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Novel Approaches to Soft X-ray Spectroscopy: Scanning Transmission X-ray Microscopy and Ambient Pressure X-Ray Photoelectron Spectroscopy

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XANES and EXAFS Spectroscopy of Materials and Biological Samples: Expanding the Range of Applications at Beamline 9.3.1

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ALS Users' Meeting and Workshops

ALS Users' Meeting: Another Record Attendance

Thanks to a surge in eleventh-hour registrations, an overflow crowd of more than 400 users, staff, and vendors materialized to enjoy a successful ALS Users' Meeting, with a program chock full of science highlights and 13 workshops, all spread over three days from October 21–22, 2005.

Berkeley Lab Director Stephen Chu started off the proceedings with encouraging words about the planned on-site user lodging facility and promised his support for keeping the ALS a top user facility in all categories, from accessibility to scientific output. Acting ALS Director Janos Kirz began his report on the state of the ALS by pointedly reiterating

the ALS mission: to support users in doing outstanding science in a safe environment. He then reviewed progress on the accelerator, beamline, and science fronts, highlighting an outstanding report card from the 2005 BES review of the ALS. Operation of the ALS is funded by the U.S. Department of Energy (DOE), Office of Basic Energy Science (BES).

Plans are one thing; the funds to carry them out are another. Michael Lubell (City College of New York and American Physical Society) reviewed the outlook for the federal science budget, including both positive and negative factors. The bottom line to users: get politically active in order to protect funding



Figure 1: Pat Dehmer (USDOE) presented Daniel Chemla (ALS) with a DOE Distinguished Associates Award.

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Figure 2: During the popular vendor reception and poster session, displays spilled over from the ALS patio into adjacent Building 7.

for light sources. In her annual “View from Washington” report, DOE Associate Director of Science for BES Pat Dehmer summarized her efforts to convince federal budget makers that, while the light sources continue to operate 95 percent or more of the schedule time, that is only part of the story. If light sources are to remain competitive, they cannot continue to operate on flat funding.

At the end of her talk, Dehmer presented retiring ALS Director Daniel Chemla with a DOE Distinguished Associates Award, and the audience saluted him with a standing ovation. The award recognized his “many accomplishments, contributions, and leadership in both science and science management,” according to the citation signed by DOE Secretary Samuel Bodman. It went on to say: “You have brought the Advanced Light Source to a prominent role as a national and international scientific user facility. Your pioneering research in nanoscience and dedication to the establishment of the Department

of Energy Nanoscience Research Centers has made it a reality.”

In other Thursday morning presentations, ALS accelerator physicist Christoph Steier reviewed progress and plans toward the new top-off mode of operation expected to be fully operational in 2007, as well as other accelerator improvements. Users Services leader Gary Krebs described upcoming changes in the badging process, and Science Director Neville Smith unveiled a draft version of a new general sciences proposal process and invited user input. Users’ Executive Committee (UEC) chair Greg Denbeaux (The University at Albany) conducted a town hall meeting that covered several issues, including candidates for the upcoming UEC election.

Thursday afternoon and Friday morning were devoted to keynote presentations and science highlights, while Friday afternoon and Saturday were dedicated to a set of 13 workshops. The full meeting agenda is posted on the meeting web site (www-als.lbl.gov/als/usermtg/index.html), where pdf files of many presentations are also available. In addition, reports of several of the workshops immediately follow this article. Workshops and organizers were:

- An Introduction to Synchrotron Experimental Techniques (Michelle Weinberger, University of California, Los Angeles);
- Forefront AMO Science: Clusters, Ions, Dressed States (John Bozek, ALS, and Nora Berrah, University of Western Michigan);
- Frontiers of Synchrotron-Based X-Ray Microdiffraction (Nobumichi Tamura and B.W. Batterman, ALS);
- New Visions in Bandmapping (Eli Rotenberg and Alexei Fedorov, ALS, and Ken Goldberg, Berkeley Lab);
- Macromolecular Crystallography I: Advanced Experimental Techniques for Getting the Best Data from Difficult Samples (Christine Trame, Berkeley Lab);
- Macromolecular Crystallography II: New Strategies for Data Processing with Automated Software Tools (James Holton, Berkeley Lab);
- Novel Approaches to Soft X-Ray Spectroscopy: Scanning Transmission X-Ray Microscopy and Ambient-Pressure X-Ray Photoelectron Spectroscopy (Hendrik Bluhm and Mary Gilles (Berkeley Lab) and Simon Mun, and Tolek Tyliczszak, ALS);



Figure 3: Meeting attendees enjoyed barbecue and corn-on-the-cob alfresco.

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- Soft and Hard X-Ray Tomography at the ALS (Alastair McDowell, ALS) and Gerry McDermott (Berkeley Lab);
- Soft X-Ray Photon-In and Photon-Out Spectroscopy: New Frontiers (Jinghua

Guo, ALS, and Zahid Hasan, Princeton University);

- THz Science and Technology Network: Opportunities and Organization (Michael C. Martin, ALS);
- Ultrafast X-Ray Science at the ALS (Bob Schoenlein and Peter Fischer, Berkeley Lab);
- XANES and EXAFS Spectroscopy of Materials and Biological Samples: Expanding the Range of Applications at Beamline 9.3.1 (Robert Szilagyi, Montana State University, and Heinz Frei, Berkeley Lab).

Thursday evening saw the inauguration of “public science” lectures as part of the meeting program. Co-sponsored by the ALS and the Berkeley Lab Friends of Science, Joachim Stöhr, newly appointed director of the Stanford Synchrotron Radiation Laboratory, spoke to almost 200 meeting attendees and members of the public on “The Mysteries of Magnetism: From Physical Attraction to Spin Doctors.” A Real Audio (.ram) file of the same presentation given earlier at the Stanford Linear Accelerator Center is downloadable from the meeting Web site and from www.lightsources.org/cms/?pid=1000904.

A pleasant California autumn evening provided the perfect environment for the now-traditional barbecue dinner on the ALS patio on Friday. Program co-chairs Simon Morton (Berkeley Lab’s Physical Bio-

sciences Division) and Jinghua Guo (ALS) then hosted the user award session. This year there were two student poster prizes: Michelle Weinberger (University of California, Los Angeles) won the first prize, sponsored by Oxford Instruments, and Benjamin Yuhas (University of California, Berkeley) took the second prize, sponsored by Vacuum Generators. Ron Slater and Ed Wong (Berkeley Lab’s Engineering Division) each bagged individual Tim Renner User Services Awards.

The Klaus Halbach Award for innovative instrumentation was won by Elke Arenholz (ALS) and Soren Prestemon (Engineering Division) for the design and implementation of a vector magnetometer for soft X-ray studies. The David A. Shirley Award for scientific achievement went to Craig Taatjes (Sandia National Laboratories), Terrill Cool (Cornell University), Philip Westmoreland (University of Massachusetts), and their colleagues on “the flame team” for the surprising and far-reaching discovery of enols in flames. And in a special presentation, Wei-lun Chao (University of California, Berkeley, and Berkeley Lab’s Center for X-ray Optics) took possession of the Werner Meyer-Ilse prize for outstanding work by a young researcher, awarded every three years at the International X-Ray Microscopy Conference. ■

ART ROBINSON
*Advanced Light Source
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ALS Users’ Meeting and Workshops

Forefront AMO Science: Clusters, Ions, Dressed States

In an unprecedented marriage of two communities, laser and synchrotron scientists met for one-and-a-half days following the 2005 ALS Users’ Meeting to discuss present accomplishments and future new directions. A

scintillating program of speakers covered topics including ultrafast AMO science, photoionization of clusters and nanoparticles, and photoionization studies of ions, atoms and molecules. The utility of the ALS as a source

of VUV and soft X-ray radiation for AMO physics experiments was widely acknowledged, with the caveat that beamtime on the oversubscribed undulator beamlines is scarce and acts as the limiting resource for the more



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Were the coffee and doughnuts that good? Smiling workshop members pose on the stairway in the ALS lobby.

exploratory research. A new beamline at the ALS to advance forefront AMO VUV/soft X-ray experiments would help alleviate this problem, and requirements for such a beamline were also discussed.

Ultrafast AMO research was discussed in the first session, which was organized jointly with the parallel "Ultrafast X-ray science at the ALS" workshop held during the same period (see report in this issue). Phil Heimann (Berkeley Lab) described the design of a new undulator beamline to produce fsec pulses of X-ray radiation by the slicing technique [see R.W. Schoenlein et al., *Synchrotron Radiation News* **14** (1), 20 (2001)]. The beamline utilizes a 3-cm-period in-vacuum undulator to produce radiation for two branchlines, a soft X-ray branch covering 120–2000 eV and a hard X-ray branch for 2–10 keV. A custom-built high-power and high-repetition rate femtosecond laser system produces two beams of about 1 mJ per pulse to generate the slicing pulses for the electron beam in the synchrotron as well as provide pump/probe pulses for the sample.

Marcus Hertlein (Berkeley Lab) described ultrafast electron correlation measurements in atomic potassium using synchronized laser

and X-ray pulses. The temporal resolution of the experiments is currently limited by the length of the ALS pulses (~60 ps) but is ready to take advantage of the ultrafast X-ray pulses from the ALS ultrafast beamline and the Linac Coherent Light Source (LCLS) at SLAC once they become available. Linda Young (Argonne National Laboratory) rounded out the joint session with a description of and results from a recently expanded facility for high-field laser/X-ray studies of atoms and molecules at the Advanced Photon Source.

Philip Bucksbaum (Stanford University) described sub-femtosecond research opportunities, both how to produce sub-femtosecond pulses and what to do with them. Electron dynamics rather than nuclear dynamics become the target in this time domain, and while it will not be accessible any time soon with accelerator-based sources, complementary studies can be carried out. Rounding out the ultrafast talks, Jerry Hastings (Stanford Synchrotron Radiation Laboratory) brought everyone up to date on the status of the LCLS project at Stanford. The LCLS will produce pulses of X-rays around 100 fsec long with a 120-Hz repetition rate and is scheduled to be ready for operations in 2009.

Clusters and nanoparticles studied with VUV and soft X-rays were discussed in several presentations. Steve Leone (University of California, Berkeley, Berkeley Lab) discussed photoionization dynamics of nanoparticles, including results from Mie scattering and photoelectron imaging experiments. The use of nanoparticles as sources of large organic molecules for photoionization and mass spectrometry using VUV ionization was also briefly discussed. Todd Ditmire (University of Texas, Austin) turned up the heat on clusters, exploding them with very intense IR laser radiation to explore their behavior in the limits of hydrodynamic expansion, where most of the electrons are retained by the cluster, and Coulomb explosion, where most of the electrons are removed. Anticipating the availability of intense X-ray pulses from the LCLS in the near future, Todd described expected differences of the interaction of intense short wavelength radiation with clusters. Daniel Rolles (Western Michigan University) presented results from recent angle-resolved photoelectron spectroscopy experiments on xenon clusters, where large deviations from atomic anisotropy parameters are observed at low photon energies.

Molecular photoionization was covered from several angles at the workshop. Erwin Poliakoff (Louisiana State University) showed highly resolved valence photoelectron spectra of molecules ranging from CO₂ to C₆F₆ that were used to examine ionization continuum dynamics through experimental and theoretical vibrationally resolved branching ratios. Timur Osipov (Berkeley Lab) described how carbon K-shell photoionization of CO and C₂H₂ studied with the COLTRIMS technique yields insight into the femtosecond electron and nuclear dynamics of the core-ionized states. The momentum-resolved electron-ion coincidence spectra of C₂H₂, for example, exhibit evidence for a vinylidene rearrangement of the core-ionized species before dissociation.

Musa Ahmed (LBNL) expanded on the advantages of VUV photoionization of biological molecules from flash-vaporized aerosol particles to yield fragment-free mass spectra.

Combined with tunable VUV radiation, the method has allowed the ionization potentials of many biologically important molecules to be accurately measured for the first time. Tim Gay (University of Nebraska, Lincoln) discussed the transfer of angular momentum upon photoexcitation of doubly excited states in H_2 by circularly polarized VUV radiation. Dennis Lindle (University of Nevada, Las Vegas) showed how non-polar photoemission is significant in atomic and molecular photoionization of valence and inner-shell electrons, even at relatively low kinetic energies. He also described first results from an attempt to measure drag currents in the photoionization of gases, a macroscopic effect of quantum mechanics.

Photoionization of ions, both positive and negative, was discussed by two speakers. Ronald Phaneuf (University of Nevada, Reno) provided insight into the utility of photoionization studies of positive ions. He gave examples of collective effects in systems such as giant resonances along with producing benchmark experimental data for modeling technologically important plasmas such as xenon and tin, which have been identified as possible sources for EUV lithography. Rene Bilodeau (Western Michigan University) discussed studies of negative ions, focusing primarily on fundamental phenomena such as multiple Auger

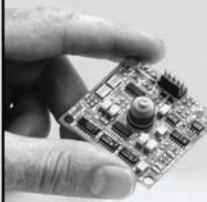
decay and threshold behavior in systems such as He^- and S^- .

The AMO workshop, attended by about 50 scientists, clearly demonstrated the excitement of the community for the field. Forefront AMO science is already being carried out at third-generation synchrotron radiation sources in the US and abroad, as demonstrated by the stimulating talks presented during the workshop. The limiting resource of beamtime at state-of-the-art undulator beamlines, however, limits the ability to fully explore non-equilibrium states of matter: clusters, ions, and dressed-states, to name a few. Expansion of the AMO program at the ALS through the development of a new undulator beamline along with the supporting infrastructure of lasers, beam sources and experimental chambers would greatly advance the research and support development of experimental programs for new femtosecond X-ray sources. ■

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and

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ALS Users' Meeting and Workshops

Frontiers of Synchrotron-Based X-ray Microdiffraction

This workshop was held on October 22, 2005, to celebrate the eightieth birthday of Professor Jamshed (Jim) R. Patel. This was the occasion to acknowledge his contributions, made over more than 50 years, to the field of X-ray scattering and materials mechanical properties and especially his almost 10 years of contributions to the growing field of synchrotron-based X-ray microdiffraction.

Opening remarks and presentations filled with personal anecdotes were given by Bob Batterman (SSRL and ALS) and Bill Nix (Stanford University), who recalled some of the many highlights of Jim Patel's distinguished scientific career, which started with his first and highly cited research paper on the effect of applied stress on the temperature of martensitic transformation, published in 1953 in *Acta Metallurgica*.

He was at that time a research assistant at MIT. In 1961, he was hired by Bell Laboratories in the Materials Physics Division, where he worked for the next 32 years. After a year as an Alexander-von-Humboldt Senior Scientist at Universität München, Germany, he moved to California in 1994, where he held a joint position at the ALS and at Stanford University. His interests have ranged from X-ray

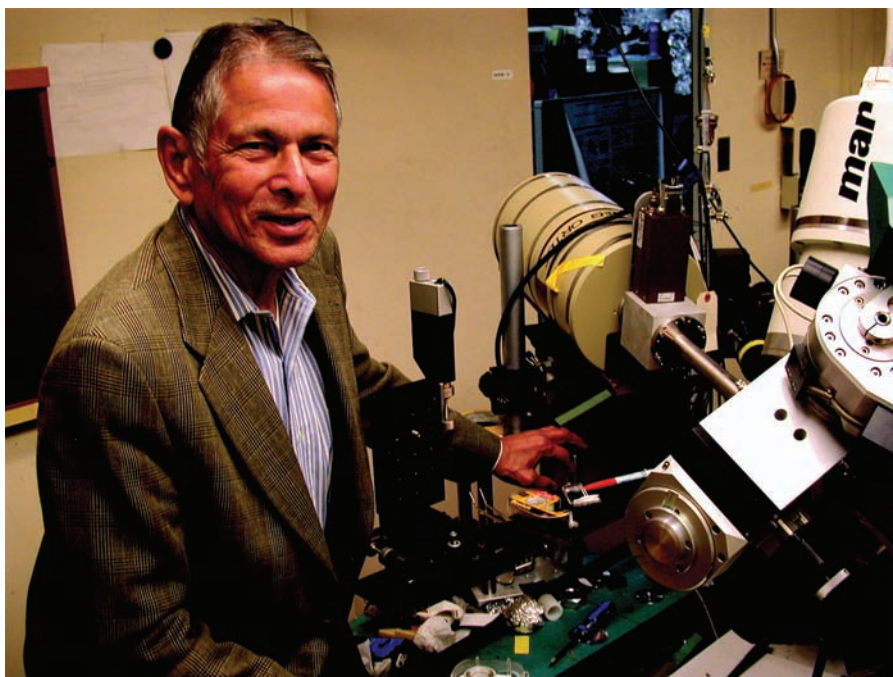
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diffraction topography of dislocations and stacking faults to X-ray standing waves and X-ray diffuse scattering associated with defects in ion-implanted silicon.

In the last decade, Jim has been instrumental in developing a successful X-ray microdiffraction program at the ALS and providing the initial funding for the Beamline 7.3.3 end-station. He initiated many of the materials science applications for the beamline, including the study of electromigration-induced plasticity in interconnect test structures and strain measurements at grain and domain boundaries in superconducting alloys.

The second part of the workshop was devoted to instrumentation to produce focused X-ray beams, which allowed the advent of spatially resolved hard X-ray synchrotron techniques, such as microdiffraction, microfluorescence and microEXAFS. Don Bilderback, associate director of CHESS, described the design and use of monochapillary optics for hard X-ray focusing and showed a string of selected applications in high-pressure physics, protein crystallography, and art and life history. He also gave a status report on the Cornell energy recovery linac X-ray source, which when constructed will offer the prospect of nanometer-sized X-ray beam. Gene Ice (Oak Ridge National Laboratory) talked about the future foreseen for synchrotron X-ray microbeam techniques. He described new ideas for obtaining diffraction-limited nanometer size beams ("nanobeams") by means of reflective mirror technology and the implication of this capability for future scientific applications.

The last part of the workshop was devoted to current scientific applications of the X-ray microdiffraction technique at various synchrotron facilities. Slade Cargill (Lehigh University) presented strain/stress measurements, obtained by using X-ray microbeams at the NSLS (white-beam energy-dispersive microdiffraction) and the APS (Laue microdiffraction), on aluminum and copper thin films and conducting lines on substrates, during elec-



Jim Patel at the ALS X-ray microdiffraction beamline.

tromigration or thermal cycling. Ben Larson (Oak Ridge National Laboratory) reviewed the technique of three-dimensional X-ray structural microscopy recently developed at the APS. This technique, which uses either white or monochromatic light, adds depth resolution to X-ray microbeam diffraction, allowing non-destructive three-dimensional mapping of grain orientation and of elastic and plastic strain with submicron resolution.

Nobumichi Tamura (ALS) gave an overview of the X-ray microdiffraction program at the ALS and of its future, which also served to introduce the next three speakers who had performed their experiments at the ALS. Apurva Mehta (Stanford Linear Accelerator Center) demonstrated the utility of white beam X-ray microdiffraction techniques to study failure modes and mechanical behavior of Nitinol endovascular stents under complex multiaxial loading, as well as to understand crack propa-

gation in metallic alloys. Arief Budiman (Stanford University) presented some recent X-ray microdiffraction measurements obtained during in-situ electromigration testing in copper conducting lines, as well as direct measurements of the plastic strain field produced by nanoindentation on a copper single crystal. Jong-Ook Suh (University of California, Los Angeles) explained how the combined use of X-ray microdiffraction, TEM, SEM, and FIB can contribute to decipher the mechanism of spontaneous growth of whisker in tin and tin alloys, as well as to understand the unique behavior of tin lines and tin solder bumps subjected to a current flow. ■

NOBUMICHI TAMURA
AND BORIS W. BATTERMAN
*Advanced Light Source
Berkeley Lab*

ALS Users' Meeting and Workshops

Novel Approaches to Soft X-ray Spectroscopy: Scanning Transmission X-ray Microscopy and Ambient Pressure X-ray Photoelectron Spectroscopy

This workshop focused on novel spectroscopies at Beamlines 11.0.2, 5.3.2 and 9.3.2 at the ALS. The workshop brought together users from a wide range of fields to highlight recent experimental and technical developments both in scanning transmission X-ray spectroscopy (STXM) and ambient pressure photoelectron spectroscopy (APPEs). The morning session, organized by Mary K. Gilles (Berkeley Lab) and Tolek Tyliczszak (ALS), featured talks on experiments involving new developments at the STXM, while the afternoon session, organized by Hendrik Bluhm (Berkeley Lab) and Simon Mun (ALS), was devoted to those using APXPS.

In the morning session, Tolek Tyliczszak discussed the improved detector developments at the STXM, such as an avalanche photodiode detector and fluorescence and electron detection, as well as the continued development of in situ cells for heating, gas flow, and electrochemical cells. Of these, only the avalanche photodiode [implemented by Hermann Stoll

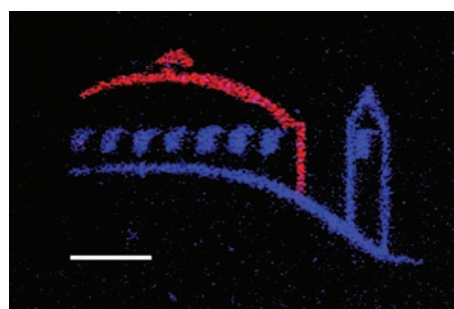
(Max-Planck-Institut für Metallforschung, Stuttgart) and Tolek Tyliczszak] in combination with a novel multichannel photon-counting system designed by Yves Acremann (SSRL) is in routine use in time-resolved studies.

Bartel Van Waeyenberge (Ghent University) presented results of magnetic imaging with a time resolution of 70–100 ps combined with a lateral resolution of 20–40 nm performed with the STXM (Beamline 11.0.2). As a complement to the time-domain “pump-and-probe” measurements, they developed a frequency-domain “sine-excitation” technique in order to study specific eigenmodes of these ferromagnetic patterns with high spatial resolution. This new approach was used to study the gyrotropic vortex motions in micron-sized ferromagnetic patterns.

Adam Hitchcock (McMaster University) presented the development, in collaboration with Daniel Guay (INRS, Varennes) and Sherry Zhang (a former postdoctoral associate), of the apparatus and techniques for applying STXM to in-situ studies of electrochemistry, in particular electrochromism in polyaniline. In addition, substantial progress was reported on a joint project with Harald Stöver (McMaster University) and PhD student Jian Wang to develop substrates and methods for chemically selective lithography of multilayer polymer systems. Selective patterns, such as that displayed in the figure, can now be written efficiently with the bend magnet STXM on Beamline 5.3.2.

Yves Acremann (SSRL) discussed time and spatially resolved X-ray magnetic circular dichroism (XMCD) experiments on spin transfer devices at the STXM (Beamline 11.0.2). These elegant experiments explore time resolved measurements of the magnetization dynamics within a 100 × 150 nm sample influ-

enced by a spin-polarized current. This experiment shows that the magnetization in these magnetic nanostructures are not uniform, as they are influenced by the Oersted field of the charge current needed to generate the spin current. The implementation of a novel multichannel photon counting system in combination with an avalanche photon detector decreased the data-acquisition time by a factor of 10, owing to its ability to resolve the structure of multi bunch mode.



Berkeley Lab logo written in a polymer bilayer by STXM. Blue color represents selective radiation damage in polyacrylonitrile (written/read at 286.8 eV), while the red color represents selective radiation damage in polymethylmethacrylate (written/read at 288.5 eV). Scale bar is 2 microns.

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Gordon E. Brown, Jr. (Stanford University and SSRL) described "Applications of STXM to Microbial Bioweathering and Biomineralization." In the interaction of bacteria with ferrihydrite nanoparticles, microenvironments that were very different than the bulk material were observed, showing that bulk thermodynamics may not be useful for predicting micro phases. Gordon also presented work showing that iron nanoparticles are attracted to the negatively charged bacteria and form a coating that reduces iron oxide minerals.

The afternoon session started with presentations by Simon Mun and Hendrik Bluhm, who discussed the current status and the future plans for the two APPES end-stations at the ALS, which are located at Beamlines 9.3.2 and 11.0.2, respectively. In both end-stations, samples can be measured in gaseous environments at pressures of up to several Torr, which makes possible the investigation of numerous phenomena, in particular in the fields of atmospheric and environmental science as well as heterogeneous catalysis. Specific examples of the application of APPES were shown in the following presentations.

John Hemminger (University of California, Irvine) reported on APPES investigations at Beamlines 9.3.2 and 11.0.2 of the interaction of alkali halide surfaces with water. The measurements showed that upon deliques-

cence (when a layer of saturated solution is formed on the salt surface), the larger, more polarizable anions occupy the solution/vapor interface, which has great implications for the reactivity of, e.g., sea salt aerosols.

Guido Ketteler (Berkeley Lab) showed results of APPES experiments on the adsorption of water on metal oxide surfaces, in particular on $\text{TiO}_2(110)$ and $\alpha\text{-Fe}_2\text{O}_3(0001)$, at ambient temperatures and under Torr pressures. Both surfaces are important substrates in environmental science, and the measurements revealed that already at relative humidities below 1%, molecular water is present at these surfaces.

Klas Andersson (SSRL and Stockholm University) then presented the results of APPES measurements of water adsorption on metal surfaces, also under ambient conditions. These investigations showed that the adsorption of water on copper(110) and (111) shows strong differences. The observations in these molecular scale investigations might help to explain macroscopic phenomena, such as the differences in the wetting of copper(110) and (111) by water.

Spiros Zafeiratos (Fritz Haber Institute of the Max Planck Society, Berlin) reported on the application of a dedicated APPES instrument at BESSY to the investigation of heterogeneous catalytic reactions. The apparatus is optimized for the investigation of realistic cat-

alysts (e.g., powders) and uses a number of different methods for the simultaneous measurement of the gas-phase composition during the APPES experiments, which allows one to correlate the properties of the catalyst surface with its catalytic activity.

Yoshiharu Enta (Hiroshima University) presented APPES investigations of two types of surface reactions, silicon homoepitaxial growth and silicon oxidation. From measurement at various oxygen pressures and sample temperatures, the oxidation rate below one-monolayer oxide coverage of the silicon surface can be explained by an autocatalytic reaction model.

The last talk of the workshop was given by Dennis Nordlund (SSRL), who presented core-hole decay spectroscopy measurements of atom-site-specific electron delocalization rates in water and ice. He also discussed the design of a new APPES system that is under construction at SSRL. ■

HENDRIK BLUHM AND MARY GILLES
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ALS Users' Meeting and Workshops

Soft and Hard X-ray Tomography at the ALS

A day-and-a-half-long workshop on soft and hard X-ray tomography was held as part of the 2005 ALS Users' Meeting. Many more participants than expected turned up, indicating the growth in interest in the technique, and unfortunately resulting in many participants having to sit on the floor!

The workshop opened with the organizers Alastair McDowell and Gerry McDermott (Berkeley Lab) giving overviews of the tomography capabilities at the ALS in the hard and soft X-ray ranges, respectively. This was followed by a comprehensive history of tomography at synchrotron sources by John Kinney (Lawrence Livermore National Laboratory). Marco Stam-

panoni (Paul Scherrer Institute) then discussed the latest developments in micro- and nano-imaging at the Swiss Light Source.

After a short break, Henry Chapman (Lawrence Livermore National Laboratory) gave an illuminating talk on the prospect of coherent diffraction imaging at a resolution of 10 nm. Given that this technique does not rely

on the use of optics (it is commonly known as lensless imaging), the potential benefits offered may be significant compared to more conventional imaging technologies. Wenbin Yun (Xradia Inc.) presented an overview of his company's tomography products and instruments, including a range of zone-plate optics and "off the shelf" X-ray microscopes for materials and biology applications with voxel resolution less than 100 nm. The day closed with an animated discussion of all of the day's presentations.

The second day of the workshop opened with a thought-provoking presentation by Liviu Tomutsa (Berkeley Lab) on applying microtomography to geosequestration research. This work is giving critical insights into the porosity of rocks, in particular their ability to serve as reservoirs of CO₂ "greenhouse gases" produced by power stations, liquefied, and pumped underground for indefinite storage. Guive Balooch (University of California, San Francisco) turned the discussion back to medical and biological applications of tomography with a talk on the structure of genetically modified mouse bones. This work could ultimately help in understanding the weakening of bones as a result of aging or disease.

After a short break, Eric Herbranson (B & H Imaging) gave an enormously entertaining presentation on the dental applications of synchrotron-based tomography, including a description of performing root canal work on a tiger! Piero Pianetta (SSRL) then presented the design of a

new tomography beamline that will soon be installed at SSRL. The beamline will incorporate a modified Xradia commercial instrument and promises to be up and running in the coming year. Weiwei Gu (University of California, San Francisco) then gave an in-depth description of the soft X-ray tomography beamline that is nearing completion at the ALS as the centerpiece of the National Center for X-ray Tomography (<http://ncxt.lbl.gov>), together with examples of work NCXT has performed with the existing X-ray microscopes at the ALS. The last talk of the day was a description by Weilun Chao (LBNL) of the recent advances in zone-plate-fabrication methodology that have extended the attainable resolution to around 10 nm.

The day closed with a wide-ranging discussion about the future funding of tomography and how to make the existing facilities even better. All in all, this was an exciting and informative workshop that served to illustrate the breadth of science that is now making use of both hard and soft X-ray tomography beamlines at synchrotrons around the world. ■

GERRY McDERMOTT

*Physical Biosciences Division
Berkeley Lab*

and

ALASTAIR MACDOWELL

*Advanced Light Source
Berkeley Lab*

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ALS Users' Meeting and Workshops

THz Science and Technology Network: Opportunities and Organization

There has been a recent explosion of interest in using terahertz (THz) radiation to investigate questions in chemistry, biology, physics, medicine and materials science.

Simultaneously, there has been a rapid development of a broad array of experimental tools for working with THz radiation. In response, we have proposed a *collaboratory* [a collabo-

ratory is "an organizational entity that spans distance, supports rich and recurring human interaction oriented to a common research area, and provides access to data sources, artifacts,

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and tools required to accomplish research tasks," from the Science of Collaboratories, 2003 report] called the THz Science and Technology Network.

The network organization and opportunities workshop was held October 21 and 22, 2005. Opening the first day of presentation, Michael Martin (ALS) welcomed those attending and introduced the first speaker, Mark Sherwin (University of California Santa Barbara), who reviewed the goals and mission of the THz Science and Technology Network. The Network's goals are to lower barriers to experimental and theoretical research involving THz radiation, to foster interaction and technological innovation, and to expand the THz community. The Network will provide necessary infrastructure that is beyond the means of any individual investigator.

A key component of the Network is a set of nodes with research tools and expertise that together span the current state of the art. These

nodes will provide open access to all qualified users. A second important component is an extensive website that will provide thumbnail sketches of terahertz research areas, detailed descriptions of the nodes, expert contact lists, a virtual journal, a spectral database, calibration data, educational materials, an interactive virtual spectrometer, a buyers guide, how-to manuals, press releases, a jobs clearinghouse, conferences, and FAQs. Other components include education, outreach, development of health and calibration standards, interface with industry, and international cooperation.

Raymond Jeanloz (University of California, Berkeley) followed with a report about the Consortium for Materials Properties Research in Earth Sciences (COMPRES), which operates a high-pressure research beamline at the ALS. After a break, John Shott (Stanford University) and Mark Rodwell (University of California, Santa Barbara)

described the National Nanotechnology Infrastructure Network. The day ended with an open discussion of the THz Network Interim Steering Committee. Present from the Interim THz Network Steering Committee were Mark Sherwin, Charlie Schmuttenmaer (Yale University), Michael Martin, and Andrea Markelz (University at Buffalo).

The second day was devoted to a series of closed steering committee round table discussions, ending with a closeout and lists of action items for committee members.

To find out more about and/or join the THz Science and Technology Network, please visit <http://www.THzNetwork.org>. ■

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ALS Users' Meeting and Workshops

Ultrafast X-ray Science at the ALS

More than 50 participants attended the workshop "Ultrafast X-ray Science at the ALS" and experienced an exciting overview of recent research and future scientific applications in the area of ultrafast dynamics on a sub-nanosecond to femtosecond time scale using X-rays at the ALS.

Sasha Zholents (Berkeley Lab) proposed a scheme for a high-flux source at the ALS based on electron bunch rotation that would allow for experiments with picosecond to femtosecond time resolution at a repetition rate of 500 MHz. The current status of the new femtosecond undulator Beamline 6.0.1 at the ALS was presented by Phil Heimann (Berkeley Lab), who reported that the first light was delivered into that beamline very recently.

The first session was organized jointly with the parallel "Forefront AMO Science: Clusters, Ions, Dressed States" workshop held

during the same period (see report in this issue). The focus on AMO research began with Marcus Hertlein (Berkeley Lab), who reported on ultrafast measurements to study electron correlation in atoms and molecules. Initial experiments investigating laser/X-ray ionization processes in potassium have been developed at the ALS. Future experiments are planned for the Sub-Picosecond Photon Source (SPPS) at SLAC. Rounding out the joint session, experiments on ultrafast dynamics at the APS were presented by Linda Young (ANL), who talked about time-resolved X-ray microprobe of strong-field processes in atoms and molecules.

Spin dynamics on a sub-nanosecond to femtosecond time scale is a major research area at the ALS, as demonstrated in several presentations. Andreas Bartelt (Berkeley Lab) reported

on the development and first experiments with an ultrafast X-ray streak camera system to study magnetization dynamics in thin films. His talk was continued by Alberto Comin (Berkeley Lab), who discussed the picosecond spin dynamics of iron-gadolinium, which was studied with time-resolved X-ray magnetic circular dichroism. The same magnetic contrast mechanism is also used for time resolved X-ray microscopy studies with high lateral resolution. This technique was described by Hermann Stoll (MPI Stuttgart), who reported on studies of precessional phenomena in ferromagnetic microelements obtained at the ALS. Yves Acremann (SLAC) showed stunning results on the direct observation of current-induced switching in spin-transfer devices. An exciting introduction into domain wall dynamics in magnetic nanowires was given by Dan Allwood (University of

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Attendees listen to Marcus Hertlein's (Berkeley Lab) presentation on ultrafast measurements of electron correlation.

field with applications in numerous scientific fields including molecular dynamics, atomic and electronic phase transitions in solids, AMO physics, and magnetization dynamics. At present, the ALS plays a leading role in ultrafast X-ray research and will continue to be an exciting and attractive facility for users with the completion of the new femtosecond X-ray beamlines. In the future, the possible addition of an elliptically polarized undulator (for ultrafast magnetization studies) and the implementation of a novel bunch-rotation scheme (for higher average femtosecond X-ray flux) will ensure that the ALS continues to play an important role in ultrafast X-ray science, with capabilities that will complement those of the new LCLS. ■

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Sheffield), who stated that in the future these studies will require both the time and the spatial resolution offered by the ALS.

The need to extend photoemission spectroscopy into the time realm was the topic of the presentation by Alessandra Lanzara (University of California, Berkeley), who clearly described the missing links to disentangle coupled phenomena that give rise to novel material phases. Strongly correlated materials were also discussed by Andrea Cavalleri (University of Oxford), who described both femtosecond X-ray diffraction of polariton dynamics in ferroelectrics and the first femtosecond X-ray absorption spectroscopy measurements of electronic structural dynamics in correlated electron materials.

An extensive overview on future experiments on ultrafast dynamics that will be accessible at the Linac Coherent Light Source (LCLS) at SLAC was given by Roger Falcone (UC Berkeley, Berkeley Lab). Aaron Lindenberg and Kelley Gaffney (SLAC) described recent experiments on non-thermal laser melting in indium antimonide by femtosecond X-ray diffraction at the SPPS source. These impressive results showed not only the devel-

opment of the liquid state within 200 fs, but also exhibited evidence of inertial anisotropy resulting from the prompt changes in the bonding initiated by the laser excitation.

Lin Chen (Argonne National Laboratory) gave an extensive overview of her work on time-resolved X-ray spectroscopy of transition-metal molecular complexes at the Advanced Photon Source (APS). This work identifies for the first time the structure of molecular intermediates forming on the 100-ps time scale. Munira Khalil (UC Berkeley, Berkeley Lab) discussed research at ALS Beamline 5.3.1 using X-ray absorption spectroscopy to investigate atomic and electronic structural dynamics associated with spin-crossover reactions in solvated transition metal complexes. Recent results show an 0.2-Å dilation of the metal-ligand bonds within 70 ps in the Fe(II) system. Future applications of these time-resolved techniques to metal-porphyrins and other biological systems were discussed.

The workshop has shown that research on ultrafast dynamics with X-rays is a scientifically exciting and technologically challenging

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ALS Users' Meeting and Workshops

XANES and EXAFS Spectroscopy of Materials and Biological Samples: Expanding the Range of Applications at Beamline 9.3.1

ALS beamline 9.3.1 produces X-ray beams in the energy range 2.4–5.5 keV, thus including the K-edges of sulfur and chlorine, as well as the L-edges of many second-row transition metals. A workshop was organized to summarize the latest scientific results obtained at BL9.3.1, update users about recent improvements to the beamline, discuss new research opportunities, and outline short- and long-term roadmaps for future development of BL9.3.1.

The scientific scope of the meeting included a wide selection of applications for X-ray absorption and X-ray emission spectroscopies, ranging from chemical biology to atomic physics. The highlighted biophys-

ical, materials science, and atomic physics topics were the sulfur and chlorine K-edge spectroscopy of sulfur-nitroso compounds and ruthenium-containing anticancer complexes, electronic and geometric structures of palladium, titanium, niobium, and copper catalysts, and X-ray emission studies of ultrafast dissociation of chlorine-containing molecules.

The workshop began with an overview of selected scientific insights gained from measurements at the beamline. The series of talks was opened by Robert Szilagy (Montana State University), who presented his results on using sulfur K-edge XAS as a diagnostic probe for detecting sulfur-nitrosated cysteines

by means of the characteristic spectral features of sulfur-nitroso thiols relative to cysteine, ethylthiolate, and methionine [1]. He also discussed recent progress in his group toward developing a quantitative analysis of palladium L-edge XAS and its use to probe the catalytic activity of palladium(II) and palladium(IV) complexes [2].

Heinz Frei (Berkeley Lab) gave a presentation explaining how XAS can aid the design of bimetallic photoreactors that accomplish redox reactions under visible light. His group used titanium K-edge XAS measurements for characterizing the geometric and electronic structures of the redox centers. XAS and time-resolved FT-IR measurements were used to



unravel the molecular mechanism of photoactivation of carbon dioxide by water, as an electron source, on a surface of functionalized mesoporous silicates. The silicates were functionalized by visible-light-absorbing bimetallic units, such as titanium(IV) or ruthenium(III) bridged by an oxo ligand to copper(I) or iron(II) [3–4].

Karen McFarlane Holman (Willamette University) summarized her XAS measurements on ruthenium complexes with chloro, DMSO, imidazole, and indazole ligands, which are potential candidates for anti-cancer complexes. The research in her group conducted by undergraduate students focuses on correlating spectroscopic properties of ruthenium(III) and ruthenium(II) complexes with anti-cancer activities.

Alex Bell (University of California, Berkeley) talked about the multi-element and multi-edge XAS studies conducted at ALS and SSRL of copper-exchanged zeolite catalysts [5]. He introduced his group's design of an *in-situ* sample cell for characterization of solid samples by XAS [6] and presented results obtained with this "lab-on-a-chip" cell at the copper L-edges. The group has used chlorine K-edge XANES to characterize and optimize the synthesis of modified Y-zeolite catalysts for methanol oxidative carbonylation and methanol partial oxidation reactions prepared from a CuCl precursor.

The second session discussed the present beamline status and improvements since a similar workshop held in 2002 [7]. Beamline scientist Fred Schlachter (ALS) provided a comprehensive and compelling presentation about the technical details behind a stable, reliable, user-friendly, and safe operation of BL9.3.1. Since the last workshop, several important upgrades have been implemented, including temperature stabilization of the monochromator, engineered crystal mounts and rotations, LabView control and data-acquisition system, user-friendly graphical user interface, *in-vacuum* motion controls and encoders, and most importantly, a feedback system in two planes to stabilize beam position on a sample. A large suite of end-sta-

tions is available for users, including an absorption cell for gases, ion and electron time-of-flight and magnetic mass spectrometers, anion-cation coincidence, X-ray emission, and EXAFS end-stations. He emphasized the responsibility of the user community to suggest new science, new capabilities, and new end-stations.

A successful new capability is represented by the development and installation of the X-ray emission end-station by Dennis Lindle and Wayne Stolte (University of Nevada, Las Vegas) and their collaborators. Marc Simon (Université Pierre et Marie Curie) gave a scientific presentation about resonant inelastic X-ray scattering results of studying the ultrafast dissociation processes of chlorine-containing molecules, the first application of this end-station.

The workshop discussion concluded that in the current beamline configuration, XAS measurements for quantitative analysis can be carried out for solid samples with similar data quality as obtainable at other beamlines in the "tender" X-ray energy range (2–5 keV) at the national synchrotron facilities. The possibility of liquid-nitrogen cooling the sample rod comes close to eliminating radiation damage and photoreduction. Ideas and suggestions were discussed for improving the data quality by installation of a new fluorescence detector with a larger collector or electron-yield detector with a large collection potential. An alternative solution for eliminating self-absorption issues of thick solid samples measured in ultrahigh vacuum would be the development of a transmission setup by installation of an additional foil (I1) behind the sample chamber.

Extension of the beamline functionality to dilute environmental and biological samples requires close to atmospheric pressure in the sample chamber. The chamber needs to be separated from the beamline by a silicon nitride (currently installed) or a beryllium window, which would reduce the beam intensity by an estimated 30%. Optics improvements are expected to improve beamline performance. A strong interest was

expressed for future installation of InSb(111) and Ge(111) crystal monochromators to access silicon and phosphorous K-edge energy ranges. ■

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MEETING REPORTS

ALS Users' Meeting and Workshops

Soft-X-ray Photon-in and Photon-out Spectroscopy: New Frontier

Advanced photon-in and photon-out soft-X-ray spectroscopy is emerging as a powerful new experimental tool in areas ranging from atomic and molecular physics to materials, molecular environmental, and biological sciences. At the 2005 annual ALS Users' Meeting, a workshop on "Soft-X-ray Photon-In and Photon-Out Spectroscopy: New Frontiers" was held on October 21 and 22. More than 50 participants registered for the workshop organized by Jinghua Guo (Berkeley Lab) and Zahid Hasan (Princeton University)

We invited a number of international experts with a wide range of interests such as complex materials, environmental and interfacial science, and atomic and molecular

phenomena, but with a common interest in photon-in/photon-out soft-X-ray spectroscopic experiments. The speakers addressed the importance of the experimental and theoretical developments in resonant elastic and inelastic scattering and the potential applications in in-situ chemical and biological applications. Resonant inelastic soft-X-ray scattering spectroscopy (RIXS) is a powerful technique, owing to its unique characteristics: (1) low energy excitations in neutral system; (2) two-photon process; (3) dispersion through momentum conservation; (4) site selectivity due to core resonance; (5) lack of core state broadening; (6) localization dependence; and (7) femtosecond dynamics.

A number of groups have applied soft-X-ray spectroscopy in the studies of liquids and liquid/solid interfaces, ion interaction and solvation as reported by Emad Aziz and Stefan Eisebitt (BESSY), and Clemens Heske (University of Nevada, Las Vegas). Photon-in and photon-out spectroscopy is finding applications in hydrogen bonding network, volatile liquids and their surfaces, and it also brings up the ongoing debate on the hydrogen-bonded molecular structure of liquid water, which may or may not be as close to the end as presented by Richard J. Saykally (University of California, Berkeley), Philippe Wernet (BESSY), and Anders Nilsson (SSRL). In-situ characterization has yielded important information on corrosive reactions and actinide chemis-

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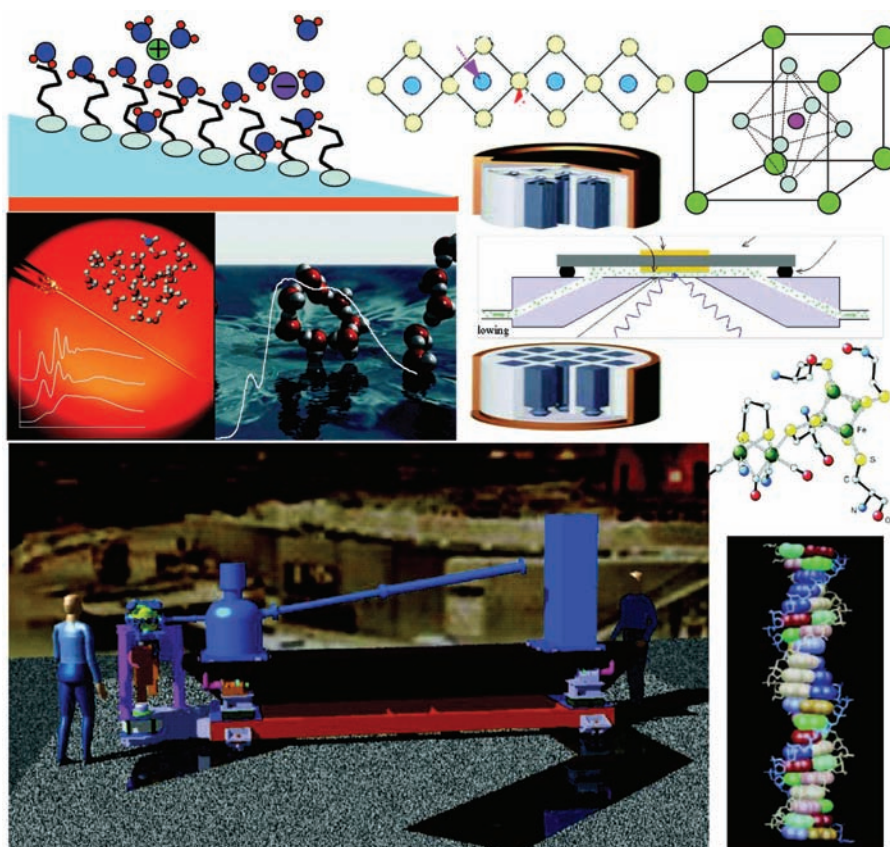
try, as reported by Laurent Duda and Sergei Butorin (Uppsala University).

As all speakers learned, theory is an essential tool, especially in understanding photon-in and photon-out spectroscopy. Akio Kotani (SPring-8 and RIKEN) showed the importance of collaborations of theory and experiment and pointed out that the combination of *ab initio* energy band calculation and the impurity Anderson model is a powerful new method for theoretical calculations of resonant X-ray emission spectroscopy. Arun Bansil and Robert Markiewicz (Northeastern University) gave presentations on a consistent picture of magnetic gap collapse in $\text{Nd}_{2-x}\text{Ce}_x\text{CuO}_4$ and on probing magnetic gap collapse within a three-band model, respectively.

Alex Moewes (University of Saskatchewan) and Simon George (LBNL) demonstrated that experimental access to the electronic structure is essential for understanding the physical and chemical properties of many chemical and biological systems. Amanda C Hudson (University of Nevada, Las Vegas) showed that Cl *K*-edge emission can be recorded at ALS.

Jessica Thomas (NSLS) and Chuck Fadley (University of California, Davis and Berkeley Lab) presented two different ways of detecting magnetic correlations in complex materials; namely, resonant X-ray diffraction and standing-wave-excited soft-X-ray resonant inelastic scattering. Low energy excitations, such as dd- and ff-excitations, can be revealed by soft-X-ray resonant inelastic scattering, as shown in the presentations of Per Anders Glans (Boston University) and Giacomo Ghiringhelli (Politecnico di Milano). Jonathan Denlinger (Berkeley Lab), Tony van Buuren (Lawrence Livermore National Laboratory), and Thorsten Schmitt (Swiss Light Source) also showed soft-X-ray photon-in and photon-out spectroscopy can be an alternative technique for determining the band gap of semiconductors and quantum confinement in nanostructured materials.

The frontier of photon-in and photon-out spectroscopy is to become a high-resolution spectroscopy. But the challenges are: (1) energy resolution is difficult to obtain because we need high $E/\Delta E$ as resolution enters the 100-meV scale; and (2) the signal is very weak



A collection of soft-X-ray photon-in/photon-out spectroscopy experiments

because of low cross sections and the use of spectrometers. In its current state of development, photon-in and photon-out spectroscopy requires a state-of-the-art synchrotron beamline as a source. One wish we all have: if beamline and analyzer monochromator resolution can be made high enough, then resonance-enhanced X-ray Raman spectroscopy will become a site-specific vibrational technique.

If photon-in and photon-out spectroscopy can be developed as a high-pressure and surface-sensitive technique, it will open a new field in basic and applied research. Challenges for the ALS lie in developing photon-in and photon-out techniques for monolayers and nanoparticles and peptide and protein monolayers at polymer-water interfaces. Instrumental challenges include better time resolution and better spatial resolution. Where are areas for future improvements? Obtaining quantifiable spectra

and studying complex materials in different phases, such as liquid and solid under surface-preparation conditions. Finally, we have to avoid and understand radiation damage.

In summary, modern soft-X-ray photon-in and photon-out spectroscopy possesses features that offer new research opportunities in materials, molecular, environmental, and biological sciences. Some of the presentations can be found on the Web at: [http://www-als.lbl.gov/ssg/ssgdirectory/guo usermtg05_xrayspec.htm](http://www-als.lbl.gov/ssg/ssgdirectory/guo%20usermtg05_xrayspec.htm) ■

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