaling and transport, and macromolecular complexes and assemblies in cellular function. Crystallography is an essential tool towards the goal of understanding the structure of the individual components and their complexes. In addition to improving throughput, it will be necessary to enable techniques that can circumvent existing bottlenecks such as the difficulty in obtaining adequate crystals. For example, microcrystallography promises to allow data collection from micron-sized crystals as well as reducing the radiation damage to an exposed micron-sized area. Beyond determining static crystal structures, it is vital to further our understanding of protein dynamics and conformational changes. This is achievable through inelastic scattering as well as through direct ultrafast scattering and spectroscopy. The behavior of proteins in conditions typical of *in vivo* systems (e.g., in solution), protein folding, protein-protein interactions, and drug/ligand binding can be studied using small-angle and wide-angle x-ray scattering (SAXS/WAXS) techniques. Connecting to proteins' function in organisms, in particular under environmental factors such as the local chemical/trace elemental environment, can be addressed by using tools such as the cryo-nanoprobe beamline. This will allow the visualization of trace elemental content in biological systems down to the level of 10's of nanometers, detecting as few as 10 metal atoms for specific elements in ideal samples. Not only will this further our fundamental understanding of biological systems and their interaction with the local environment but also have significant impact on applications in health and disease as well as environmental stewardship. Ancillary equipment will facilitate correlative experiments as well as allow supplemental studies (e.g., optimum freezing/vitrification techniques).

We will give an overview of projects planned with regards to the proposed APS upgrade, with emphasis on (1) cryo-nanoprobe beamline, (2) enhanced SAXS/WAXS beamline, (3) microfocus macromolecular crystallography (MX) beamline, (4) enhanced time-resolved MX beamline, and (5) cryo sample prep/analysis facility.

U-07

Picosecond X-ray Science

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The Short Pulse X-ray (SPX) facility will extend time-resolved x-ray scattering and spectroscopy to the picosecond timescale while retaining the powerful characteristics of synchrotron radiation, i.e., user-controlled continuous tunability of energy, polarization, and bandwidth combined with exquisite x-ray energy and pulse length stability over a wide energy range. Experiments at the SPX facility will produce one-picosecond stroboscopic snapshots of molecular rotations, molecular excited-state transient structures, stress/strain wave propagation, magnetic domain wall dynamics, phase transitions, and the coupling between electronic, vibrational, and magnetic degrees of freedom in condensed matter systems. Time-resolved studies of transient dynamics will be possible with simultaneous picosecond time resolution and picometer structural precision for a variety of atomic, molecular, supramolecular, nanoscale, and bulk material systems. Transformational developments are now taking place in high-average-power pulsed laser technology, with substantially increased repetition rates that promise to make highly efficient use of the MHz x-ray repetition rates of the SPX. We propose to develop, in the context of the APS Upgrade, five end stations with scattering, diffraction, spectroscopy, imaging, and microscopy capabilities.

U-08

The Advanced Photon Source Upgrade (APS-U): Interfaces in Complex Systems

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X-rays offer a unique opportunity to penetrate many complex environments to probe the structure and chemistry of surfaces and buried interfaces. These interfaces between media with distinct physical and chemical properties offer unique opportunities for discovery in diverse scientific areas such as catalysis, oxide film growth, semiconductors, geochemistry, surface physics, biomembranes, corrosion, nanoscience, environmental science, tribology, and electrochemistry.

A dedicated X-ray Interfacial Science (XIS) facility at a sector of the APS will fully exploit the unique capabilities of the APS to advance our understanding of interfacial science. The XIS facility will provide state-of-the-art x-ray scattering, spectroscopy, and microscopy tools for multi-length-scale, element-specific studies of vacuum/solid, gas/solid, liquid/solid, solid/solid interfaces, and supported nanostructures. The proposed facility has two optimized canted undulators producing four separate x-ray beams: one with variable energy and three with selectable fixed energies. In addition, the bend-magnet port will be developed to support those measurements that don't require the brightness of the ID beamlines. A tandem experimental hutch arrangement on each beamline will include both general-purpose diffractometers and custom-designed spectrometers to provide *in situ* controlled environment and growth chambers for various advanced materials processing and synthesis activities.

In addition to the proposed new sector, a canted undulator front-end source will provide a dedicated beamline for liquid surface scattering. This line will be tunable over the range 7–30 keV and will be outfitted with a custom liquid interface scattering instrument. Improved x-ray facilities for *in situ* surface and interface science will also provide enhanced capabilities for the ultra-high vacuum surface diffraction, and for molecular beam epitaxy surface science programs (surface x-ray absorption fine structure and surface resonant scattering).

The APS Upgrade provides new and enhanced x-ray facilities for *in situ* surface and interface science that will fundamentally change the paradigm for the study of the structure and properties at interfaces in fabricated systems as well as those found naturally in our environment. The opportunity to include integrated imaging techniques, time-resolved measurement capabilities, and the infrastructure to enable *in situ* experiments in very complex environments to probe interfacial processes holds great promises for discovery in interfacial and surface chemistry, dynamics, fundamental growth processes and fabrication, and nano-scale sciences.

U-09

Enabling Technical Capabilities

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Enabling technical capabilities provide the foundations (literally in some cases) on which parts of the APS Upgrade will be built. This somewhat eclectic area encompasses Conventional Facilities, IT Infrastructure, and Software Infrastructure. Although at this early stage it is difficult to define solid requirements, here we present some ideas and concepts that we believe may be required to support the upgraded APS. We would like to provoke thought and discussion and welcome feedback.

A-01

In Situ High-energy X-ray Diffraction for Advanced Material Discovery and Design

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It has been widely accepted that the structure, chemical, and physical defects of materials are critical factors to determine the performance of devices using such materials. A comprehensive understanding of the structure-property relationship of materials can provide valuable insight for the discovery and rational design of advanced materials with improved performance. The accomplishment of this challenging task will need critic support from state-of-the-art characterization techniques such as *in situ* high-energy x-ray diffraction.

For instance, $LiFePO_4$ was first reported by Goodenough and coworkers as a potential positive electrode material for lithium ion batteries in 1997 [1]. Because of its low electronic conductivity, the full potential of $LiFePO_4$ for lithium ion batteries was not unlocked until Chiang and coworkers reported that high rate application of $LiFePO_4$

can be achieved by chemical doping of metal metals supervalent to Li in nanostructured $LiFePO_4$ [2]. However, the mechanism of the performance improvement is still under debate [3, 4].

In this work, *in situ* high-energy x-ray diffraction technique was used to investigate the phase formation and defect evolution of $LifePO_4$ during solid state synthesis. The *in situ* experiment showed that $LiFePO_4$ phase started to form at a temperature as low as 200°C, and a pure $LiFePO_4$ was obtained at about 300°C. It was also found that impurity phases, which were identified as a mixture of Fe_2P and Fe_3P, started to appear when the $LiFePO_4$ sample was baked at a temperature above 400°C. Electrochemical data showed that $LiFePO_4$ synthesized at <400°C exhibit poor electrochemical activity while those samples prepared at >500°C have great performance. With the combination of *in situ* data and electrochemical data, we realized that the Fe_2P/Fe_3P impurity phases are critical for the performance improvement.

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A-02

The X-ray Beam and Biological Crystal Visualization for Macromolecular Crystallography

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The SBC on-axis visualization system allows viewing of x-ray beam and biological crystals from x-ray beam direction without parallax distortion. The system was constructed using non-dispersive optics: a long-working-distance Maksutov–Cassagrain reflective microscope and right-angle (45°) mirror. This on-axis geometry allows crystal visualization during diffraction data collection with full Kappa geometry.

An x-ray beam and biological crystal imaging system during data collection has been developed. The direct x-ray beam uses x-ray excited ultraviolet (UV) fluorescence. The high-energy radiation such as x-ray and middle UV (MUV) radiation excite "visible" light luminescence from biological materials, which can be imaged with CCD cameras. The fluorescence from biological crystals is primarily emitted as near-UV (NUV) wavelengths between 300–360 nm depending on the biological material and surrounding environment. We demonstrate detection of biological crystal location using x-ray excited UV fluorescence. We discuss techniques for biological crystal location using intrinsic x-ray excited and MUV excited UV fluorescence from biological crystals.

The x-ray beam can be characterized using a scintillator (phosphor or a single crystal) that converts x-ray photons into visible light photons, which can be imaged using SBC on-axis optics. The x-ray penetration is dependent on the composition of the scintillator (especially effective Z) and x-ray energy. Several scintillators have been used to visualize x-ray beams. Here we compare $CdWO_4$, $PbWO_4$, $Bi_4Ge_3O_{12}$, $Y_3Al_5O_{12}$:Ce (YAG), and Gd_2O_2S:Tb (phosphor). The synchrotron x-ray beam profile studies were done using on-axis and off on-axis imaging. We determined that scintillators made of $CdWO_4$ and similar high-Z single-crystal materials are best suited for the energy range (7–20 keV) and are most suitable for beam visualization for macromolecular crystallography applications. These scintillators show excellent absorption, optical, and mechanical properties.

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A-03
In-field Scattering Studies Using a Cryogen-free Magnet at APS

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Over the past several years a number of high-resolution in-field scattering studies have been performed at the APS using cryogen-free magnets. While these magnets produce relatively modest magnetic fields (4 tesla) compared to larger magnet systems, they have a number of unique advantages such as providing greater angular access to the sample and more flexibility in the scattering geometry. More importantly, however, these magnets are extremely simple and inexpensive to operate, which makes them more conducive to use by novice users. A horizontal-scatting diffractometer, which can accommodate either of two nearly identical cryogen-free superconducting magnets, has been used on both the 4-ID-D and 6-ID beamlines for in-field scattering experiments. One magnet applies a vertical-field perpendicular to the scattering plane while the other applies a horizontal-field in the scattering plane. This poster will present recent results obtained using these magnets and layout plans for upcoming upgrades.

A-04
High-field (30 tesla) Pulsed-magnet Instruments for X-ray Studies of Materials

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Pulsed magnets have emerged as a viable approach at synchrotron x-ray facilities for studying materials in high magnetic fields. We present two extremely high-field (30 tesla) pulsed magnet systems for x-ray diffraction studies of materials at the Advanced Photon Source (APS). These instruments employ unique two-cryostat schemes. The first is a split pair of mini-coils (duration ~1ms) with applied magnetic field normal to the scattering plane [1]. The second is a single 18-mm bore solenoid (duration ~4 ms) that allows the applied field to be parallel to the scattering plane. Both coils have been designed and built at Tohoku University using high-tensile-strength and high-conductivity CuAg wires. Dual-cryostat schemes have been developed at the APS in order to cool the coil and the sample independently. The split-pair magnet is conduction cooled using a close-cycle cryostat while the solenoid is cooled in liquid nitrogen (LN). While a repetition rate for the split-pair magnet is ~10–20 minutes for peak fields in the range of 20–30 tesla, LN cooling allows a repetition rate of a few minutes for peak fields near 30 tesla in the solenoid. Pulsed fields are generated by a capacitive discharge using a configurable bank (3 kV, 40 kJ). Time-resolved diffraction data are typically collected using a fast avalanche photodiode or a one-dimensional strip detector. Results from preliminary scattering studies of structural effects and magneto-striction in a geometrically frustrated magnet will be presented.

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A-05
IMCA-CAT Insertion Device Beamline Upgraded for High-throughput, Shutterless, Continuous-rotation Data Collection

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The IMCA-CAT insertion device beamline, 17-ID, has been upgraded to a microfocused high-flux diffraction beamline for automated high-throughput macromolecular crystallography. The energy range is 6–20 keV, allowing multi-wavelength anomalous dispersion/single-wavelength anomalous dispersion experiments at energies for commonly used derivatives. The full beam is focused to 65 μm × 30 μm at the sample position and the GM/CA-

CAT mini-quad collimators provide the user with selectable beam sizes of 300, 20, 10, and 5 μm. Beam stability is achieved with custom software that automatically positions the beam with ±2 μm positional accuracy. Automated sample mounting is performed with the Rigaku ACTOR robot, and samples are viewed with the Maatel on-axis viewing system. The new ALIO goniometer has a small (1.2 μm) sphere of confusion, thus maintaining accurate sample positioning. A new detector, the PILATUS 6M pixel-array from DECTRIS, permits shutterless, continuous-rotation data collection. The high volume of data is managed with a 64 TB storage system that consists of a highly available Lustre-distributed parallel file system with Fibre Channel and InfiniBand interconnects. Custom software provides an intuitive interface for controlling the beamline. Rigaku JDirector software for data collection enables queuing of data collection jobs for automated data acquisition. Both unattended and remote data collection modes are supported. The automated and rapid data collection capabilities of beamline 17-ID are ideally suited for high-throughput crystallography projects such as pharmaceutical industry drug discovery programs.

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A-06

Characterization of the Two Mutants Y29F and Y29A of Vitreoscilla Hemoglobin

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Vitreoscilla, an obligately aerobic bacterium, synthesizes a relatively large amount of soluble, homodimeric hemoglobin (VHb) under hypoxic conditions. VHb can support aerobic growth in *Escherichia coli* with impaired terminal oxidase function. This ability of VHb to improve the growth properties of *E. coli* has important applications in fermentation technology assisting the over expression of recombinant proteins and antibiotics. The three-dimensional structure of VHb, obtained through x-ray crystallography has shown that the structure confirms to the well-known globin fold. The polypeptide segment connecting helices C and E is disordered and residues E7–E10 do not adopt the usual alpha helical conformation. A key residue in the VHb distal site, Tyr29 (B10), which is highly conserved in various bacterial hemoglobins, may be involved in modulating the oxygen binding properties of VHb. We have introduced two mutations at this site, namely Y29F and Y29A, to see if the side chains of tyrosine (Tyr) have any role in stabilization of oxygen and if any structural change in this position can cause a major change in the functional properties of the protein. The high-resolution crystal structures obtained from these two mutants shows that the Y29F mutant crystallized in the same space group (P21) like that of the wild type with a dimer in the asymmetric unit and the Y29A crystallized in a different space group with the monomer in the asymmetric unit. The missing residues in the disordered region (44, 45, 46, 47, and 50) were built in and showed that the region is helical. In addition, E7 (Q53) is in close contact to the propionate on heme, which is significantly different from the wild type and the Y29F mutant where it faces away. Further studies on these two mutants using the circular dichroism spectra and the carbon monoxide difference spectra has shown that there isn't a major change in the helicity and the binding of heme in the two mutants, which emphasizes that the ordering seen in the Y29A mutant is an artifact of crystallography.

A-07

Synchrotron Powder Diffraction Simplified: the High-resolution Diffractometer 11-BM at the Advanced Photon Source

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Synchrotrons have revolutionized powder diffraction. They enable the rapid collection of high quality powder diffraction patterns with tremendous resolution and superb signal to noise. The high penetration and exceptional data sensitivity possible at high-energy light sources like the Advanced Photon Source (APS) allow synchrotrons to explore trace containment levels, extreme sample environments, and crystallographic site occupancies. Despite these advantages, relatively few scientists today consider using a synchrotron for routine powder diffraction studies.

To address this, the new synchrotron powder diffractometer beamline 11-BM at the APS now offers rapid and easy mail-in access for routine structural analyses with truly world-class quality data. This instrument offers the highest resolution available in the Americas and is a free service for non-proprietary users. The instrument can collect a superb pattern suitable for Rietveld analysis in less than an hour, is equipped with a robotic arm for automated sample changes, and features variable temperature sample environments. Users of the mail-in program receive high-resolution data within two weeks of sample receipt. The diffractometer is also available for on-site experiments that require more specialized measurements.

Our presentation will describe the instrument, highlight its capabilities, and explain the types of measurements currently available. More information about the 11-BM diffractometer and its mail-in program is available at http://11bm.xor.aps.anl.gov.

A-08

Enhancing the Context of X-ray Fluorescence Imaging: Highlights from the Redevelopment of Beamline 8-BM

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The development of x-ray fluorescence (XRF) microscopy has opened a new window into the role of trace metals in the life and environmental sciences. While very successful, this approach is severely limited in the scope of study due to the time necessary to acquire statistically significant data in a scanning microprobe, in particular for low-probability events. We are now developing an existing bending magnet beamline (8-BM) into a dedicated beamline for the high-throughput analysis of trace elements in the life sciences. We are commissioning dedicated instrumentation that allows the high-throughput analysis of micro-arrays and tissue sections to improve our understanding of trace metals and their essential role in health and disease. In particular, the instrumentation will enable the rapid acquisition of large data sets necessary for clinically relevant interpretation. We will also incorporate x-ray cytometry to study the total trace element composition of single cells in order to examine large cell populations on an individual basis, enabling experiments with a large scope that are currently not possible. Lastly, by combining XRF with both traditional and cutting-edge proteomics technologies, we will enable unprecedented scientific inquiry into metal mediated regulation of biological pathways.

A-09

Elucidating Mechanisms of Ba and Sr Selectivity in Desmid Green Algae with X-ray Fluorescence Microscopy

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The removal of ^{90}Sr from radioactive waste is complicated by the chemical similarity of Ca^{2+}, Sr^{2+}, and Ba^{2+} cations, which also leads to indiscriminate transport of these ions by most organisms. However, desmid green algae of the genus *Closterium* have the ability to selectively precipitate $(Ba, Sr)SO_4$ crystals within vacuoles, indicating that they possess a rare mechanism for selectivity of Sr and Ba vs. Ca. Using synchrotron x-ray fluorescence microscopy, we have quantified the uptake and sequestration of Ba and Sr into crystals and have observed time-dependent changes in intracellular Sr concentrations. The kinetics of uptake and efflux of Sr appear to be dependent on external Ca concentrations, and Sr and Ca show similar cellular localization. We conclude that selectivity for Sr versus Ca does not occur at the whole cell level. Instead, high S levels detected in the vacuole suggest a selective precipitation mechanism. We propose a "sulfate trap" model in which high sulfate levels in the vacuole leads to preferential precipitation of $(Ba, Sr)SO_4$ solid solutions due to their low solubilities relative to $CaSO_4$. An elucidation of the mechanisms of selectivity in these organisms will enable and inspire innovation of chemical and bioremediation approaches to ^{90}Sr cleanup operations.

A-10

Development of X-ray Photon Correlation Spectroscopy in the Ultrasmall-angle X-ray Scattering Scale and Time Regime

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Over the last decade the ultrasmall-angle x-ray scattering (USAXS) instrument at the Advanced Photon Source has been continually upgraded and has become a world-class instrument for materials research. Meanwhile, x-ray photon correlation spectroscopy (XPCS) has emerged as a measurement technique that offers unprecedented sensitivity to the dynamics of structural changes in materials. However, existing XPCS facilities are limited to microstructure length scales smaller than ~50 nm, thus eliminating large classes of materials that are of major technological importance. Recently, the range of this technique has been extended dramatically by combining XPCS speckle measurements with USAXS studies at the Advanced Photon Source. While USAXS characterizes microstructures over the nanometer-to-micrometer scale range, use of a small entrance slit allows the coherence of the undulator x-ray beam to be exploited to give synergistic XPCS measurements of internal microstructure dynamics. At the large end of the scale range, the correspondingly slower dynamics are well matched to USAXS scan times. Progress in XPCS/USAXS development will be demonstrated with reference both to well-characterized colloidal dispersions and to composite systems for biomedical applications.

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A-11

Gold Cluster Labeling as a New Tool to Study Protein Structures Using Small-angle X-ray Scattering

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A new methodology for using gold cluster labeling as special probes in solution x-ray scattering is presented. Gold cluster labeling provides strong scattering interferences between gold atoms and other atoms. These interferences give the structural information of proteins. We showed how to reconstruct labeled protein structures and extract 1D structural information from solution scattering data. Simulated scattering curves of templates were compared to those of trial structures. The trial structures were built by rigid body modeling (RBM) for a fixed protein with mobile gold cluster(s). Rigid body searches were performed in two steps. First, inter-body distances were estimated from fine-grid RBM without rotation. Secondly, gold center positions were predicted from distance-corrected coarse-grid RBM. Inter-body distances of each trial structure were corrected by least square minimization during RBM. In the analysis, double-labeled and single-labeled data were combined using the best performing scoring function, which improves the prediction accuracy of gold center positions (1.4–8.9 Å error). We also investigated the effect of inter-body distance error and grid error to obtain the nearest neighboring trial structure as a top scoring solution; these trials indicate that 0.2 Å of inter-body distance error and 0.5 Å of grid error are the maximum allowed errors.

A-12

Buckled Membranes in Mixed-valence Ionic Amphiphile Vesicles Analyzed by X-ray Scattering

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We demonstrate that charged amphiphilic molecules, including molecules with biological motifs, organize into non-spherical shapes expected for elastic membranes. Specifically, we demonstrate that anionic (-1) water insoluble amphiphiles and cationic amphiphiles (+3) (which form micelles in water), can co-assemble into small buckled vesicles [1]. The strong electrostatic interaction between the +3 and -1 head groups increases the cohesion energy of the amphiphiles and favors the formation of two-dimensional, flat ionic domains on the vesicle surface, resulting in edges and a buckled shape. Small-angle x-ray scattering/wide angle x-ray scattering (WAXS) experiments were conducted in APS 5-ID-D. WAXS measurements confirm the presence of crystalline domains induced by these ionic correlations.

1. *J. Am. Chem. Soc.*, **131**, 2030–12031 (2009).

A-13

Excited State Axial Ligation in Metalloporphyrins as Revealed by Transient X-ray Absorption Spectroscopy

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The photoinduced axial ligation mechanism of a metalloporphyrin, nickel(II) tetramesitylporphyrin (NiTMP) is investigated by transient x-ray absorption spectroscopy at the Ni K-edge (8.333 keV). Photoexcitation of the porphyrin Q-band induces a $(3d_{z2}, 3d_{x2-y2})$ configuration and promotes axial ligation. The x-ray transient absorption at $1s \rightarrow 4p_z$ transition energy taken at different time delays after the photoexcitation presents a time sequence of the excited state and ligation processes. A unified axial ligation reaction mechanism for the ground state and the photoexcited state is proposed based on the elucidation of the excited state structural dynamics by transient optical and x-ray absorption spectroscopy as well as by density functional theory calculations. The results obtained from this study can be broadly applied to study other metalloporphyrins and metal complexes for their photocatalytic activities involving the transient ligation.

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A-14

X-ray Photon Correlation Spectroscopy Measurements of Dynamics within Concentrated Eye Lens Protein Suspensions

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X-ray photon correlation spectroscopy (XPCS) has been applied in recent years to the study of dynamics within a wide range of soft materials. Its application to protein diffusion has been hindered by the weak scattering and the susceptibility of proteins to damage. We report the first application of XPCS to study the dynamics of concentrated suspensions of alpha and gamma crystallin proteins extracted from bovine eye lens. The limitations of low flux and x-ray damage were overcome through a combination of a fast, high-efficiency CCD, a kinoform lens, and by limiting exposures to short intervals. Dynamics were measured both for alpha crystallin suspended in a concentrated matrix of gamma crystallin and concentrated alpha crystallin suspensions, showing time constants of a few tenths of seconds at length scales corresponding to the protein diameter.

A-15

Polarization Switching Dynamics of Dielectric/Ferroelectric Superlattices

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The domain switching dynamics of the ferroelectrics are fundamentally interesting because the time and the length scales involved depend on the applied electric field. The nanoscale dielectric/ferroelectric superlattices are thought to possess domains with a uniform polarization penetrating the individual components. A population of domains with opposite signs exists at zero-external electric field to minimize the total energy. The dynamics of the process by which these domains are rearranged in an applied electric field have not been experimentally probed. To study the dynamics of the initial response of ferroelectric/dielectric superlattices to applied fields, we performed time-resolved x-ray microdiffraction studies of a $2(BaTiO_3)/4(CaTiO_3)$ superlattice in a pulsed electric field. The results of these experiments show that the polarization switching of the superlattice occurs at a broad range of applied electric fields, which agrees with theoretical predictions on the different coercive fields of individual component of the superlattice [1]. In addition, the strain is proportional to the applied electric field above 0.4 MV/cm when the polarization is fully switched on the millisecond timescale, but the strain is strongly non-linear up to 1.5 MV/cm on the nanosecond timescale due to the incomplete polarization switching during shorter times.

1. S. Lisenkov, *et al.*, *Phys. Rev. B*, **79**, 024101, (2009).

A-16

Solvation Structure and Dynamics of Room-temperature Ionic Liquids

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Room-temperature ionic liquids have emerged as a new class of solvents that have several potential applications due to their unique properties (e.g., low volatility, large electrochemical window, high conductivity, etc.). One of their more exciting possibilities is their potential use in nuclear fuel reprocessing for the next generation of nuclear reactors. The selectivity of a given imidazolium ionic liquid depends strongly on the choice of the counter anion. In this contribution we will present new results using both static and time-resolved extended x-ray absorption fine structure (approx. 1-ns resolution) on a series of bromide-containing imidazolium salts. The static results provide detailed information of the solvation shell of the bromide ion and the time-resolved data shed light on the nature and chemical behavior of the lowest lying charge transfer band.

A-17

How to Extract Time-resolved Signal from Laue Diffraction by an Energy-chirped Hard X-ray Pulse: a Proposal

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A method to generate an energy-chirped hard x-ray pulse using a scheme based on overcompression of electron bunches is currently being developed at the Linac Coherent Light Source. These energy-chirped pulses are expected to reach 1–1.5% bandwidth at 8 keV and a subpicosecond temporal width. We propose to use these chirped pulses to study light-initiated reactions in biological macromolecules like myoglobin and photoactive yellow protein at ultrafast time resolution in the time domain from 100 picoseconds to 10 femtoseconds. One of the research and development areas required by this study is an effective numerical algorithm to extract time-resolved signal from Laue diffraction images produced by these chirped pulses. We will present a proposal of such an algorithm and some preliminary data.

A-18

Dynamic and Static Aspects of Bimodal Colloidal Suspension

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Structure and dynamics of bimodal suspensions of latex spheres in supercooled glycerol were investigated using coherent synchrotron radiation via small-angle x-ray scattering and x-ray photon correlation spectroscopy. The purpose of this work was determine the correlation between microstructure and observed dynamics for moderately concentrated bimodal colloidal suspensions with the size ratio of the constituent species close to five, which is large enough to show a well-pronounced depletion effect but not sufficient for the small spheres to fill exactly the space between the big ones. Measurements were performed for a series of samples spanning the full range of relative concentration ratios of large-to-small species. The arrangement of large particles was found to be well described in the frame work of the sticky hard spheres model. Strength of the attraction between large spheres is inversely proportional to their concentration. Outcome from the structure analysis is in good agreement with x-ray photon correlation spectroscopy data. All bimodal suspensions show dramatic slowing down of dynamics as compared to monomodal cases. This effect becomes less pronounced with increasing concentration of large spheres. Beside largest length scales at highest applied temperature, the intermediate scattering function followed stretched exponential scheme. Both characteristic time scale for particle motion and stretched exponent show nontrivial wave-vector dependence. Model assuming depletion effect driven phase separation and formation of monomodal domains was proposed to explain obtained data.

A-19

Nonlinear Strain Generation in Ultrafast Laser-excited Semiconductors

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We have investigated the laser fluence dependence of the lattice response of indium antimonide and gallium arsenide crystals to ultrafast laser absorption using time-resolved x-ray diffraction. In both materials, slow thermal cooling follows an initial acoustic strain impulse. For indium antimonide, where the laser photon energy is significantly above the band gap, we find that both acoustic and thermal lattice expansions increase linearly with increasing laser fluence. The band gap and photon energy are much closer in gallium arsenide where we find that while the thermal response remains linear with laser fluence, the magnitude of the acoustic impulse is highly nonlinear, exhibiting an initial saturation and recovery far below the laser damage threshold limit. Several hypotheses have been put forward of different nonlinear processes that could lead to this behavior. To place additional constraints on these models, we have upgraded the data acquisition system at APS 7-ID to record high-resolution diffraction lineshapes that can be directly compared to semiconductor strain models incorporating the transport of sound, heat, and charge.

A-20

Toward Correlating Structure and Function in Proteins: A LITR-XAS Study of the Photodissociation of Carbonmonoxymyoglobin

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A large portion of protein research has involved the use of ultrafast spectroscopic techniques to determine the correlation between structure and function in the photodissociation of carbonmonoxymyoglobin (MbCO), using this system as a model for protein dynamics as a whole. Accomplishing this goal requires the use of techniques with sufficient spatial resolution to measure the minute motions in the protein metal center associated with photoexcitation. Preliminary laser-initiated time-resolved x-ray absorption spectroscopy (LITR-XAS) experiments have been performed to determine the structural changes that occur in the iron heme center of MbCO upon photoexcitation. These experiments show a shift in Fe K-edge energy and the loss of a pre-edge peak, indicative of the loss of CO and motion of the Fe out of the heme plane. These results mark the first successful use of ultrafast XAS experiments to probe the structural dynamics of a protein sample, serving as a template for future studies on other proteins. Future experiments will focus on quantifying the structural changes in the protein and comparing with those in free heme to determine the effect that the protein matrix has on the photoexcited dynamics.

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A-21

Spectroscopic Characterization of the Water Oxidation Intermediates in the Ru-based Catalysts for Artificial Photosynthesis

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Utilization of sunlight requires solar capture, light-to-energy conversion, and storage. One effective way to store energy is to convert it into chemical energy by fuel-forming reactions such as water splitting into hydrogen and oxygen. Ruthenium (Ru) complexes are among few molecular-defined catalysts capable of water splitting. Insight into the mechanism of their action will help to design future robust and economically feasible catalysts for light-to-energy conversion.

We characterized structures and electronic configurations of intermediates of water oxidation by "blue dimer" *cis,cis*-$[Ru_2O_3(H_2O)_2(bpy)_4](PF_6)_4$ and recently discovered monomeric Ru-complexes [1]. Intermediates of water oxidation were prepared chemically by oxidation of Ru-complexes with defined number of Ce(IV) equivalents and freeze-quenched at controlled times. Changes in the oxidation state of the Ru atom were detected by x-ray absorption near-edge structure at Ru $L_{2,3}$- and K-edges. We demonstrate that both L and K-edges are very sensitive to changes in Ru oxidation state for Ru(II), (III), (IV) and (V) complexes and thus allow a clear assignment of Ru oxidation state in intermediates. Extended x-ray absorption fine structure at Ru K-edge demonstrated structural changes in intermediates. We detected considerable changes in distances for Ru-N and Ru-O interactions as well as changes in the angle of Ru-O-Ru fragment in "blue dimer" molecule.

1. J. J. Concepcion, J. W. Jurss, J. L. Templeton, and T. J. Meyer, "One Site is Enough. Catalytic Water Oxidation by $[Ru(tpy)(bpm)(OH_2)]^{2+}$ and $[Ru(tpy)(bpz)(OH_2)]^{2+}$," *Journal of American Chemical Society*, **49**, 130, 16462–16463, (2008).

A-22

Mirco-SXRF Imaging of Transition Metal Distributions in the Brains of Rats after Chronic Mn Exposure and Healthy Controls

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Although manganese (Mn) plays a vital role for normal development and body function of all mammals, excessive Mn exposure produces symptoms resembling those of idiopathic Parkinson's disease. Manganese is known to pass across the blood-brain barrier and interact with neurons. However, the knowledge on molecular mechanisms of its neurotoxicity remains incomplete. There are indications that exposure to Mn affects homeostasis of iron in the brain.

We used x-ray fluorescent imaging to quantitatively examine changes in distribution of biologically relevant metals (Ca, Mn, Fe, Cu and Zn) in selected areas in the brain (cortex, caudate putamen, globus pallidus, and hippocampus) in the condition of chronic exposure to Mn. We found that Mn accumulates preferentially in caudate putamen, globus pallidus, and hippocampus. Iron (Fe) content is elevated in most areas in the brain affected by chronic exposure to Mn. The largest difference in Fe content was determined inside ventricles—areas filled with choroid plexus cells and cerebrospinal fluid (CSF). This is in agreement with earlier studies that demonstrated considerable (+167% or threefold) increases of Fe in CSF. Thus, excessive accumulation of Mn in brain cells as well as altered Fe content may contribute to neurotoxic effect of Mn.

A-23

Design of Polarization Analyzer for RIXS

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Resonant inelastic x-ray scattering (RIXS) is a powerful x-ray scattering technique for the study of complex electronic excitations in correlated electron systems.

The photons scattered by the sample are collected by the instrument and changes of energy, momentum, and polarization can be measured thus characterizing the nature of the excitations. Only the first two are routinely measured by existing instruments due to technical challenges and low count rates. However, the outgoing polarization provides crucial symmetry information about the intermediate states involved. It also can differentiate overlapping spectral features, and greatly reduce the elastic background at same time.

We will present preliminary results with a flat graphite polarization analyzer, as well as calculation and designs for improved optics. The effective polarization factor, effective reflectivity and focusing for different shapes also have been calculated.

A-24

Vibrational Anisotropy from Single-crystal NRVS of [Fe(OEP)(NO)]

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Nuclear resonance vibrational spectroscopy (NRVS) on single crystals of [Fe(Por)(L)] has traditionally been done with the incident x-ray beam either perpendicular or parallel to the mean porphyrin plane. Iron out-of-plane modes are observed when perpendicular and iron in-plane modes when parallel. Previously, the parallel orientation was made without specifying a radial angle, so any inherent directional dependence of in-plane mode intensities could not be observed. We conducted NRVS on single crystals of [Fe(OEP)(NO)] oriented so that spectra along any in-plane direction were achievable. In-plane anisotropy was found to be axial-ligand directed with the iron motion of nearly all modes parallel or perpendicular to the Fe-NO plane. Comparisons to density functional theory-predicted spectra allow for mode assignments and demonstrate the sensitivity of NRVS to very small iron displacement. Vibrational modes can now be more accurately categorized as purely in plane, purely out of plane or an admixture of the two. This more exhaustive characterization of vibrational modes may be useful in the elucidation of the heme-NO binding associated with biological signaling.

A-25

Morphological Phenotyping of Zebrafish Mutants Using Synchrotron-based Micron-scale Computed Tomography

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The study of vertebrate organisms with single-gene defects is important for probing the function of genes and for translating such work to analogous defects in humans. Because these defects often operate at the level of single cells, their documentation requires information from the length scale of single cells to the length scale of the entire organism. Optical modalities are ideal for probing small organisms, tissues sections, or even single cells in two and three dimensions; however, optical methods are severely limited by tissues thicker than ~200 microns and rarely shed light on the continuum of length scales between the single cell and the whole organism within a single data set. As an alternative to optical methods, we have employed the use Argonne National Laboratory's Advanced Photon Source to conduct micron-scale computed tomography of the small vertebrate model system, the zebrafish. Reported are the results of scanning larval and juvenile zebrafish, demonstrating the continuum of length scales from the whole organism down to single cells within a single scan. As part of the zebrafish phenome project, it will be possible to quantitatively document morphological changes within the zebrafish caused by mutation in each protein-encoding gene (~20,000 genes).

A-26
SAXS Study of Protein Cold Denaturation

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We present the first comprehensive study of the cold denaturation of proteins using small-angle x-ray scattering (SAXS). The radius of gyration of equine cytochrome-c is determined at varying salt conditions from -25 to 60°C and compared to a two-state model. The incorporation of a temperature-dependent pH and solvent dielectric constant is critical to model electrostatic interactions over this broad temperature range and properly predict the observed protein stability from sequence. At suitable conditions, the protein can be made to increase in size by nearly 9 Å (over 60% of its native radius of gyration) when dropped in temperature from 0 to -25°C. This result is promising for future studies of ultrafast protein folding using time-resolved SAXS where initially cold denatured protein will be suddenly jumped in temperature by an infrared laser to initiate folding.

A-27
Cytochrome c Compressibility Measured with Two Synchrotron-based Techniques

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We used two synchrotron-based techniques (nuclear resonance vibrational spectroscopy and inelastic x-ray scattering) to measure the adiabatic compressibility of cytochrome c. Compressibility characterizes three interconnecting properties of a protein: dynamics, function, and structure. This is the first report of the compressibility of any material measured with this method. Unlike the methods previously used (which produced contradictory results) this novel approach probes the protein globally, at ambient pressure, does not require the separation of protein and solvent contributions to the total compressibility, and uses samples that contain the heme iron, as in the native state.

A-28
Solving Various STR Structures of Dystrophin Protein

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Muscular dystrophy is a debilitating disease that varies in severity but is the result of either the complete absence of or a modified malfunctioning form of the dystrophin protein. Dystrophin is a rod-shaped protein consisting of amino- and carboxy- terminal binding domains linked by a central rod composed of 24 copies of a spectrin-type repeat (STR) motif and four non-homologous regions termed hinges. Historically it has been thought that STRs are repeating and homologous. However, recent work has shown that various STRs exhibit markedly different stabilities, and that some regions appear disordered and flexible and suggest that STRs may function as hinge-like regions. We have obtained protein crystals of the d5 and d16–17 STR's from the human dystrophin protein and collected < 2-Å resolution data at SER-CAT. Molecular replacement methods have proven unsuccessful to date as a suitable model remains elusive. Multiple isomorphous replacement with heavy atoms has also proven fruitless as the soaked P1 crystals have lacked the redundancy and isomorphism required for single-wavelength anomalous dispersion and multiwavelength anomalous diffraction phasing. Alternative approaches are currently underway to overcome these obstacles and produce the first high-resolution STR structure and glean direct evidence as to the role of these STRs.

A-29

Probing Radiation Damage in Protein Crystals with Micron-sized X-ray Beam

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X-ray-induced radiation damage to macromolecular crystals remains a major hindrance in diffraction data collection and interpretation of results in structural biology. Energy deposition by photoelectrons, emitted during irradiation of crystals, is thought to be a primary cause of radiation damage at data collection temperatures (~100 K). Photoelectrons are emitted preferentially along the polarization vector and according to Monte-Carlo simulations at 15 keV will be reabsorbed 4–4.5 microns from the point of emission. Subsequently, with a small enough beam, photoelectrons will carry energy outside the footprint of the beam resulting in less damage to the sample. Studies of radiation damage as function of beam size indicated that smaller beams indeed reduce damage to the diffracting volume of a protein crystal. Radiation damage as function of dose and of distance from the beam center was also mapped with a 1-μm beam at two energies: 15.1keV and 18.5 keV. Concurrent with the theories, the damage was higher along the polarization vector than in perpendicular direction, the width of the damaged area was 2–3 times larger than the incident beam size, and the damage did not transfer beyond 4–5 μm from the incident beam center.

A-30

Rapid Temperature Switching for Time-resolved Measurements

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The ability to probe materials and reactions in real time under real operating conditions is pivotal to understanding of their structure and functional behavior. Towards this goal it is important to develop appropriate sample environments generating non-ambient operating conditions. In particular, probing the kinetics and mechanism for a reaction rely on the ability to initiate the process on a time scale that is fast relative to the reaction itself. Here we present apparatus that enables the rapid switching of temperature or reactive gas streams to initiate and characterize solid-state reactions.

A-31

Activity and Ordering of Mixed Phosphatidylethanolamine/Dihydrocholesterol Monolayers

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Cholesterol is thought to be important for the structure and assembly of lipid rafts, and its interaction with other membrane lipids has been a topic of great research interest. The interactions between 1,2-dimyristoyl-sn-glycero-3-phosphoethanolamine (DMPE) and dihydrocholesterol (Dchol) in Langmuir monolayers are probed using fluorescence microscopy (FM), beta-cyclodextrin (CD) desorption assays, and grazing-incidence x-ray diffraction (GIXD). Similar to our previous results for 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC)/Dchol monolayers [1], FM and CD assays show two regimes for the DMPE/Dchol system. Short-ranged lateral ordering was observed using GIXD that was also consistent with our recent work on sphingomyelin (SM)/Dchol monolayers [2]. We investigate how the smaller headgroup of DMPE effects Dchol chemical activity and lateral structure compared to monolayers of Dchol with DMPC or SM.

1. *Biophys. J.*, 93, 2038–2047, (2007).
2. *Phys. Rev. Lett.*, **103,** 028103, (2009).

A-32

Organic Nanoscale Materials for Energy and Charge Transfer

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The development of efficient artificial systems for solar energy conversion is essential for creating a sustainable energy source. Natural photosynthesis relies upon non-covalent interactions between chromophores to regulate the direction, yield, and rate of energy and electron flow. In order to create solar materials, we employ bio-inspired molecular designs based on self assembly of organic chromophores that are economical and versatile yet sufficient to mimic the functionality of the natural system. Small-angle and wide-angle x-ray scattering (SAXS/WAXS) experiments are used to elucidate aggregate structures in solution. Coupled with transient optical spectroscopy and electron paramagnetic resonance spectroscopy, these data allow correlation between supermolecular structures and photophysical properties of chromophores studied.

For example, perylene-3, 4-dicarboximide (PMI) was covalently linked to melamine (Mel) through a two-phenyl spacer and shown to form large-scale aggregates through hydrogen bonding, pi stacking, and microsegregation. SAXS/WAXS results in methylcyclohexane indicate that the observed aggregation is the formation of multi-(PMI-Mel)subunit cylindrical structure. Optical experiments further support large-scale structural aggregation through the observance electron transfer (ET) from Mel to PMI as seen only in aggregating solvents (i.e. MCH). No ET is observed when PMI-Mel is assumed to be in its monomeric form, indicating that aggregate ET is likely intermolecular.

A-33

Using SAXS to Study the Storage Potential of Supercritical Carbon Dioxide in Deep Rock Formations and Saline Aquifers

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Capture and sequestration of carbon dioxide gas are important processes for the reduction of carbon dioxide in the Earth's atmosphere. A few potential storage sites within the Earth's crust include deep geological formations consisting of porous rocks such as sandstone or basalt and saline aquifers contained within these formations. We are experimentally investigating the carbon dioxide storage potential of such rocks using time-resolved small-angle x-ray scattering. Our *in situ* experimental approach is to utilize a sample cell that can be pressurized with supercritical carbon dioxide while the rock sample contained within is exposed to an intense x-ray beam every few minutes for a period of 12 to 15 hours. The resulting time-resolved x-ray scattering curves for the rocks at these conditions yield quantitative information on the filling of pores and the evolution of pore sizes. In this way, quantitative analysis of the amount of carbon dioxide in the rock as well as swelling and changes in pore structure as a function of time may be determined *in situ*. Further, the potential retention of carbon dioxide in these samples, following pressurized injection, has been determined. Data for sandstone, shale, and clay rocks will be shown.

A-34

Evidence for an Electron Nematic Phase Transition in Underdoped Iron Pnictide Superconductors

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High-temperature superconductivity often emerges in the proximity of a symmetry breaking ground state in strongly interacting electronic materials. In the case of the superconducting iron pnictides, in addition to the antiferromagnetic groundstate of the parent compounds, an ubiquitous but small structural distortion breaks the crystal's C4 rotational symmetry in the underdoped part of the phase diagram. Here we report measurements of the in-plane resistivity anisotropy of detwinned crystals of the representative iron pnictide $Ba(Fe_{1-x}Co_x)_2As_2$. We find that the resistivity along the shorter b-axis develops an insulating temperature dependence, while the resistivity along the longer a-axis remains metallic. This conspicuous resistivity anisotropy bears witness to a dramatic electron nematic phase transition, with consequences for the electronic structure and also the superconducting pairing mechanism.

A-35

Magneto-structural Phase Diagram for $LuFe_2O_4$

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In this work we will present magnetization measurements and field-dependence diffraction on highly stoichiometric $LuFe_2O_4$ single crystals and the related complex magnetic-temperature phase diagram. The magnetic behavior of $LuFe_2O_4$ depends very sensitively on its oxygen stoichiometry; for high-quality samples, the compound shows a sharp antiferromagnetic transition at T_N ~240K. A metamagnetic transition between a ferri- and antiferromagnetic phase occurs below T_N by applying magnetic fields in the c direction. This transition does not involve any structural changes. Below 170K, frequency-dependent AC susceptibility suggests a phase with "glassy" dynamics. This glassy phase is separated from the rest of the phase diagram by a transition with structural component. The complex magnetic phase diagram likely derives from combined ferri- and antiferromagnetic clusters and a structural distortion. Neutrons also indicate a magnetic cluster structure with shorter correlation length for the "glassy" state. The latter accounts for an apparent kinetic hindrance of a transition between two of the phases, leading to a very high value of coercivity >9T at low temperatures. The structural transition has also a time dependence on the scale of hours.

A-36

2-Magnon Peak in $La_{2-x}Sr_xCuO_4$ Observed with RIXS at the Cu K-edge

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We present a comprehensive study of the temperature and doping dependence of the 500 meV peak observed at q=(π0) in resonant inelastic x-ray scattering (RIXS) experiments on La_2CuO_4. The intensity of this peak persists above the Néel temperature (T_N=320K), but decreases gradually with increasing temperature, reaching zero at around T=500K. The peak energy decreases with temperature in close quantitative accord with the behavior of the two-magnon B1g Raman peak in La_2CuO_4 and with suitable rescaling, agrees with the Raman peak shifts in $EuBa_2Cu_3O_6$ and K_2NiF_4. The overall dispersion of this excitation in the Brillouin zone is found to be in agreement with theoretical calculations for a two-magnon excitation. Upon doping, the peak intensity decreases analogous to the Raman mode intensity and appears to track the doping dependence of the spin-correlation length. Taken together, these observations strongly suggest that the 500 meV mode is magnetic in character and is likely a two-magnon excitation.

A-37

In Situ XAFS Characterization of the Nanostructural and Electronic Properties of Pt/C and PtNi/C as Cathode Catalysts in PEMFCs

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The electrocatalysis enhancement of Pt-based binary alloys (PtM), in comparison of pure Pt, has been systematically investigated by conducting *in situ* x-ray absorption spectroscopy characterization of PtNi/C and Pt/C nanoparticles as cathode catalysts in an Air-Breathe fuel cell. We observed that Pt-O formation at higher operation potentials was significantly inhibited in PtNi alloys, which prevents free active sites from being poisoned by oxygen adsorbate, leading to better reactivity. Two effects were confirmed to account for the inhibition. Electronic effect is the Ni reduces the bond strength of Pt-O by modifying the physical (geometric effect) and electronic (ligand effect) properties of Pt. Its side effect, an increase of open circuit voltage, was also detected. Attraction effect is that the more reactive alloying element (Ni) attracts and holds oxygen adsorbate more strongly than Pt. Furthermore, the reactivity trends among transition elements as alloyed metal in PtM was simulated by Feff calculation. Our development of the electrocatalysis enhancement mechanism can be a guidance to search for powerful catalysts in replacement of Pt.

A-38

Magnetoelastics of a Spin Liquid: X-ray Single-crystal Diffraction Studies of $Tb_2Ti_2O_7$ in Pulsed Magnetic Fields

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High-resolution single-crystal x-ray diffraction measurements of the frustrated pyrochlore magnet $Tb_2Ti_2O_7$ have been performed using a novel low-temperature pulsed-magnet system. This unique instrument allows for a thorough characterization of structural degrees of freedom to temperatures as low as 4.4K and in applied magnetic fields as high as 30 tesla. We show that $Tb_2Ti_2O_7$ manifests a number of intriguing structural effects under the application of magnetic fields, including strongly anisotropic giant magnetostriction, a restoration of local symmetry in low magnetic fields, and ultimately a structural phase transition in high magnetic fields. A treatment of spin-liquid physics in this compound based on spin degrees of freedom alone seems simplistic as these results show that magnetoelastic coupling plays a significant role in $Tb_2Ti_2O_7$ at low temperatures.

A-39

X-ray Characterization of Rhenium Speciation in Supported Pd/Re Catalysts for Aqueous Perchlorate Treatment

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Perchlorate is a widespread contaminant detected in groundwater and surface water throughout the United States. Strict drinking water regulations are anticipated due to its health effects and growing public concerns, so there is a need for technologies that effectively treat perchlorate-contaminated water. Recent work at the University of Illinois demonstrates that heterogeneous palladium/rhenium (Pd-Re) bimetal catalysts reduce aqueous perchlorate to chloride using hydrogen as electron donor at ambient temperature and pressure orders-of-magnitude faster than other chemical reduction processes. The proposed mechanism for catalytic perchlorate reduction assumes redox cycling of Re on the surface of the catalyst, and recent work suggests that immobilization and stability of Re is dependent on solution redox condition. This presentation will describe recent efforts to characterize the Re surface and bulk speciation on the bimetal catalyst under different operating conditions (e.g., exposure to hydrogen- versus air-saturated water) and assess how Re coordination is influences by these variables. The results of x-ray photoelectron spectroscopy measurements and preliminary analysis of Re L3-edge x-ray absorption near edge structure and extended x-ray absorption fine structure data collected at the Advanced Photon Source will be presented, and results will be used to interpret the molecular properties of catalytically active Re species.

A-40

Optimizing High-pressure Pair Distribution Function Measurements in Diamond Anvil Cells

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Pair distribution function (PDF) methods have great potential for the study of diverse high pressure phenomena. However, the measurement of high quality, high resolution x-ray PDF data (to $Q_{max} > 20$ Å^{-1}) remains a technical challenge. Here we present an optimized approach to measuring high-pressure total scattering data for samples contained within a diamond anvil cell (DAC). This takes into account the coupled influences of instrument parameters (photon energy, detector type and positioning, beam size/shape, and focusing), pressure cell parameters (target pressure range, DAC type, diamonds, pressure transmitting media, backing plates, pressure calibration), and data reduction, on the resulting PDF. The efficacy of our approach is demonstrated by the high-quality, high-pressure PDFs obtained for representative materials spanning strongly and weakly scattering systems and crystalline and amorphous samples. These are the highest resolution high pressure PDFs reported to date, including for α-alumina (to $Q_{max} = 20$ Å^{-1}), $BaTiO_3$ (to $Q_{max} = 30$ Å^{-1}), and pressure amorphized zeolite (to $Q_{max} = 20$ Å^{-1}).

A-41

Pressure Dependence of Atomic Structure, Magnetism, and Elastic Properties of Fe_3C: a Combined Synchrotron X-ray Diffraction and Nuclear Resonant Scattering Study

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In this work, we demonstrate the feasibility of studying atomic structure, magnetism, and elastic properties with combined synchrotron x-ray diffraction (XRD) and nuclear resonant scattering methods on Fe_3C at beamlines 3-ID, 11-BM, 13-BM, and 13-ID at the Advanced Photon Source. XRD probes the long-range order of a material and is a classic method for measuring density. From density-versus-pressure data, elastic properties such as bulk modulus and its pressure derivative can be derived from equation-of-state (EoS) fitting. For EoS fitting, we developed a Java program, FitEoS, which is available to the public. Nuclear resonant scattering technique includes synchrotron Mössbauer spectroscopy and nuclear resonant inelastic x-ray scattering methods. These methods provide information on magnetic properties and lattice vibration. A combination of these methods provides a unique understanding on the behavior of a material under pressure from various perspectives. In this study, we observed two discontinuities in the XRD compression data on Fe_3C. These two discontinuities were suggested to be related to magnetic and electronic transitions from our nuclear resonant scattering measurements. The implications about the Earth's inner core inferred from this work will also be discussed.

A-42

Effect of High Pressure on the Electronic Structure of Rubidium Evidenced from Synchrotron X-ray Absorption and Diffraction Studies

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It's well known that pressure can be used to tune the electronic structure of elements such that they exhibit altered reactivity and can form new compounds. The fundamental change in the electronic structure of alkali metals upon compression has been proclaimed in many works. It has been established that energies of s levels in these metals increases more rapidly under pressure than those of d levels, leading to a transition to a bonding state that contains only d electrons. The new electronic state opens the possibility of the formation of novel compounds, e.g., alloys of alkali and transition metals. The latter are expected to form Lave's phases, which exhibit many interesting magnetic and electronic properties and considered for the potential use as hydrogen storage materials.

So, it becomes of great importance to perform studies on response of the electronic structure of alkali metals to applied pressure. We present the synchrotron x-ray absorption and diffraction studies on rubidium under high pressures to above 50 GPa. The results show, for the first time, the signatures of pressure induced electronic structure changes of an alkali metal. The simultaneous use of absorption and diffraction techniques provided an insight into electronic-structural interrelation for the case.

A-43

Iso-structural Phase Transition of Iron-based Superconductor $FeSe_{0.82}$ under High Pressure

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See poster for abstract.

A-44

High Pressure Synergetic Consortium Overview

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High-pressure (HP) synchrotron radiation (SR) research has been regarded as one of the most impactful directions in physical science and a top priority in leading synchrotron facilities around the world. The integration of HP and SR may potentially lead to the accomplishment of grand challenges in various disciplines. Current HP-SR activities are conducted with the combination of dedicated HP-SR beamlines and widespread HP research at general-purpose SR beamlines. These activities are invaluable for the recent advances in high pressure research but still insufficient and ineffective for realizing the grand challenges. The integration of HP and SR would therefore require a special infrastructure to harness the two fast-moving giants and reach their full potential.

The recent established High Pressure Synergetic Consortium (HPSynC) at the Advanced Photon Source will focus on novel, high-reward, high-pressure synchrotron radiation science and technology that are very difficult and challenging to develop by synchrotron beamline personnel and user groups, and to make the novel high-impact science and technology available to general HP-SR users. This poster will give a broad overview of overall effort, focusing developing areas and science highlights from the achievement.

A-45

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

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See poster for abstract.

A-46

Latest BluIce-EPICS Developments Including Collection along a Vector

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Collection of partial data sets at multiple sites on a crystal using the GM/CA CAT "mini-beam" enables structure solution for radiation-sensitive samples. This radiation damage mitigation technique, which many had done manually, is now automated and well integrated in BluIce-EPICS.

A vector is defined by centering two endpoints using standard sample controls. Additionally, four parameters define collection along the vector: distance between collection sites, frames per site, total number of sites, and overlap frames per site. During collection, the vector and collection sites are shown projected over the live video stream from the high-resolution on-axis camera. This aids both in defining the vector and in monitoring the collection progress.

Collection along a vector is also used to screen for potential collection sites. If a single frame is taken at each site with high x-ray beam attenuation, the damage will be minimal, and the diffraction quality can be determined by visually examining the diffraction images. For some crystal types, this may be preferred over the 2D rastering capability in BluIce-EPICS, since the vector is defined in arbitrary 3D space.

Other BluIce-EPICS advances in 2010 include a fully WebIce-connected Java screening tab, rastering history (multiple raster run tabs), and rastering controls synchronization between multiple instances of BluIce-EPICS.

A-47

The Optics Upgrade of 17-ID Crystallographic Beamline

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The 17-ID macromolecular crystallography beamline has recently been upgraded to a micro-focused high-flux diffraction beamline. With newly purchased cryo-cooled double-crystal monochromator and bimorph KB mirror systems, the full undulator beam is focused into a 30-µm (V) by 65-µm (H) beam at the sample location, with 2-µm beam position stability under beam positional feedback. The beamline is fully operational in the designed energy range from 6 keV to 20 keV. This high-flux, micro-focused, and stable beamline combined with an upgraded end-station makes 17-ID an excellent high-throughput protein crystallographic beamline. Optical equipment, optical tuning experience, and results will be shared in this poster.

A-48

Research and Development of Monochromators and Crystal-based Optics at APS

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The Crystal Optics section of the Optics and Detectors Group at the APS is for design, fabrication, and characterization of monochromators, crystal-based x-ray optical components, and related research and development. In this presentation, we will introduce our facilities, capabilities, and future upgrade and improvement plans on the development of crystal-based optics. We will also present some of our recent research work, including (1) fabrication of novel crystal monochromators, (2) theoretical studies of virtual source spread by monochromators and its effect on focusing, coherence, and imaging, (3) computerized Laue method for accurate determination of crystal orientations, and (4) computer programs for designing and modeling of high-resolution monochromators.

A-49

Recent Progress in X-ray Detector Development

David Kline and Steve Ross

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We report on progress in our x-ray detector work, which we do together with several small companies (Small Business Innovative Research process, etc.), universities, and other national laboratories. We have several on-going developments, including a timing and control system for 7-ID single-element avalanche photo diode (APD) detectors, APD array detectors for sub-nanosecond timing and fast counting, a 40 x 6 direct detect thick CCD that will readout at the APS P0 rate (10^6 frames/sec), a silicon strip detector for fuel spray applications, and others. We continue our work with application-specific integrated circuits working with several CAD packages and in collaboration with students at Georgia Tech and Northern Illinois University (NIU). One application-specific integrated circuit project just beginning here involves ultrafast electronics with goals of timing in the picosecond range. We continue to work in areas of device physics (typically working in university clean rooms for fabrication) for the benefit of both our development programs and the NIU university curriculum.

A-50

A Compact-Short Focal Distance Bent Crystal Laue Analyzer for Copper Speciation Studies

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X-ray fluorescence emission is used for x-ray absorption determinations of diluted biological samples like tissue sections and cultured cells, where metal concentrations are typically a few parts per million. This low metal concentration required a high-flux incident beam capable of generating enough fluorescence x-rays of the element of interest to be detected. The main drawback in this configuration is the high intensity of the scattering signal from the supporting matrix. Measuring copper Kα fluorescence from tissue samples, for instance, can be critically masked by the strong scattering signal, reducing the sensitivity level and potentially saturating conventional solid-state photon-counting detectors. A bent crystal Laue analyzer (BCLA) is a suitable alternative tool to fluorescence x-ray absorption spectroscopy measurements when high rejection of the unwanted scattering signal is required. We have designed a compact-short focal distance log spiral BCLA for Cu Kα fluorescence measurements. A solid aluminum bender provides the proper bending shape for the silicon crystal wafer to preserve the correct angle of incidence between the fluorescence x-rays and the diffraction planes. The BCLA is mounted on an XY motorized linear stage for easy alignment. The size of the vertical beam profile is a critical parameter for the BCLA energy resolution (~18 eV @ Cu Kα line) thus a microbeam of ~5 microns FWHM and intensity of $1.0 \cdot 10^{12}$ photons/sec is used at the

18-ID BioCAT beamline. The scattered (nondiffracted) x-rays travel straight through the crystal and are blocked by molybdenum soller-slits. The diffracted x-rays are detected by a SDD detector and the corresponding Kα peaks are discriminated by preselected single channel analyzer regions of interest. This configuration allows reducing even more of the background signal. The designed BCLA will be used on copper speciation studies in biological samples with specific applications to cancer biology.

A-51

Development of a Grazing-incidence Insertion Device X-ray Beam Position Monitor

Soonhong Lee, Patric Den Hartog, Bingxin Yang, and Glenn Decker

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Beam stability is always a concern in synchrotron light source facilities, and accurate and stable x-ray beam position monitors (XBPM) are key elements in obtaining desired user beam stability. Currently, APS is preparing to upgrade its facility to increase productivity and to provide better beam stability. To achieve beam stability of 3.0 μm horizontally and 0.3 μm vertically within a frequency band up to 200 Hz, a grazing-incidence insertion device x-ray beam position monitor (GRID-XBPM) is proposed for the insertion device beamline front ends instead of the current photoemission-based XBPM. In principle, if we measure the distribution of the x-ray beam footprint on a front-end component, its x-ray fluorescence footprint can be imaged to infer the position of the photons. Users often discard more than 50% of the insertion device beam power outside of the monochromatic beam. Thus, our conceptual design of the GRID-XBPM is to slice the collimator into right and left halves and to displace them along the beam direction with a grazing-incidence angle to increase the beam footprint and to provide room for the beam footprint measurement. The development of a GRID-XBPM will be summarized, which includes the thermal simulation results and the design of the relevant upstream and downstream components of the GRID-XBPM such as a fixed mask and a photon shutter.

A-52

Real-time Processing of XPCS Data on a Field-programmable Gate Array

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In x-ray photon correlation spectroscopy (XPCS) experiments at the APS, a high-speed area detector is used to capture x-ray data at 100's of frames per second and data rates of up to 125MB/sec. Because of this, large-data-rate camera calibration data cannot be applied to raw images, disk drives cannot keep up to store the data, and extremely large and unmanageable datasets are generated. To mitigate these problems, a field programmable gate array (FPGA), a type of digital integrated circuit, was developed at the APS to perform image processing in real time. The FPGA, residing on a commercial frame grabber, performs real-time image processing before the data enters the computer. In this way, the computer's internal busses are not bogged down, disk drives can keep up with the camera, and manageable data sets are saved. The FPGA, performing image averaging, noise measurements, dark subtraction and compression in real time, is controlled by an EPICS interface, and allows the fast camera to collect XPCS data continually.

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.

A-53

Multilayer Monochromators for 5 keV to 20 keV Photon Energy Range

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Seven multilayer structures were deposited on flat silicon substrates by using conventional magnetron sputtering technique [1]. Parameters of the structures are the following:

1. Ru/B_4C, d = 2.83 nm, N = 170, gamma = 0.41
2. Ni/B_4C, d = 4.52 nm, N = 100, gamma = 0.47
3. SiC/B_4C, d = 5.3 nm, N = 300, gamma = 0.3
4. V/B_4C, d = 3.02 nm, N = 300, gamma = 0.26
5. V/B_4C, d = 2.3 nm, N = 500, gamma = 0.28
6. T/B_4C, d = 4.01 nm, N = 250, gamma = 0.3
7. W/B_4C, d = 1.5 nm, N = 300, gamma = 0.3

The multilayers were tested at the Swiss Light Source Optics beamline (X05DA). It is a bending magnet beamline working in the range between 5.5–22.5 keV, with a flux of >1 x 10^{11} ph/sec (@ 10 keV/400 mA) [2]. Silicon photodiode (AXUV100) with a quadratic area of 1 cm^2 was used as a detector. A double-slit system with slit size of 0.5 x 0.5 mm was installed between the samples and the vacuum pipe. Software automatically set the energy and the corresponding Bragg angle by using multilayers d-spacing values as one of input parameters. Scan tool adjusted the Bragg angle to the given energy and made rocking curve scans around the Bragg peak.

Peak reflectivity and resolution of the multilayer structures were measured. A good correspondence was found between calculated and experimental data.

1. Y. Platonov et al., *Proc. of SPIE*, **3152**, 231 (1997).
2. U. Flechsig, A. Jaggi, S. Spielmann, H. A. Padmore, and A. A. MacDowell, *Nuclear Instruments and Methods in Physics Research A*, **A609**, 281–285 (2009).

A-54

areaDetector: New Features for Release 1–6

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areaDetector Release 1–6 provides many enhancements to the plugins available in the EPICS areaDetector module, including:

- NDPluginStats plugin. This new plugin calculates statistics on an array, including basic statistics, centroid position, and width. It also computes X and Y profiles including average profiles, profiles at the centroid position, and profiles at a user-defined cursor position.
- NDPluginProcess plugin. This new plugin does arithmetic processing on arrays, background subtraction, flat field normalization, offset and scale, low and high clipping, recursive filtering in the time domain, and conversion to a different output data type.

- NDPluginOverlay plugin. This new plugin adds graphic overlays to an image. It replaces the "Highlight ROIs" function that was previously provided in the ROI plugin. However, it is much more general and can be used to display not only ROIs but multiple cursors, user-defined boxes, etc.

- NDPluginROI. The new version is much simpler with many functions moved to new plugins. One new function has been added—the ability to divide the array by a scale factor, which is useful for avoiding overflow when binning or converting data types.

- NDPluginColorConvert. Added conversions from mono to RGB1, RGB2, and RGB3, and from RGB1, RGB2, and RGB3 to mono.

A-55

Performance of Single- and Four-element Large-area Silicon Drift Detector X-ray Spectrometers for Synchrotron Applications

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Advanced x-ray spectroscopy using synchrotron beams, such as extended x-ray absorption fine structure and x-ray absorption near-edge structure are powerful means for material studies in a wide range of fields such as chemistry, biology, surface and material sciences, geology, and environmental science as well as the state-of-the-art nanoscience. These applications typically require large area detectors or detector arrays with a high-count-rate capability and, for some of synchrotron applications that require magnetic fields such as in techniques utilizing the x-ray magnetic circular dichroism effect, the detector performance must also not be compromised by the magnetic fields. A silicon drift detector (SDD) offers a large active area and excellent energy resolution together with a high-count-rate capability for a wide variety of industrial and scientific applications [1, 2].

We have developed the Vortex® SDD that has a large active area of ~ 45 mm^2, fabricated on ~0.35-mm thick, high-resistivity n-type silicon. These SDDs operate with thermoelectric cooling and feature excellent energy resolution (<130 eV FWHM at 5.9 keV and optimum peaking time). They also exhibit a very short signal rise time (< 100 ns) allowing pulse processing using very short peaking times (~0.25 μs) to achieve very high signal throughput (300–500 kcps output rate). Based on this SDD design we have developed single- and four-element x-ray spectrometers (the Vortex-EM® and Vortex-ME4™) that utilize SDDs installed directly on thermelectric coolers with the heat removed through an innovative heat pipe heat transfer system. This design enables us to develop customized x-ray spectrometers with a detector snout length up to 800 mm for a variety of synchrotron applications, including an ultra-high-vacuum compatible version. The spectrometers utilize the X-Ray Instrumentation Associates digital pulse processors, in particular the 4-channel DXP-xMAP that, in conjunction with the National Instruments PXI/Compact PCI module, offers 4 MB on-board high-speed memory and ~100 MB/s data transfer speed. Performance data of the Vortex® spectrometers, including performance in a high magnetic field, will be presented.

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A-56

Stiffness and Resolution Test of the APS T8-31 Vertical Nanopositioning Weak-link Stage

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Precision ball-bearing- or roller-bearing-based positioning stage systems provide large travel range. However, it is not possible to meet sub-nanometer positioning resolution, high tilting stiffness, and sub-microradian straightness of trajectory repeatability with a single guiding system. It has always been a dream to have a compact single flexure stage to cover a large travel range with very high positioning resolution. Based on an advanced structure design using the laminar overconstrained weak-link technique, we have designed and constructed a 2D linear precision weak-link stage system for nanopositioning of a specimen holder for a nanofocusing system based on multilayer Laue lenses at APS sector 26 [1, 2]. This system provides sub-nanometer resolution coupled with sub-nanometer metrology at a travel range of several millimeters.

In this poster we present the preliminary stiffness and positioning resolution test results of the APS T8-31 vertical linear precision weak-link stage.

1. D. Shu and J. Maser, U.S. Patent application in progress for ANL-IN-09-024.
2. D. Shu, H. Yan, and J. Maser, to be published in the *Proceedings of SRI-2008*, *Nucl. Instrum. and Methods A.*

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A-57

Fast CCD X-ray Detector Collaborations between ANL and LBNL

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This poster presents an update on the fast CCD (FCCD) detector collaborations between the Advanced Photon Source (APS) at Argonne National Laboratory (ANL) and the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory.

The detector collaboration now consists of the development of two different x-ray detectors. The initial collaboration developed two 480 x 480 fast CCD detector, with a nearly column parallel readout. These detectors are in use at the APS and ALS with plans for it to be tested at the Linac Coherent Light Source. The poster will present improvements made to these detectors based on inputs from beam line scientist and users. This poster will also describe the next phase of this collaboration, which is to develop a 1-K frame transfer FCCD detector.

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A-58

Structure and Thermal Evolution of a Metallic Glass that Grows from the Melt through a First-order Transition

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In recent decades, the structures of metallic glasses have been documented with increasing precision, and yet our understanding of these materials is still at best incomplete. The most direct proof of this is the proliferation of partially successful models attempting to classify the underlying generalized structure. To approach a more fundamental understanding of metallic glasses, it can be illuminating to investigate with care the extreme examples of a materials class. We have investigated by means of high-resolution x-ray pair-distribution function methods the structure of an unusual metallic glass that grows from the melt by a first-order phase transition in the manner of a crystal, within a prescribed AlFeSi stoichiometry, but scatters as a metallic glass. Well-defined nano domains (1.2 nm) that closely resemble atomic arrangements within alpha-AlFeSi were identified. While the alpha phase is a crystalline approximant to the neighboring icosahedral phase, the motifs in the glass structure are quite distinct from those in the icosahedral phase. Under a 305°C isothermal anneal, the glass rejects aluminum and relaxes into a more stable, longer-range order configuration that persists for hours. The quality of this glass is such that it becomes possible for the first time to approach an understanding of intermediate range packing (i.e., ordering in the range of 1–3 nm). As the anneal continues, there is a peritectic reaction in which the rejected aluminum-plus-glass undergoes a first order phase transition to a crystalline phase entirely unrelated to the nano-ordered domains in the original glass, just as if no nano ordering existed.

A-59

Surface X-ray Diffraction Studies of Chemically Functionalized Epitaxial Graphene

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To realize the potential of graphene in next-generation technologies, graphene must be incorporated with a variety of materials to form devices. Recently, the growth of self-assembled organic monolayers on epitaxial graphene has shown promise in functionalizing the bare graphene sheet for additional post-growth chemistry. In this work, we present crystal truncation rod (CTR) studies of perylene-3, 4, 9, 10-tetracarobxylic dianhydride (PTCDA) on graphene. A model-independent vertical electron density profile the SiC/graphene interface is retrieved from the CTR data by means of Fienup-based analysis, and initial modeling of the PTCDA/Graphene/SiC system is reported. These results should help to elucidate the effect of the interface structures on the electronic properties in device-like conditions.

A-60

In Situ X-ray Standing Wave Atomic-scale 3-D Imaging of Catalysts Supported on Oxide: $VO_x/\alpha\text{-}TiO_2$ (110)

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Supported vanadia on oxides are important catalysts. If the atomic-scale surface structure of VO_x could be predicted, this would impact our understanding of numerous chemical processes. Thus, atomic layer deposition-grown VO_x on rutile (110) was used for finding the positions of vanadium (V) with respect to the support lattice and its sensitivity to the reduction-oxidation (redox) cycle. *In situ* x-ray standing waves (XSW) are used to determine the structure changes during the redox reaction. The results for 3/4 ML V show that V cations on the surface have different structures in the oxidized and reduced states. The 3-D atomic density maps created from XSW measurements show V cations surface site locations directly. X-ray photoelectron spectroscopy is used to correlate the V 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. With this model-dependent analysis, it shows that V at atop sites mainly participated in the redox reactions, indicating a top site could be a catalytic active site. The reversibility in redox reactions implies the important application in catalysis.

A-61

Probing Phase Transitions in Ultrathin Ferroelectric Films Using *In Situ* X-ray Scattering

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Ionic or electronic charge compensation at the surface of an ultrathin ferroelectric film is critical in determining the polarization structure of the complete film. Grazing-incidence x-ray scattering (GIXS) provides a powerful probe of the atomic displacements that underlie this polarization structure, enabling tracking of phase changes and domain formation in ferroelectric films. Performing GIXS measurements *in situ* allows us to study the roles that temperature and chemical environment play in determining the equilibrium polarization of a ferroelectric film. We present here studies of the equilibrium polarization in epitaxially strained $PbTiO_3$ films on conducting $SrRuO_3$ electrodes on (001) $SrTiO_3$ as a function of temperature, chemical environment, and film thickness. Changes in the ionic compensation at the surface due to changes in the oxygen partial pressure of the environment produce strong effects on the Curie temperature. High or low oxygen partial pressures (pO_2) produce outward or inward film polarization, respectively. At intermediate pO_2 values we observe a strong suppression of the Curie temperature. These experimental observations are qualitatively consistent with a thermodynamic model we have developed based on Landau-Ginzburg-Devonshire theory, which takes into account the chemical interaction between a gaseous environment and the polar surface of a ferroelectric.

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A-62

Ion Distributions at the Electrified 1, 2-Dichloroethane/Water Interface

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See poster for abstract.

A-63

Phase-contrast Imaging of Fatigue Crack Propagation through Grain Boundaries

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Single-crystal nickel-base superalloys are used in critical aeronautic and power-generation applications where they undergo very high-cycle fatigue. They can have complicated dendritic microstructures and grain boundaries. The microstructure can drastically influence fatigue-crack growth, a dominant failure mode, affecting the growth rate and morphology. Synchrotron x-ray radiography is ideal for imaging crack growth *in situ* and in real time, and the spatial coherence allows phase-contrast imaging of the interfacial topology of minimally opened cracks. We installed a custom-built ultrasonic-fatigue instrument at sector 32-ID for stable fatigue-crack growth in thin, dog-bone-shaped specimens. Samples were held with a mean tensile stress and cycled in tension at 20 kHz at $R = -0.1$. When crossing low-angle grain boundaries at large stress-intensity factors ($K > \sim 7$ MPa·m$^{1/2}$), cracks remained on {111} planes without changing their growth rate appreciably. At lower K near the crack-initiation threshold, cracks followed grain boundaries by shifting among planes in the {111} family. At large-angle grain boundaries, cracks were arrested, requiring larger K to propagate further. After traversing the boundary, they would frequently continue to propagate intergranularly or non-crystallographically. These results are directly relevant to the behavior of superalloys under actual operating conditions.

A-64

Anharmonic Phonon Behavior in BCC Iron at High Temperatures

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The phonon density of states (DOS) of bcc ^{57}Fe was measured from 300 K to 1223 K using nuclear resonant inelastic x-ray scattering. All phonons shift to lower energies (soften) with temperature. The different phonons soften in a similar way, at least below the Curie temperature of 1043K. At temperatures above the Curie temperature, the low transverse modes soften more rapidly. Interatomic force constants for the bcc phase were obtained by iteratively fitting a Born von-Karman model to the experimental phonon spectra. Trends in these force constants are used to interpret the thermal softening of the different phonons. The unusually large phonon anharmonicity of bcc Fe at elevated temperatures is discussed in terms of the temperature dependences of the first and second-neighbor interatomic force constants.

A-65

Phonon Densities of States of C15 Rare-earth Iron Compounds at Elevated Temperatures

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Nuclear resonant inelastic x-ray scattering measurements were performed on YFe$_2$, ErFe2, and TbFe$_2$ at elevated temperatures. The spectra were converted into phonon densities of states. A large decrease of the average phonon energy was observed with increasing temperature, consistent with a Grüneisen parameter of about four. This value is in good agreement with measurements of the thermal expansion, bulk modulus, and heat capacity from the literature. A preliminary Born-von Karman analysis of the inter-atomic force constants is presented.

A-66

Application of X-ray Absorption Spectroscopy and Inelastic X-ray Scattering Techniques to Study Lithium Ion Battery Materials

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Lithium ion batteries have attracted worldwide attention in the past decades due to their high energy and power density. They are currently widely used in portable devices and power tools and are poised for use in vehicular applications. It is important to understand the redox mechanism from a fundamental point of view to help develop new battery materials with improved properties. Various techniques such as nuclear magnetic resonance, conventional diffraction, short-range and long-range x-ray probes, Mössbauer spectroscopy, x-ray absorption fine structure, electron energy loss spectroscopy, etc. have been employed to date. The interesting redox chemistry occurs at light elements (Li, O, C) and TM 3d orbitals during Li-ion de/intercalation. We use x-ray absorption spectroscopy (XAS) and inelastic x-ray scattering (IXS) techniques to understand the charge compensation mechanism in operating batteries. The technique of XAS has been used to probe the local atomic and electronic structure details around specific probe atoms and provide key information on metal oxidation states and site symmetry. Using hard x-ray non-resonant IXS, which is a photon in/photon out technique, we demonstrate the ability to obtain truly bulk sensitive soft x-ray absorption—like information under normal battery operating conditions. The appropriate edge spectra of low-Z elements as well as metal were obtained using the lower-energy-resolution inelastic x-ray scattering spectrometer [1]. Recent studies of charge compensation in battery materials using XAS in Li$_5$FeO$_4$ [2] and IXS in LiCoO$_2$ will be presented.

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A-67

In situ Characterization of delta-Bi$_2$O$_3$ Stabilized by Epitaxial Growth on Single Crystal Oxide Substrates

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The cubic phase of Bi$_2$O$_3$, delta-Bi$_2$O$_3$, has the largest ionic conductivity of any oxide material, but is stable only from 725°C to 825°C. We observe that the delta-Bi$_2$O$_3$ phase is stabilized to room temperature by the epitaxial growth of nanostructures onto either (001)-oriented SrTiO$_3$ or (001) pseudo-cubic-oriented DyScO$_3$ single-crystal substrates.

The morphology of the nanostructures can be controlled by the miscut of the substrate. Synchrotron x-ray scattering observations at controlled temperatures and oxygen partial pressures reveal that the delta-Bi_2O_3 nanostructures are coherently strained to the substrates at room temperature, but have an unexpected superstructure. Annealing the nanostructures at 600°C causes gradual conversion of the (001)-oriented delta phase to an unidentified strain-relaxed phase. Recent experiments have shown that the delta phase can also be stabilized by the growth of epitaxial thin films on (111) Y_2O_3-stabilized ZrO_2 and (0001) alpha-Al_2O_3. Future work will combine the structural characterization of thin films with *in situ* electrical measurements to determine the source of the observed superstructure and give insight into the origin of the high ionic conductivity of delta-Bi_2O_3. This work will contribute to the fundamental understanding of the origin of superionic conductivity in oxide materials.

A-68

Argonne's Center for Nanoscale Materials

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The Center for Nanoscale Materials (CNM) at Argonne National Laboratory is a premier user facility providing expertise, instruments, and infrastructure for interdisciplinary nanoscience and nanotechnology research. The CNM user program provides access to equipment and technical expertise and is open to academia, industry, government agencies, and research institutes worldwide. There are three calls for proposals per year with deadlines on the APS cycle. No charge is assessed for work that is intended for the public domain. Access is also available on a cost-recovery basis for proprietary research that is not intended for publication.

The CNM's main research portfolio is organized around six key areas: (a) Electronic & Magnetic Materials & Devices, (b) Nanobio Interfaces, (c) Nanofabrication & Devices, (d) Nanophotonics, (e) Theory & Modeling, and (f) X-ray Microscopy. The main facilities include scanning probes, materials synthesis, computational nanoscience, and a cleanroom. The hard x-ray nanoprobe facility at sector 26 is jointly managed by the CNM and APS. It is a next-generation hard x-ray microscopy and x-ray imaging beamline with the highest spatial resolution in the hard x-ray range. These capabilities are of use to the broader nanoscience community in studying nanomaterials and nanostructures, particularly for embedded structures. It's key capabilities are: (a) scanning probe x-ray diffraction microscopy, (b) scanning probe x-ray fluorescence microscopy, and (c) full-field two-dimensional transmission imaging and tomography.

A-69

Structure/Function Studies of Atropine: Cucurbituril Complexes

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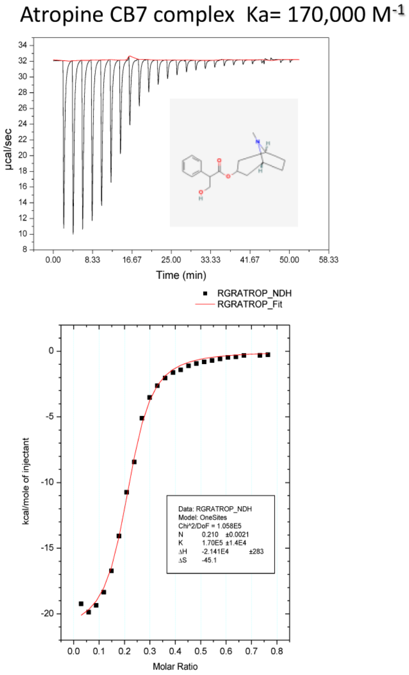
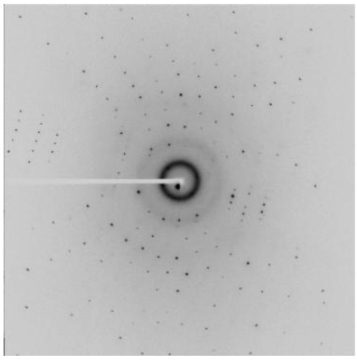
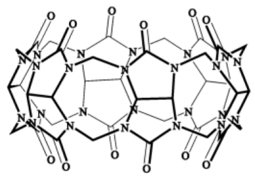
Introduction: There are over one million accidental exposures to organophosphate insecticides annually. Weaponized organophosphates (sarin) released in a Tokyo subway caused many casualties. The antidote, atropine, is difficult to titrate and overdose leads to an anticholinergic toxidrome. We are investigating whether excess atropine can be chelated with a readily available macrocycle to help control its side effects.







Cucurbit[7]uril

Methods: The binding constant of atropine to cucurbit[7]uril (CB7) was measured using isothermal titration calorimetry. Crystallization of the complex was achieved by controlled evaporation and x-ray diffraction data were measured at the Advanced Photon Source, Life Sciences Collaborative Access Team (LS-CAT) beamline 21-ID-D. The crystals are orthorhombic and diffract to 0.97-Å resolution.

Results: The binding constant of atropine to CB7 was determined to have a K_a=170,000 M^{-1}. The crystal structure reveals that the portal carbonyls of the cucurbituril interact with the nitrogen of the atropine tropane ring.

Significance: These studies will help design new drug antidotes. In the interest of homeland security, anesthesiologists and other first responders will have additional tools in their armamentarium to reverse the effects of weaponized chemicals.

A-70

In Situ Curvature and Stress Analyses for Sputtered WSi_2/Si Multilayer Thin Films on Silicon Wafers†

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Multilayered thin films with layer thicknesses of a few nanometers have numerous applications including x-ray mirrors, MEMS/NEMS structures, and semiconductor heterostructures. A particular application of interest presently is sputtering of multilayer-Laue lenses [1, 2]. Several x-ray optics groups are interested in multilayer thin films for focusing x-rays. Stress develops layer by layer during growth. High stress from the multilayers can have adverse effects; for example, it can result in delamination. We report on a study of wafer curvature of dc magnetron sputtered amorphous WSi_2/Si bilayers on Si wafers. Sputtering was interrupted and wafer curvature was measured *in situ* five times per layer with a laser-based optical system. Stoney's equation was used to analyze the curvature. The variables studied were: 1) substrate orientation, 2) substrate wafer rotation with respect to the wafer flat, and 3) Ar plasma pressure in the deposition chamber. Results show that clamping influences did not distort the data and that layers grown at lower pressures were in compression while layers at higher pressures were in tension. Similarly to other sputter deposited materials, there is a quick transition between compressive and tensile stress as plasma pressure is increased. It is found that during deposition the sputtered Si layer was more stressed than the WSi_2 layer.

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A-71

Grazing-incidence Small-Angle X-ray Scattering Study of Readily Controllable Palladium Nanoparticle Formation on Surface-assembled Viral Nanotemplates

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Transition metal nanoparticles possess unique size-dependent properties that differ significantly from their bulk properties. However, nanoparticle synthesis is often difficult to predict and control. Biological supramolecules offer attractive templates for controllable nanoparticle synthesis due to their precise structure and size. Viruses in particular have gained significant attention as nanoparticle synthesis templates in nanodevice fabrication for applications such as nanoelectronics, batteries, and catalysis. In this work, we exploit genetically modified Tobacco Mosaic Virus (TMV1cys) for readily controllable palladium (Pd) nanoparticle synthesis in mild aqueous conditions by simply tuning reducer concentration. Grazing incidence small-angle x-ray scattering (GISAXS) was employed to provide an accurate and statistically meaningful route to investigate the broad size ranges and uniformity of the Pd nanoparticles formed on TMV templates. Further, we studied the performance range of these structures by examining their thermal stability via *in situ* GISAXS. Specifically, we show that the stability of the Pd nanoparticles on TMV is significantly enhanced as compared to particles on the solid substrate surface. We envision that the results and methodology demonstrated in this study could be applied to better understand the properties and dynamic behaviors of organic-inorganic hybrid materials and nanodevices in various applications.

A-72

Viral-templated Palladium Nanocatalysts for Dichromate Reduction

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We demonstrate a viral template-based bottom-up assembly approach for palladium (Pd) nanocatalyst synthesis for dichromate reduction. Specifically, genetically displayed cysteine residues on each coat protein of Tobacco Mosaic Virus (TMV) templates provide precisely spaced thiol functionalities for tunable surface assembly and Pd nanoparticle formation. Reaction kinetics studies by *in situ* UV-VIS monitoring reveal catalytic activity of Pd nanoparticle preferentially formed on TMV. In-depth characterization via atomic force microscopy, grazing-incidence small-angle x-ray scattering, and x-ray photoelectron spectroscopy show preferential Pd nanoparticle formation on TMV, stability, and nanoparticle size that correlate well with the reaction kinetics results. We further present two facile routes to control Pd nanocatalyst surface loading based on tunable and selective surface assembly of TMV. In the first method, the surface density of Pd-TMV complexes and dichromate conversion rate are controlled by varying the concentration of TMV solution for surface assembly. In the second method, the Pd catalyst loading and reaction rate are controlled by varying the gold surface area on patterned silicon chips via standard photolithography. We envision that our approach for readily controllable surface assembly of catalytically active Pd nanoparticles under mild aqueous conditions would provide a facile route to catalyst synthesis in a wide range of applications.

A-73

Fabrication Effects on the Morphology and Electronics in Organic Photovoltaic Solar Cells

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Developing novel organic photovoltaic (OPV) devices is important as demands for energy increase. The main obstacle in conventional OPVs is the low efficiency at ~6%, far below what requires for commercial applications. We have studied the morphology of a group of polymers that contain alternating units of thieno[3,4-b]thiophene and benzodithiophene units (denoted PTB), which was done in collaboration with Luping Yu at the University of Chicago. We have shown through grazing incidence x-ray scattering techniques that these polymers do in fact have an optimal packing structure for solar cell devices compared to more conventional polymers used in OPV devices. The highest solar cell efficiency observed in these materials is 8%, which is the current record efficiency to our knowledge. We have also focused on the role that aliphatic side chains play in the optoelectronic and morphological properties in these polymer systems. In this work, the spectroscopic, x-ray, and kinetic characteristics of seven PTB polymers are discussed in detail. Although these polymers differ by their side chains, the PTB backbone is the same in all polymers. Slight structural changes elucidate the electronic and morphological characteristics that make these polymers ideal for OPV devices.

A-74

Development of a Superconducting Undulator for the APS

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See poster for abstract.

A-75

Spin States and Structural Phases in Novel Superconducting Iron Pnictides

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Over the past few decades the understanding of the electronic structure of solids has become important now that it can be used as the basis for direct prediction of the entire range of dielectric and bonding properties of solids. Spin-crossover or valence transformation upon crystallographic phase transitions in many materials, especially in strong correlated electron systems takes place [1, 2]. Since the discovery of the cuprates some decades ago, the metal pnictides are considered as the second important class of high-T_c superconductors [3–5]. The pnictides having strong electron-electron correlations and show an interesting relationship between unconventional superconductivity and magnetism. Recently, we studied the local electronic structure of 3d transition of Fe ions in $SrFe_2As_2$ by x-ray emission spectroscopy [6]. The HS-LS spin crossover effect (high-spin low-spin transition) induced by high pressure at room temperature in the range 4–6 GPa is observed in $SrFe_2As_2$. We expect the most interesting processes at low temperatures. Changes in electronic and magnetic states are followed by or cause the change of interatomic distances and angle bonds.

Therefore, I will present our study of the structural and electronic phase transitions in $SrFe_2As_2$ at high pressures and low temperatures. To reveal the evolution of superconductivity and magnetism, the interplay between these two collective phenomena, electronic structures, and spin states in these materials we performed high-pressure, low-temperature studies of the $SrFe_2As_2$ using x-ray emission spectroscopy and x-ray diffraction. As a result of the study, we obtained the detailed crystal structure of $SrFe_2As_2$, the structural V-P phase diagram, and established the relationship between structural, electronic, and magnetic properties of the iron-arsenide superconductors.

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A-76

Diagnostic Potential of Phase-sensitive Radiography

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Phase-sensitive imaging is based on the use of information arising from the modification in amplitude and phase of x-rays as they traverse an object. This allows the detection of properties, such as x-ray refraction, which is not possible with conventional radiography. Thus, detection of tissues and tissue properties that do not garner contrast through x-ray attenuation, especially at high energies where radiation dosage is reduced, is made possible through phase-sensitive imaging. Tissues that benefit from this imaging parameter are the soft tissues that do not have the elemental make-up for x-ray attenuation. The phase-sensitive technique that is used by our laboratory is an analyzer-based system called diffraction enhanced imaging (DEI), whereby an analyzer is positioned between the object and the detector to allow changes in x-ray angle to be converted to changes in x-ray intensity at the detector. By altering the angle of the analyzer both refraction and absorption can be recorded. Our laboratory has applied DEI to the detection of cartilage lesions of early osteoarthritis that are invisible with conventional radiography. We have shown, with x-rays from both the synchrotron and a compact source that DEI allows the visualization of soft tissues and their associated lesions.

A-77

X-ray Microdiffraction Studies of Phase Separation in Complex Oxides

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Structural inhomogenieties in complex oxides range from nanoscale composition or strain fluctuations to macroscopic phase-separated domains and can be strongly coupled with the electronic and magnetic properties. We are investigating phase separation and domain interactions in several complex oxide systems using polychromatic and monochromatic x-ray microdiffraction at APS sector 34-ID-E. Model systems with distinct, relatively large micron-scale phases have been studied as a first step towards understanding nanoscale systems. Directionally-solidified manganite crystals [e.g., EuYMnO and (LaSr)LuMnO] were found to consist of mesoscale lamellar domains with alternating pseudocubic perovskite and multiferroic hexagonal crystal structures. X-ray microdiffraction was used to study the domain orientations, morphologies and interfaces, and to map the local strain fields within the domains. In vanadium dioxide microcrystals, temperature-dependent measurements reveal coexisting monoclinic and tetragonal phases and strain variations near the metal-insulator transition. In all of these studies, the goal is to combine spatially resolved x-ray microdiffraction with other local probes to understand how domain interactions give rise to complex electronic and magnetic properties in strongly correlated oxides.

Research at the APS supported by the U.S. DOE, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering.

A-78

Awareness, Preference, Utilization, and Positioning Research for the Spallation Neutron Source and High Flux Isotope Reactor

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The Neutron Sciences Directorate at Oak Ridge National Laboratory (ORNL) conducted survey research during 2009–2010 to evaluate awareness of, preference for, and utilization of the Spallation Neutron Source (at ORNL), the High Flux Isotope Reactor (at ORNL), the Advanced Photon Source (APS at Argonne National Laboratory), and the National Synchrotron Light Source (NSLS at Brookhaven National Laboratory). We were interested in gaining an understanding of the (1) level of understanding regarding benefits that neutron scattering might afford members of these scientific communities, (2) awareness levels among those in the scientific communities regarding what ORNL has to offer relative to the two neutron sources, and (3) whether there were any perceptions negatively impacting utilization of each facility. The research employed a mix of qualitative methods and an on-line survey. Meetings were held with key stakeholders from ORNL as well as members of the ORNL communications team and leadership from APS and NSLS. One-on-one interviews, traditional focus groups (face to face) and on-line focus groups with users were held to gain insights into the user experience from awareness of a facility and submitting a proposal through conducting the experiment at a national laboratory or other world-class facility. An on-line survey was then used to develop quantitative information. The survey consisted of 16 questions and seven demographic categorizations, nine open-ended queries, and 153 pre-coded variables; the survey took an average time of 18 minutes to complete. The survey will provide a way to quantify difference—if any—between scientists based on key demographics, age, area of science, length of time in field, years experience with light/neutron sources, etc.

The results of the survey indicated that the awareness of SNS or HFIR is relatively low outside the ORNL user group. Users of APS and NSLS tended to first become aware of the facilities through an academic advisor whereas users of SNS and HFIR first become aware through a colleague. When users were asked about their expectations of a facility, the following five ranked the highest for all facilities: (1) sample environment, (2) reliability of the beam, (3) intuitive software, (4) easy access, and (5) 24/7 notification of beamline status. Additional results will be available on the poster including users preferences for educational opportunities, methods of communication with the facility's user office and scientists, obtaining information from written and electronic sources, and beliefs about proposal submissions.

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E-01

Environmental Impacts of Acquiring Fossil Fuel Photocatalytic Property

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The increased burning of fossil fuels every year has a negative environmental impact on the atmosphere. The problem could be mitigated through photocatalysis of the hazardous materials evolved from fuel burning. To achieve this goal, the fuels are acquired photocatalytic property where the exhaust gases can be converted photocatalytically to less hazardous materials in the atmosphere.

Fossil fuels contribute many gas pollutants to the atmosphere resulting in global warming, acid rain, ozone, and sooty carbonaceous materials. These pollutions could be resolved through photocatalysis in the atmosphere. The acquired fuel for the photocatalytic reactivity can be inexpensively activated using either the ultraviolet (UV) or the UV-visible spectrum. The synchrotron-based analytical techniques offer unequaled capacity to follow different possibilities of the proposed scenarios of the hazardous gas flue. The SO_x, NO_x, and CO_2 gases could be directed to keep the atmosphere in a more "green" condition.

E-02

The Development of Anodic Aluminum Oxide-based Micro-channel Plate for Large-area Photo Detector

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See poster for abstract.

E-03

Direct Observation of Tribological Behaviors of Materials

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Although the relative motion of two bodies in contact involves a number of forces across the interface influencing the nature of sliding and wear, frictional forces play a central role in everyday experience as it was in ancient times. The understanding and evaluation of friction, especially atomic-scale friction, remains a challenge. It is suggested that defects motion mainly contributes frictional force at defective surface. On the other hand, the destruction of crystalline structure dominates the frictional process at the surface of a single crystal. Last but not least, we also present direct evidence of tribological recrystallization [1] and grain growth in a polycrystalline gold thin film induced.

The friction here is studied by observing the structure change driven by sliding of the probe in combination with an applied load of a nanoprobe. In this study, a Nanofactory AFM-TEM holder is used in a transmission electron microscope (TEM) system.

1. Y. Liao, S. K. EswaraMoorthy, and L. D. Marks, *Philosophical Magazine Letters*, 1–5, (2010), (DOI: 10.1080/09500830903571384).

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E-04

Barrier Evolution of Magnetic Tunnel Junction by Annealing and under Biased Condition

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Energy-filtered transmission electron microscopy (EFTEM) and *in situ* electron holography were applied to study changes to the tunnel barrier behavior of CoFe/MgO_x/CoFe magnetic tunnel junctions as a function of annealing and applied electrical bias. During annealing oxygen moved to the MgO_x to form a more stoichiometric and homogenous crystalline tunnel barrier, and Co diffused into the barrier. There is no significant change in Fe distribution. Annealing also results in a reduction of the barrier height. The effect of varying the bias voltage from -1.5 V–1.5 V is to change barrier asymmetry and to decrease the effective barrier width. These changes are a result of charge accumulation at the interface.

Argonne National Laboratory is operated under Contract No. DE-AC02-06CH11357 by U.S. DOE. The electron microscopy was accomplished in the Argonne National Laboratory Electron Microscopy Center for Materials Research.

E-05

Structure-property Relationship of Self-assembled Multiferroic $CoFe_2O_4$-$PbTiO_3$ Nanocomposites

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Self-assembled multiferroic $CoFe_2O_4$ (CFO)-$PbTiO_3$ (PTO) nanocomposites have been fabricated on a $SrTiO_3$ (001) substrate by metalorganic chemical vapor deposition. Ferrimagnetic CFO filaments were observed to form in a ferroelectric PTO matrix due to the competition of surface and interfacial energies as verified by energy-dispersive x-ray spectroscopy using the FEI Tecnai F20ST at the Electron Microscopy Center. Traditional cross-sectional transmission electron microscopy shows the filaments are of different shapes, suggesting they may be branched. Scanning transmission electron microscopy (STEM) tomography was performed to better resolve the “Y” shaped CFO filament in one sample. The lower atomic weight of CFO has a lower STEM high-angle angular dark field contrast than the PTO matrix, allowing for a tomographic STEM reconstruction. The shape anisotropy from the high aspect ratio of CFO filaments (20 nm in diameter vs. 300 nm in length) realigns the direction of the easy axis of magnetization in the filaments to the out-of-plane [001] direction as opposed to the in-plane [100] direction favored in blanket CFO thin films. However, the close packed nature of the CFO filaments may hinder the volume change of the PTO matrix resulting from an applied electric field, which makes it difficult to probe the ferroelectric properties of the heterostructures at the resolution of piezoresponse force microscopy.

E-06

Combined Transmission Electron Microscopy and Atom-probe Tomography of Magnetic Tunnel Junctions

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Magnetic tunnel junctions (MTJs) are of tremendous technological interest for the next generation of hard disk drive readers and as memory elements in magnetic random access memories. The microstructural features and multilayer interfaces of this thin film heterostructure determine the magnetic and electronic properties and ultimately the performance of the MTJ. In this work, CoFeB/MgO MTJs have been investigated by a combination of transmission electron microscopy (TEM) and atom-probe tomography (APT). TEM is used to analyze the crystallography and grain structure of the multilayers, while APT is used to map the three-dimensional elemental distribution. As deposited, the CoFeB is amorphous and the MgO has a poor crystalline texture. Annealing results in partial crystallization of the CoFeB and improved MgO crystallinity, enhancing the properties of the MTJ. Here TEM is performed on the individual APT specimens allowing for a direct correlation of the crystal structure with the elemental distribution. TEM is further used to follow the tip-shape evolution of the APT specimen, yielding insights into the resulting three-dimensional reconstruction. These studies have revealed unique local compositional differences between the crystalline and amorphous grains of CoFeB that have not seen previously by any other technique, offering new insights into the resulting MTJ performance.

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INFORMATION

Advanced Photon Source

Web site: http://aps.anl.gov
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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 2010, the APS staff numbered around 260. In fiscal year 2009, more than 3,500 users visited the APS an aggregate total of more than 11,500 times to carry out more than 3,600 experiments and published more than 1,000 papers (as of April 5, 2010).

Electron Microscopy Center

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

Facility Summary and Mission

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 within the Materials Science Division at Argonne National Laboratory 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 instrument 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.

Instrumentation Overview

The EMC currently operates and administers nine instruments, together with support facilities for specimen preparation and image analysis. The instrumentation in the Center is organized into three major areas according to specialized functions:

- Sub-Ångstrom Microscopy and Microanalysis Laboratory, which houses the Argonne Chromatic Aberration-corrected TEM
- Analytical Electron Microscopy facility (standard STEM, TEM, FIB, SEM, ESEM)
- IVEM-Tandem facility (300 kV TEM interfaced to two ion accelerators).

User Access

User access to the EMC for research is through a proposal submission process. 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 meet the occasional need for enhanced access.

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.

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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 standalone 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 material 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.
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Blake Industries, Inc. 660 Jerusalem Road Scotch Plains, NJ 07076 USA Phone number: 1.908.233.7240 Fax number: 1.908.233.1354 E-mail: blake4xray@att.net	Blake Industries is a leader in supplying high-precision x-ray and neutron instrumentation to research labs, university, and synchrotron beam lines 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.

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.
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Instrument Design Technology Ltd. Unit 2, Turnstone Business Park Mulberry Avenue, Widnes Waterfront Widnes, WA8 0WN, UK Phone number: 011.44.151.601.4111 Fax number: 011.44.151.601.4110 URL: www.idtnet.co.uk	Instrument Design Technology Ltd. designs and builds complete hard x-ray beamlines and all optical monochromators including cryo-cooled, water-cooled, and scanning as well as large and small mirror systems plus high heat load slits and filters, beam diagnostics, and optical tables." We have have delivered 5 complete beamlines to facilities worldwide and can provide full support from concept to commissioning with beam.
InSync, Inc. 2511C Broadbent Pkwy. Albuquerque, NM 87107 USA Phone number: 1.505.345.2660 x615 Fax number: 1.505.345.2589 URL: www.InSyncOptics.com	InSync, Inc. is a custom, high-tech optical manufacturing company. Specialty is the unusual "state of the art" polished optic up to 1500 mm in size. Capabilities include: Computer Design, Finite Element Analysis, Slicing, Grinding, Feature Machining, Etching, Bonding, Lapping, Polishing, Coating, Metrology. Polished Shapes; Cylindrical, Spherical, Flat. Substrate Designs; Internally Cooled, Externally Cooled, Uncooled, Rectangular, Round, Bendable for other polished shapes. Substrate Materials; Silicon, Glass, Metals.

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Primatics & Tek-Matic **Primatics32114 Mallard Ave.** Tangent, OR 97389 USA Phone number: 1.888.754.3111 Fax number: 1.541.791.9410 URL: www.primatics.com **Tek-Mati** 7324 Forest Hills Road Loves Park, IL 61111 USA Phone number: 1.815.282.1775 Fax number: 1.815.282.1862 URL: www.tekmatic.com	Primatics designs and manufactures custom and standard precision and rotary positioning stages. Primatics can provide customized versions of their standard offering, as well as fully engineered solutions. Micron, sub-micron, and arc second positioning solutions are available. Tek-Matic (a partner with Primatics), provides electromechanical automation and precision motion control systems. Tek-Matic can provide product solutions, as well as fully integrated motion control systems. Our engineering sales staff can assist with product sizing, selection, and support.

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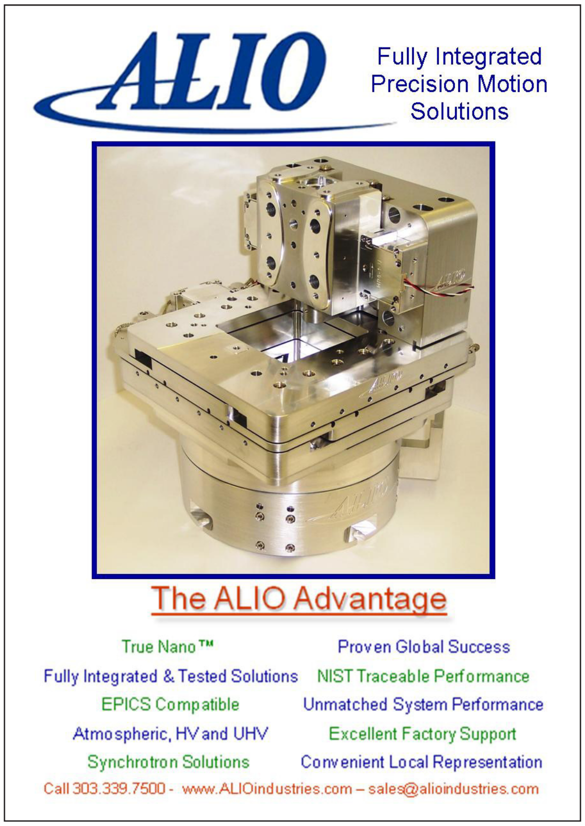

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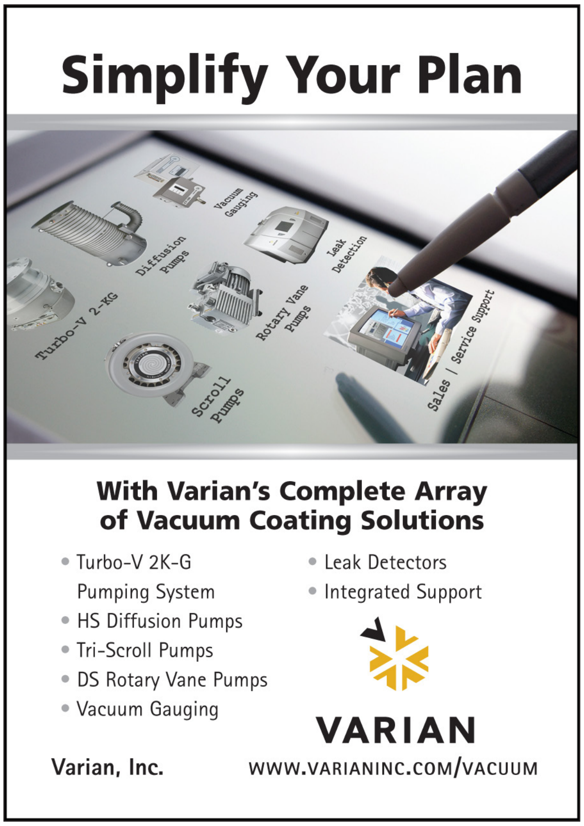

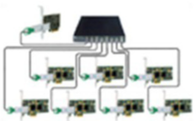


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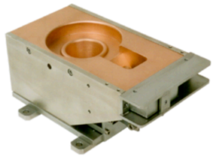

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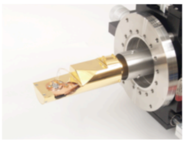

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GENERAL
INFORMATION

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 401, Room B1200 (first floor, front hallway). 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 a continental breakfast and coffee breaks each day and the poster session reception. Buffet lunches will be served Monday through Wednesday in the tent on the patio on the lower level of the conference center. 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 4, at at the Argonne Guest House. Banquet tickets are nonrefundable. 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. The Beanline coffee shop in the Building 401 lobby will be open for its usual hours of 2:30 to 4:30 pm. The 401 Grill will be closed throughout the meeting. 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

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 make a telephone call, a pay phone is located downstairs near the restrooms at the back of the gallery. If you need to send or receive a fax, a fax machine is located in the APS User Office (Bldg. 401, Rm. B1154). The number of this machine is 630.252.9250.

Transportation

Conference staff can make limousine reservations for you during the meeting as long as you make your request before 1:00 pm on Wednesday. Alternatively, A-1 Limousine has an online reservation form (http://www.a1limousine.com/).

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:

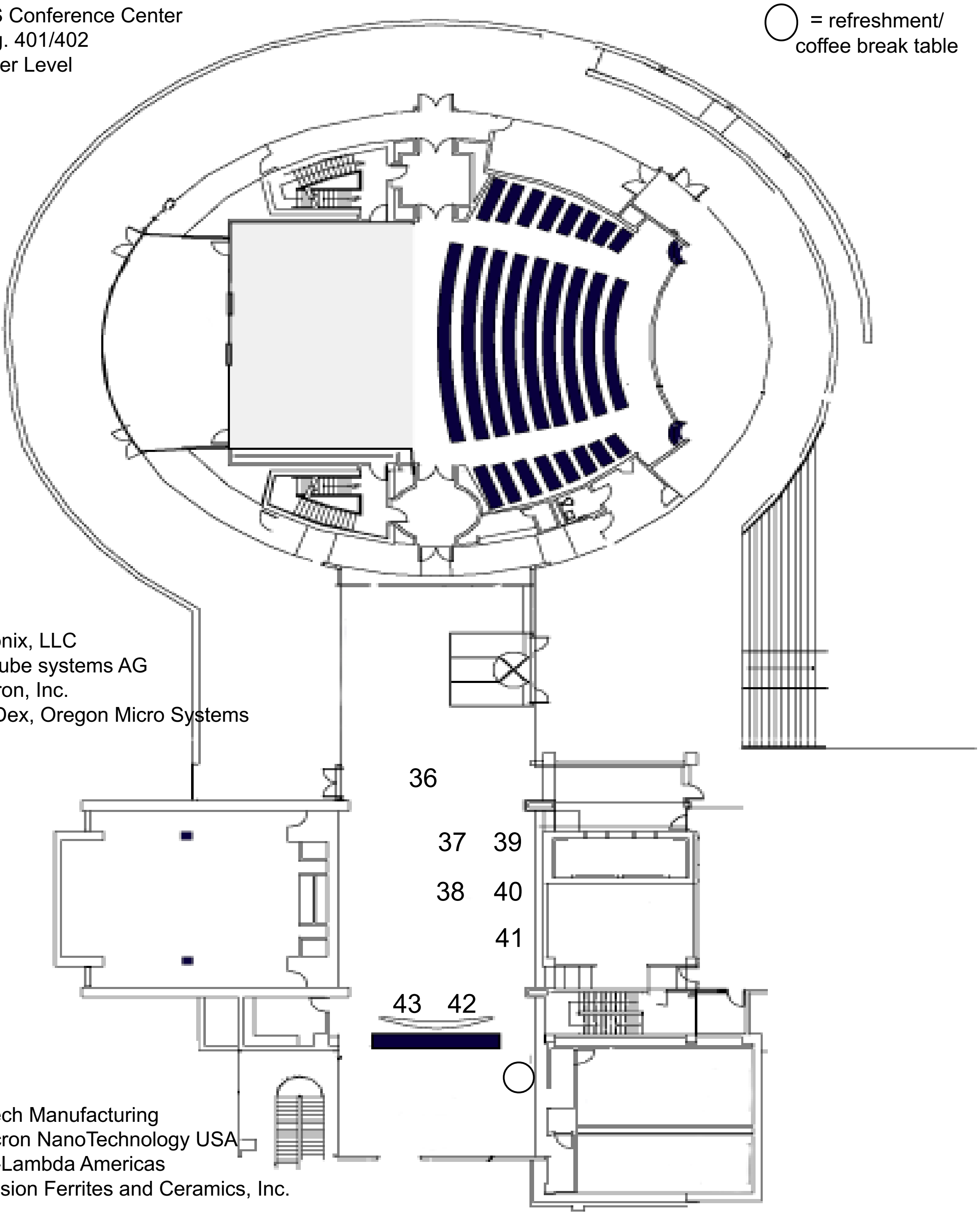
1. Open the wireless connection on your computer (either the 401 or 402 networks).
2. Read and accept the Argonne internet access policy, which will appear as a Web page on your desktop. After you click "accept," a registration Web page will appear.
3. The registration web page asks you for the following 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 – 2010 APS/EMC Users Meeting
Argonne National Laboratory

APS Conference Center
Bldg. 401/402
Upper Level

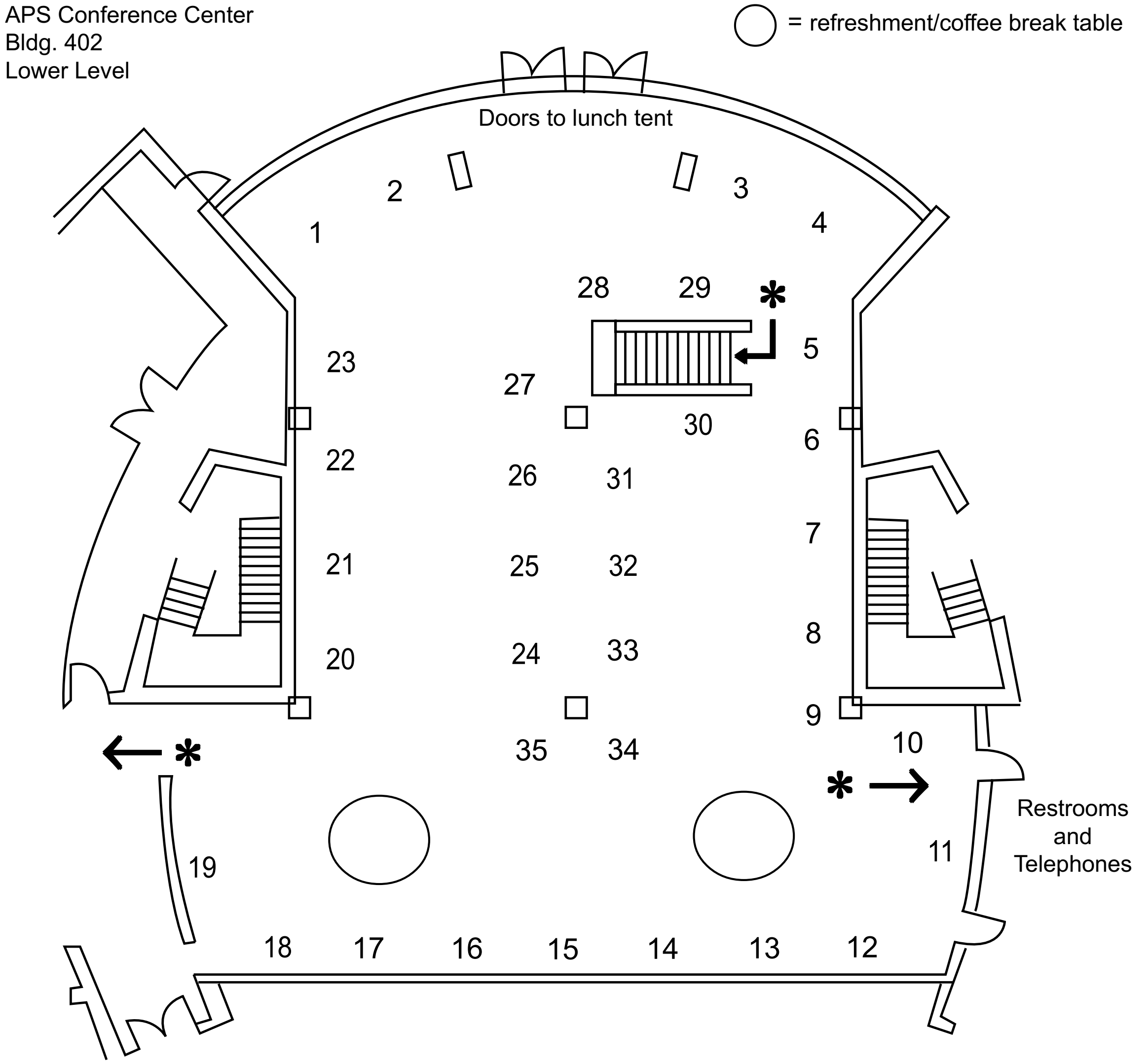
○ = refreshment/
coffee break table



36. Rayonix, LLC
37. attocube systems AG
38. Phytron, Inc.
39. Pro-Dex, Oregon Micro Systems

40. Hi-Tech Manufacturing
41. Omicron NanoTechnology USA
42. TDK-Lambda Americas
43. Precision Ferrites and Ceramics, Inc.

Vendor Locations – 2010 APS/EMC Users Meeting
Argonne National Laboratory



1. CMA/Flodyne/Hydradyne
2. Oerlikon Leybold Vacuum USA
3. MICOS USA LLC
4. Instrumentation Technologies
5. Blake Industries, Inc.
6. Aerotech, Inc.
7. XIA LLC
8. Thermo Fisher Scientific
9. Steinmeyer, Inc.
10. InSync, Inc.
11. James R. Johnson & Assoc., Inc.
12. MDC Vacuum Products, LLC
13. Vacuum One
14. Rigaku Innovative Technologies
15. Primatics & Tek-Matic
16. Pfeiffer Vacuum
17. Bruker AXS Inc.
18. Kurt J. Lesker Company
19. ALIO Industries
20. Midwest Vacuum, Inc.
21. Carl Zeiss MicroImaging, Inc.
22. PI (Physik Instrumente) LP
23. Varian, Inc.
24. Instrument Design Technology Ltd.
25. Thermionics Vacuum Products
26. Advanced Control Systems Corp.
27. Tungsten Heavy Powder, Inc.
28. Advanced Design Consulting USA, Inc.
29. Technimark, Inc.
30. Newport Corporation
31. Left Coast Instruments
32. Advanced Research Systems, Inc.
33. FMB Oxford
34. Alan Burrill Technical Sales
35. Princeton Instruments

* UPSTAIRS/ATRIUM EXHIBITS PLUS REFRESHMENT TABLES

SCHEDULE AT A GLANCE
2010 APS/EMC Users Meeting

	Monday, May 3	Tuesday, May 4	Wednesday, May 5
Morning	**Registration** 7:00 – 5:00 401 Atrium	**Registration** 8:00 – 5:00 401 Atrium	**Registration** 8:00 – 1:00 401 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
	Continental Breakfast 8:30 – 9:30	Continental Breakfast 8:30 – 9:30	Continental Breakfast 8:15 – 9:30
	Opening Session 8:50 – 12:00 402 Lecture Hall • 8:50 Welcome – Fuoss/Peters • 9:05 DOE Perspective – Kung • 9:35 Congressional Report – Platz • 10:00 APS Update – Gibson • 10:30 *Break* • 11:00 EMC Update – Miller • 11:15 CNM Update – Petford-Long • 11:30 Shenoy	**APS Science Session I** 8:50 – 12:00 402 Lecture Hall • 9:00 Miceli • 9:30 Lee • 10:00 Reis • 10:30 *Break* • 11:00 Harder • 11:30 Jaramillo	**WK1 – Industrial Research** 8:50 – 12:00 (& PM) 402 Lecture Hall
			WK2 – Beamline 2.0 8:30 – 12:00 (& PM) 402 Room E1100/E1200
			WK3 – Structure/Magnetism in Novel Materials 8:45 – 12:15 (& PM) Bldg. 440, Rm. 105/106
			WK4 – XFELO 8:30 – 12:30 401 Room A1100
			WK5 – Biotemplated Materials 8:40 – 12:25 401 Room A5000
Noon	Lunch 12:00 – 1:30 Tent **Postdoc Lunch Talk** 12:15 – 1:00 401 Room A1100	Lunch 12:00 – 1:30 Tent	Lunch 12:00 – 1:30 Tent **APSUO St. Com.** 12:00 – 1:00 401 Fifth Floor
Afternoon	**Plenary Session** 1:25 – 5:00 402 Lecture Hall • 1:30 Student talk • 1:50 Hendrickson • 2:30 *Break* • 3:00 Blasie • 3:40 Parise • 4:20 Mills	**APS Sci. Session II** 1:25 – 4:30 402 Lecture Hall • 1:30 Seidler • 2:00 Guthrie • 2:30 Clarke • 3:00 *Break* • 3:30 Boyanov • 4:00 Fischetti **EMC Sci. Session** 1:25 – 4:45 402 Room E1100/E1200 • 1:30 Ciston • 2:00 Ferreira • 2:30 Sharma • 3:00 *Break* • 3:15 Ross • 3:45 Browning • 4:15 Minor	**WK1 – Industrial Research** 1:30 – 4:30 402 Lecture Hall
			WK2 – Beamline 2.0 1:30 – 5:30 402 Room E1100/E1200
			WK3 – Structure/Magnetism in Novel Materials 1:30 – 5:30 Bldg. 440, Rm. 105/106
			WK6 – Actinide Research 1:00 – 5:20 401 Room A1100
		APSUO "Town Hall Meeting" 4:30 – 5:00 402 Lecture Hall **EMC Users Exec. Comm. & Election** 4:45 – 5:15 402 Room E1100/E1200	**WK7 – Imaging/Microscopy for Biology & Medicine** 1:30 – 4:45 401 Room A5000
Evening	**Poster Session and Reception** 5:00 – 7:00 401 Room 1200 (library)	**Banquet** 6:00 – 9:00 Argonne Guest House	
	Partner User Council 7:30 – 9:00 Argonne Guest House		

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