ALS SPECTRUM

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What's Inside:

U.S. DEPARTMENT OF ENERGY
Office of Science

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ALS ADVANCED LIGHT SOURCE

ALS COMMUNICATIONS

Exquisite X-Ray Optics

by Patrick Naulleau, Dimitri Voronov, and Howard Padmore

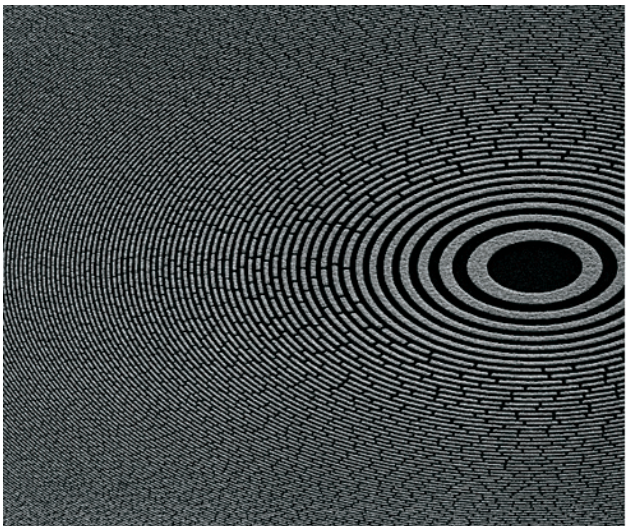

Image of a zone-plate lens showing progressively finer concentric zones going from the center to the outer edge.

At the heart of many instruments at the ALS are diffractive optical elements: zone plates for microscopy and gratings for spectroscopy. Here we describe ongoing efforts to improve the characteristics of both types of element, using fabrication techniques with precision in the nanometer regime and beyond. These techniques are bringing us a new generation of optics, capable of unprecedented spatial and spectral resolution and that are expected to have a dramatic impact on the scientific program of the ALS.

Diffractive optics such as Fresnel lenses are the key to ultrahigh-resolution soft x-ray microscopes. Fresnel lenses are functionally equivalent to the more common refraction lenses but work by bending light via diffraction through concentric ring sections (zones) that get progressively finer from center to edge. The ultimate resolution that can be produced by a Fresnel lens is determined by the finest zone width at the outer edge of the lens. A simple rule of thumb is that the achievable resolu-

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Falcone: Looking Forward

by Roger Falcone

Roger Falcone

After 18 years of round-the-clock operations, the ALS continues to serve a broad and growing community of 2000 users, resulting in 600 peer-reviewed journal articles per year. These metrics demonstrate a strong record of achievement to the Department of Energy (DOE) and to Congress. Also, I believe that they are appropriate measures of productivity and represent a significant return on the investment of taxpayer dollars.

Additionally, we have other responsibilities and we contribute in many other important ways. These are not necessarily obvious or static because of our evolving role as a national user facility and in our rapidly changing economic situa-

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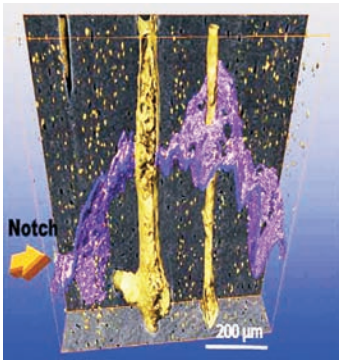
Science Roundup

Irradiation Effects on Human Cortical Bone Fracture Behavior

Barth et al., *Bone* **46**, 1475 (2010)

Human bone is strong but still fallible. To better predict fracturing in bone, researchers need a mechanistic framework to understand the changes taking place on different size scales within bone, as well as the role of sustained irradiation damage. Combining in situ mechanical testing with synchrotron x-ray diffraction imaging and/or tomography is a popular method of investigating micrometer deformation and fracture behavior in bone. However, the role that irradiation plays in these high-exposure experiments, and how it affects the properties of bone tissue, are not yet fully understood. A team of researchers used microtomography at Beamline 8.3.2 to investigate changes in crack path and toughening mechanisms in human cortical bone with increased exposure to radiation.

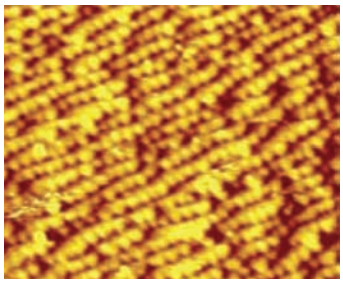


X-ray tomography shows a crack's path (purple/pink) in non-irradiated human bone. The yellow structures are canals containing nerve and blood supplies. The crack deflects upon encountering regions of compact bone surrounding the cavities.

Platinum Nanoclusters Out-Perform Single Crystals

Tao et al., *Science* **327**, 850 (2010)

When it comes to metal catalysts, platinum is the standard. However, at about $2,000 an ounce, the high cost of the raw material presents major challenges for the future wide-scale use of platinum in fuel cells. Berkeley Lab research suggests that one possible way to meet these challenges is to think small. Researchers from Berkeley Lab's Materials Sciences Division have found that under high pressure—comparable to the pressures at which many industrial technologies operate—platinum surfaces can change their structure dramatically in response to the presence of high-coverage reactants. High-pressure scanning tunneling microscopy (STM) and ambient-pressure x-ray photoelectron spectroscopy (AP-XPS) at Beamlines 9.3.2 and 11.0.2 allowed researchers to study catalysts' structure and composition under realistic conditions.

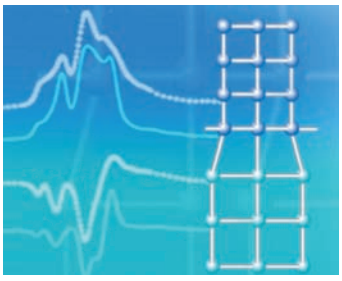


STM images showed that, when the surface of a platinum catalyst is covered by CO at a pressure of one torr, the adsorbed CO causes the platinum surface to fracture into triangular-shaped nanoclusters. AP-XPS spectra revealed a change in CO electron binding energies.

Probing Strain-Induced Changes in Electronic Structure with XMCD

van der Laan et al., *Phys. Rev. Lett.* **105**, 067405 (2010)

The difference in the lattice constant of a substrate compared to a thin film deposited on top of it or a matrix compared to a nanocolumn embedded in it induces a lattice distortion—i.e., strain—in the nanostructure as compared to the bulk. The strong coupling of electronic properties with structural parameters in transition-metal oxides then allows tuning and ultimately controlling the physical characteristics of nanoarchitectures through strain. A research team has now demonstrated that soft x-ray magnetic circular dichroism (XMCD) techniques possible at Beamline 4.0.2 are uniquely suited to provide detailed information on the impact of strain on the electronic properties of magnetic oxide nanoarchitectures in an element-, valence-, and site-specific way.



It is of great practical interest for the strain engineering of novel materials—i.e. for tuning and controlling materials properties through lattice distortions—that a very small axial distortion completely determines the angular dependence of the XMCD.

New Morphological Paradigm Uncovered in Organic Solar Cells

Collins et al., *J. Phys. Chem. Lett.* **1**, 3160 (2010)

Organic solar cells are made of light, flexible, renewable materials; they require simple and inexpensive processing and could produce an economically competitive and environmentally friendly energy source. Models describing critical functions such as charge separation and transport often depend on simplistic morphological assumptions, including discrete interfaces between pure electron donor and acceptor materials. In contrast, recent spectroscopy and scattering studies conducted at Beamlines 5.3.2 and 7.3.3 found a substantial amount of molecular mixing between model materials (polymers and fullerenes) currently used in bulk heterojunction organic solar cells. This suggests that the amorphous portions of these devices do not have pure domains, and the paradigm of device operation may need to be refined to accommodate this newly discovered complexity.

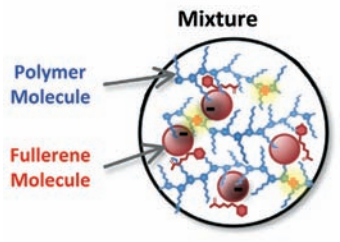


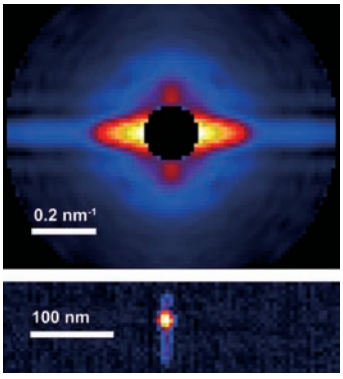
Illustration of the high level of molecular mixing in the amorphous phases of photovoltaic devices—a size scale smaller than was previously thought proper for obtaining good charge transport and high device efficiencies.

Read more about these and other science highlights at www-als.lbl.gov/index.php/science-highlights/science-highlights.html

A New Light on Disordered Ensembles

Saldin et al., *Phys. Rev. Lett.* **106**, 115501 (2011)

Because individual biomolecules are very small, x-ray scattering experiments usually determine their structures by an analysis of scattering from a large number of them. In crystallography, scattering by many molecules in identical orientations vastly enhances the signal from a single molecule. However, not all biomolecules form crystals. They are more usually found in disordered ensembles in aqueous solutions or in biomembranes. Researchers have now performed at Beamline 9.0.1 the first experimental demonstration of a method that amplifies the information in the x-rays that scatter from disordered biomolecules, allowing the reconstruction of an image of a single molecule from fluctuations in the scattering from an ensemble of randomly oriented copies.

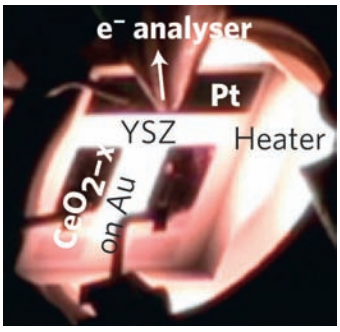


Top: Reconstructed diffraction pattern of a single particle from a statistical analysis of numerous measured diffraction patterns generated by an ensemble of randomly oriented particles. Bottom: Real-space image of a nanoparticle reconstructed from the diffraction pattern above.

AP-XPS Measures MIEC Oxides in Action

Zhang et al., *Nat. Mater.* **9**, 949 (2010); DeCaluwe et al., *J. Phys. Chem. C* **144**, 19853 (2010)

Oxides with mixed ionic-electronic conductivity (MIEC) can conduct both electrons and oxygen ions. Uses for MIEC oxides include solid-oxide fuel cells, high-temperature electrolysis for synthetic fuel production, and oxygen-separating membranes for chemical processes or NO_x-free combustion. However, because laboratory-scale x-ray sources have traditionally required ultrahigh vacuum, it was previously impossible to measure the surface oxidation states of active electrode surfaces using x-ray photoelectron spectroscopy (XPS). A team of researchers used Beamlines 9.3.2 and 11.0.2 to overcome the limitations of conventional XPS using ambient-pressure x-ray photoelectron spectroscopy (AP-XPS), providing the first in situ measurements of local surface oxidation states and electric potential in active MIEC electrodes.

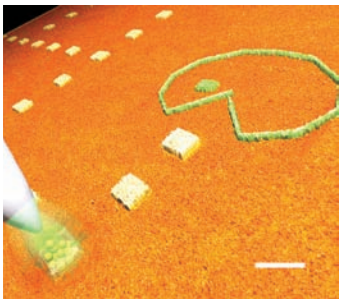


Single-chamber solid-oxide electrochemical cell. This geometry exposes all cell components to the x-ray beam. Combined with the use of AP-XPS at temperatures and pressures representative of reaction conditions, this setup lets researchers study MIEC oxides' active electrode surfaces.

Direct-Write of Silicon and Germanium Nanostructures

Torrey et al., *Adv. Mater.* **22**, 4639 (2010); Vasko et al., *Nano Lett.* **11**, 2386 (2011)

Nanostructured materials (nanowires, nanotubes, nanoclusters, graphene) are attractive possible alternatives to traditionally microfabricated silicon in continuing the miniaturization trend in the electronics industry. To go from nanomaterials to electronics, however, the precise one-by-one assembly of billions of nanoelements into a functioning circuit is required—clearly not a simple task. An interdisciplinary team has devised a strategy that could make this task a little easier. They have demonstrated the ability to directly "write" nanostructures of Si, Ge, and SiGe with deterministic size, geometry, and placement control. As purity is essential for electronic-grade semiconductors, the resulting patterns were carefully evaluated for carbon contamination using photoemission electron microscopes at Beamlines 7.3.1 and 11.0.1.

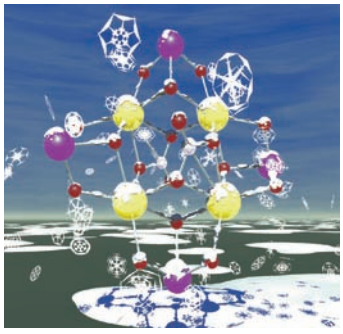


The Pac Man (germanium) and one Pac Dot (silicon) are actual nanostructures created by the direct-write process. The height of the structures is about 5 nm. Scale bar = 5 µm. A similar image was featured on the cover of *Advanced Materials,* November 2, 2010.

Cool Magnetic Molecules

Karotsis et al., *J. Am. Chem. Soc.* **132**, 12983 (2010)

Certain materials are known to heat up or cool down when exposed to a changing magnetic field. In most cases, this effect is too small to be technologically useful. Recently, however, the search for special molecules with a surprisingly large capacity to keep cool has heated up, driven by environmental and cost considerations as well as by recent improvements in our ability to design, assemble, and probe the structure and chemistry of small molecules. Researchers have utilized Beamline 11.3.1 (small-molecule crystallography) to characterize the design of such "molecular coolers." The work targets the synthesis of molecular cluster compounds containing many unpaired electrons ("nanomagnets") for applications involving enhanced magnetic refrigeration at very low temperatures.



The "frozen" $[Mn_4Gd_4]$ cluster pictured is a promising candidate for refrigeration in the ultra-low-temperature region, providing for example a valid alternative to the use of helium-3, which is becoming rare and expensive.

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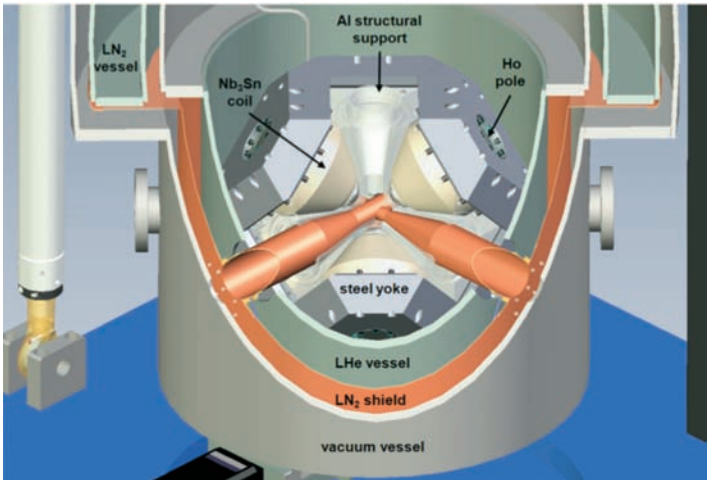
SCIENTIFIC SUPPORT GROUP (SSG): MAGNETS, MERLIN, & MORE

by Zahid Hussain, Mike Martin, and Eli Rotenberg

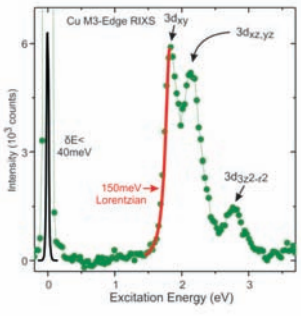
Vector Magnet for Soft X-Ray Spectroscopy. The development of new materials with specific electronic and magnetic properties designed for use in novel, low-cost, and energy-efficient nanodevices with improved performance and multiple functionalities is a major challenge for materials science and engineering today. Soft x-ray magnetic dichroism—i.e. the dependence of the x-ray absorption cross section on the relative orientation of x-ray polarization and magnetic moments—is a unique tool for improving our understanding of these nanoarchitectures because it provides element-, valence-, and site-specific magnetic and electronic information with high sensitivity and tunable probing depth.

The ALS is now developing a superconducting eight-pole magnet providing magnetic fields of up to 5 T in arbitrary directions, which will allow combined x-ray magnetic circular and linear dichroism studies to investigate the magnetic and electronic structure of heteromagnetic nanosystems. Magnet center fields exceeding 5 T leads to 12-T peak fields on the conductor, necessitating the use of Nb_3Sn superconducting wire for the magnet coils. Moreover, holmium will be used for the magnet poles because it has a higher saturation field than iron and provides a larger contribution to the magnet center field for the same current density. The design of the cryogenic system includes liquid nitrogen and liquid helium reservoirs with volumes that are dictated by the desired hold time and heat load on the system.



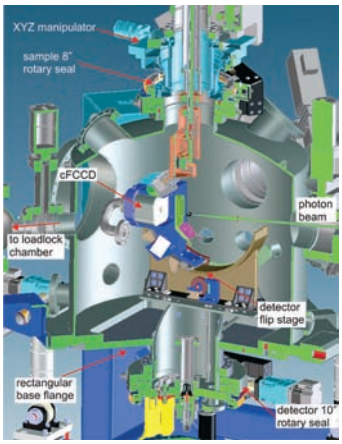
Design of the superconducting vector magnet.

MERLIN Making Progress in Commissioning and Producing Science. Beamline 4.0.3 (MERLIN) is a low-photon-energy beamline with an energy range from ~10 eV up to 120 eV and is dedicated to high-energy-resolution angle-resolved photoemission (ARPES) and resonant inelastic x-ray scattering (RIXS) spectroscopy. The beamline is under commissioning but is now open for a limited number of general-user proposals. The ARPES endstation is equipped with a Scienta R8000 electron spectrometer and a low-temperature (< 6 K) sample cryostat manipulator with six axes of motion. The RIXS endstation is equipped with a 2-m-long x-ray emission spectrograph, and with current K-B mirrors, a combined resolving power (beamline plus the spectrograph) on the order of 2000 can be achieved. Such energy resolution (< 40 meV at the Cu M_3 edge) enables researchers to observe the intrinsic lifetime of dd excitons in cuprates without being limited by the resolution of the instrumentation. This resolution is also allowing the study of the many-body quantum structure of dd excitons (paper under preparation).



Resonant inelastic x-ray scattering spectrum at the M_3 edge of the cuprate $SrCuO_2$.

New Scattering Chamber for Studying Charge Dynamics at the ALS and LCLS. A resonant elastic x-ray scattering (RSXS) endstation was constructed to carry out static and time-resolved RSXS experiments at the ALS and the Linac Coherent Light Source (LCLS). This endstation, with a compact fast CCD (cFCCD) detector (designed at Berkeley Lab) mounted on an in-vacuum goniometer, is capable of detecting superlattice reflections from electronic orderings in the lower hemisphere of the experimental chamber. The six-degree-of-freedom sample goniometer has temperature control (from ~12 K up to 400 K) and the flexibility to orient the sample with respect to photon polarization. This endstation has been extensively used at Beamline 8.0.1 at the ALS and the SXR beamline at the LCLS to study the charge, spin, and orbital ordering phenomena in transition-metal oxides like cuprates, nickelate, manganite, and magnetities.



A CAD model showing the major components of the RSXS chamber [Doering et al., *Rev. Sci. Instrum.* **82**, 073303 (2011)].

Lensless X-Ray Imaging in Reflection. The advent of free-electron x-ray laser sources has led to a burst of research in lensless imaging, which is proving

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Scientific Support Group

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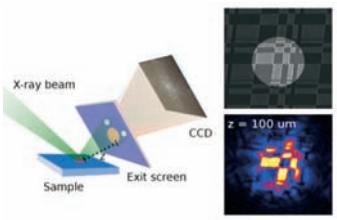
to be a key component for single-shot, high-resolution x-ray imaging of both complex materials and biological samples. Existing techniques are typically designed for transmission geometry and use isolated objects, requiring special sample fabrication and restricting the type of samples under investigation. While many specimens can be prepared to work in transmission, others—particularly in materials physics—will require samples that are both extended and nontransmissive. There is currently no easy way to perform such measurements. Recently, researchers from the SSG and the University of Oregon have shown that it is possible to form a hologram in reflection by using the light scattered from a sample [Roy et al., *Nat. Photonics* **5**, 243 (2011)], opening the door to coherent imaging of a wealth of new material samples. Combining holography with ultrafast dynamics experiments will open up real-time direct observation of surface or interface dynamics.



Diffractive imaging in reflection geometry.

Mentoring and Pipeline for Future Scientists. Establishing a pipeline for future beamline and accelerator scientists is vital to all DOE BES user facilities. Many SSG members mentor students at all levels throughout the year. The SSG has also demonstrated the ALS to be a leader in preparing future synchrotron scientists by setting up graduate student and postdoctoral fellowship support programs (www.als.lbl.gov/als/fellowships/).

Awards and Honors. Several SSG members received prestigious awards and honors in the past year. Eli Rotenberg received the inaugural 2010 VUVX conference award for his development of an advanced ARPES facility and its insightful application to a broad variety of forefront problems. Zahid Hussain, with Miquel Salmeron, D. Frank Ogleletree, and Hendrik Bluhm, received a 2010 R&D 100 Award for the development of APPELS: a differentially pumped ambient-pressure photoeletron lens system for photoemission studies. Tolek Tyliszczak and David Kilcoyne shared with others the 2010 Klaus Halbach Award for innovative instrumentation at the ALS for hitting the 10-nm-resolution milestone with soft x-ray microscopy.

New Science. The following selected highlights from the past year not only reflect world-class science but also illustrate the unique capabilities made available at the ALS by the SSG: AP-XPS Measures MIEC Oxides in Action, *Nat. Mater.* **9**, 949 (2010); Superconducting Topological Insulators, *Nat. Phys.* **6**, 855 (2010); Regarding Confinement Resonances, *Phys. Rev. Lett.* **105**, 213001 (2010); Platinum Nanoclusters Out-Perform Single Crystals, *Science* **327**, 5967 (2010); Probing Strain-Induced Changes in Electronic Structure with XMCD, *Phys. Rev. Lett.* **105**, 067405 (2010); and Site-Selective Ionization in Nanoclusters Affects Subsequent Fragmentation, *Phys. Rev. A* **81**, 021201(R) (2010). These highlights are posted online at www-als.lbl.gov/index.php/science-highlights/science-highlights.html.

EXPERIMENTAL SYSTEMS GROUP (ESG): PROBING POLYMERS WITH X-RAY SCATTERING

by Howard Padmore and Cheng Wang

Understanding polymer morphology. Polymers pervade all areas of modern life, not just in the obvious things like plastics and packaging of all types, but in surprising applications such as microelectronics as well. Since the 1977 Nobel Prize–winning discovery by Alan Heeger, Alan G. MacDiarmid, and Hideki Shirakawa of high conductivity in oxidized and iodine-doped polyacetylene, there has been an explosion of research in polymers for all types of electrical applications, the most important perhaps being recent work in solar energy conversion. The aim of this type of research is to produce inexpensive solar cells in ways similar to the way paper is printed in large volume.

Current research is aimed at pushing power-conversion efficiencies as high as possible and overcoming the effects of environmental degradation. Efficiencies have already gone from less than 1% in 1995 to an impressive 8.13% in 2011 with PTB7, a semiconducting alternating copolymer. In organic solar-cell devices, electron donor and acceptor materials form an interpenetrating network that enables photon absorption, charge separation, and charge transport. Improving efficien-

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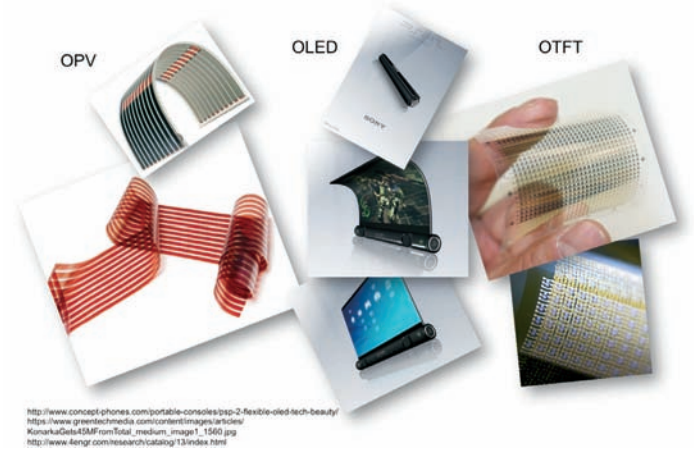


The promise of polymer-based electronics: flexible, lightweight, and inexpensive.

ESG: X-Ray Scattering *continued from page 5*

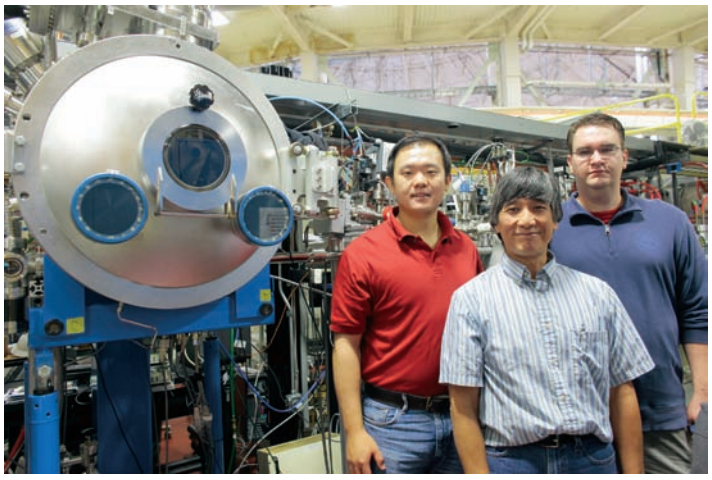
cies enough to realize the large-scale commercial potential of polymer solar cells (>10% efficiency) requires deeper understanding of and control over their molecular organization and nanoscale morphology.

Characterizing the three-dimensional chemical structure of organic blends with sub-10-nm resolution poses technical challenges. Various techniques such as scanning probe microscopy, electron microscopy, x-ray microscopy, neutron scattering, and x-ray scattering have been utilized to study these important morphologies. Amongst these, x-ray scattering is widely used as a high-resolution, nondestructive way to characterize polymer nanostructures. Because the scattering represents a statistical average over a large sample area, it is a great complement to direct-imaging techniques such as electron microscopy and scanning probe microscopy.

At the ALS, we have developed a suite of scattering tools, including conventional small- and wide-angle x-ray scattering (SAXS/WAXS), resonant soft x-ray scattering (RSoXS), and an upcoming coherent scattering and microscopy (COSMIC) facility, offering tools that will greatly enhance our existing scattering program for polymer science.

Small-and wide-angle x-ray scattering. At Beamline 7.3.3, SAXS/WAXS is utilized for a wide range of research on polymers. The beamline uses a bend-magnet source and operates at 10 keV. The x-ray flux is optimized by a multilayer monochromator and a toroidal focusing mirror producing 10^{11} photons/second with a resolving power (E/ΔE) of 100. Various sample environments are available (cooling and heating for both solids and liquids, in situ solvent annealing chamber, float cells, tensile stage) that allow studies of both static and dynamic samples and providing detailed, time-resolved information on spatial variations in electron densities. This enables the elucidation of phase separation and ordering processes in polymer systems during the ordering/disordering of components. Experiments are performed in transmission mode on bulk materials, while grazing-incidence scattering (GISAXS/GIWAXS) is used to investigate the characteristics of thin films (~100 nm). Simultaneous SAXS and WAXS measurements are extremely useful in characterizing atomic- and nano-sized correlations during, for example, the solidification of a polymer or the self-assembly of nanoparticles.

A recent major upgrade to the SAXS/WAXS beamline replaced our original CCD detectors with pixel detectors. These offer extremely high dynamic range, a great signal-to-noise ratio, and a fast frame rate (up to 300 Hz), orders of magnitude better than the old CCD detectors. With a more user-friendly interface and an automated experimental geometry setup, this upgrade is leading to high-throughput material discoveries that will set a new bar for x-ray scattering facilities. Furthermore, consistent with the ALS strategic plan, significant gains in performance could be achieved by moving the beamline from the current bend magnet to a more powerful superbend source, reducing exposure times by more than an order of magnitude.



ESG scientists Cheng Wang, Tony Young, and Alex Hexemer at Beamline 11.0.1.

Resonant soft x-ray scattering. While the hard x-ray SAXS/WAXS program described above probes bulk and near-surface structure, a new soft x-ray scattering program probes ultra-thin polymer films and surfaces with chemical specificity. This x-ray scattering approach, RSoXS, has been developed at the ALS to overcome the low electron-density contrast of organic thin films. RSoXS combines conventional x-ray scattering with x-ray spectroscopy, offering en-

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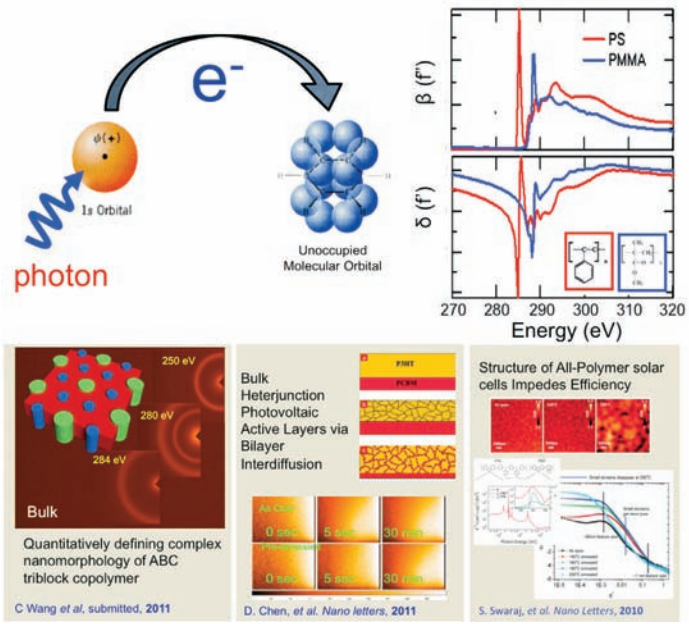


Top left: Basic principle of resonant absorption/scattering processes. Top right: Examples of the complex index of refraction near the carbon K-edge. Bottom: Recent applications of RSoXS on different polymer systems.

ESG: Probing Polymers *continued from page 6*

hanced scattering contrast as well as elemental and chemical sensitivity. It is ideally suited for studying the nanomorphology of organic photovoltaic devices.

A dedicated soft x-ray scattering endstation has been constructed at Beamline 11.0.1 and has begun general user operation. Sourced by a state-of-the-art elliptically polarizing undulator (EPU), the scattering chamber is designed to accommodate various scattering geometries with a customizable sample environment. The beamline began commissioning in early 2010, general user activities started in July 2010, and interest from various scientific communities involved in energy science, materials science, biology, etc. has been growing ever since.

Coherent Scattering and Microscopy. A further development of the RSoXS technique will be possible with the newly funded COSMIC project. COSMIC will have an energy range similar to Beamline 11.0.1 and will also have an EPU for polarization control. It will have two endstations, one for time-resolved coherent scattering (speckle) and one for a combination of coherent imaging, scattering, and spectroscopy. The basic instrument will have a zone plate to condense the beam to a 300-nm spot size and a CCD to detect the diffraction pattern. It will therefore be possible to do RSoXS in reduced areas, picking out individual features such as phase-separated domains. It will also be possible to phase the diffraction pattern using ptychographic techniques, to extend to sub-10-nm resolution if needed, preserving critical chemical information obtained from spectroscopy.

The ALS now has a useful toolbox of scattering techniques for polymer science and has enjoyed great success in their application to critical research areas, establishing and expanding Berkeley Lab's leadership in this area. With the development of a software package that combines soft x-ray spectroscopy and x-ray scattering analysis, scientific questions about the chemical morphology of organic/inorganic materials and devices will be addressed through systematic, collaborative studies of materials with potentially high-impact applications. The generality, strength, and ease of the x-ray scattering approach will have significant and immediate impacts in many areas of energy science and technology.

UNIQUE CELL-IMAGING CAPABILITY AND HIGH THROUGHPUT AT NCXT

by Gerry McDermott

The National Center for X-Ray Tomography (NCXT) soft x-ray microscope, Beamline 2.1, is now completing its second year of operation. During the past year, groups from around the globe have used the microscope to image a dizzying assortment of cells that ranged from simple bacteria, through fungi and algae, to highly complex stem cells and other mammalian cells. All of these projects have benefitted from having access to a unique combination of imaging technologies. In tandem with soft x-ray tomography, NCXT staff and collaborators have also been developing high-numerical-aperture light microscopy. This gives users of the facility the option to image the same specimen using multiple imaging modalities—in particular, cryogenic fluorescence microscopy to determine the location of specific molecules and soft x-ray tomography to visualize the subcellular architecture. Data from different techniques are combined to produce a highly informative composite view of the cell and its contents.

Throughput is one of the most important attributes in any biological imaging technique. Living cells vary significantly, even if they are considered to be of "the same" type. Therefore, it is enormously important that specimens can be imaged quickly. In a recent paper, the Drubin group (UC Berkeley), in collaboration with NCXT staff, used Beamline 2.1 to image and quantify morphological changes that take place in yeast cells as they progress through the cell cycle. Completing this work required the team to visualize hundreds of cells. This unprecedented achievement is not possible using any other imaging modality. As a result of their efforts, the team discovered that cells maintain critical volumetric ratios that are invariant of cell type, phenotype, genetic makeup, or cell-cycle stage. This finding is fundamental. It establishes metrics for differentiating between cells that are functioning normally and abnormally. Being able to make this distinction is essential not just in basic research, but in applied fields such as the search for effective new pharmaceuticals and the development of cheap, plentiful biofuels.

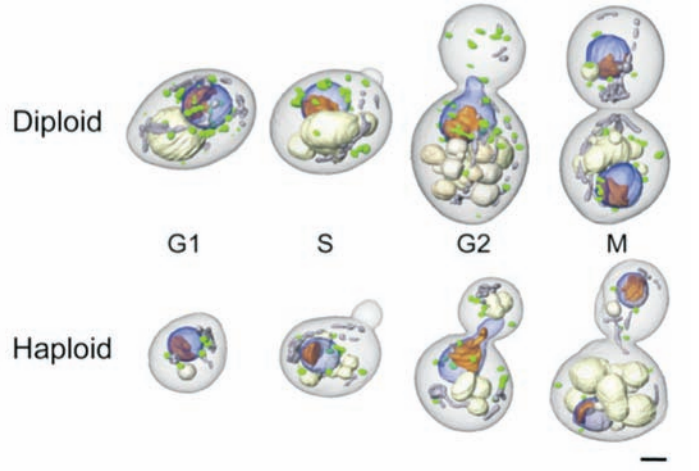


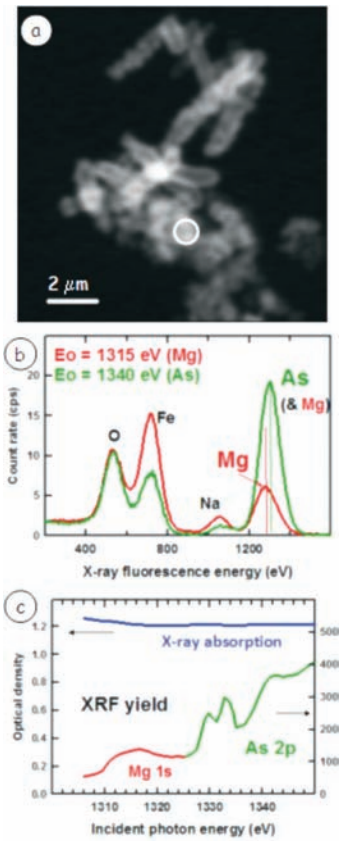
Figure taken from Uchida et al., *Yeast* **28**, 227 (2011), showing segmented tomographic reconstructions of haploid and diploid yeast cells at the four key stages of the cell cycle. Scale bar = 1 μm.

MOLECULAR ENVIRONMENTAL SCIENCE AT BEAMLINE 11.0.2

by Hendrik Bluhm, Mary K. Gilles, Tolek Tyliszczak, and David K. Shuh

Beamline 11.0.2 continues to be productive scientifically while technical advancements for existing and new endstations proceed. Scanning transmission x-ray microscopy (STXM) experiments here look at soils, catalysts, star dust, actinides, energy, atmospheric aerosols, and the magnetic properties of materials. Experiments at the ambient-pressure photoemission spectroscopy (APPES) endstation focus on environmental science, catalysis, and energy science.

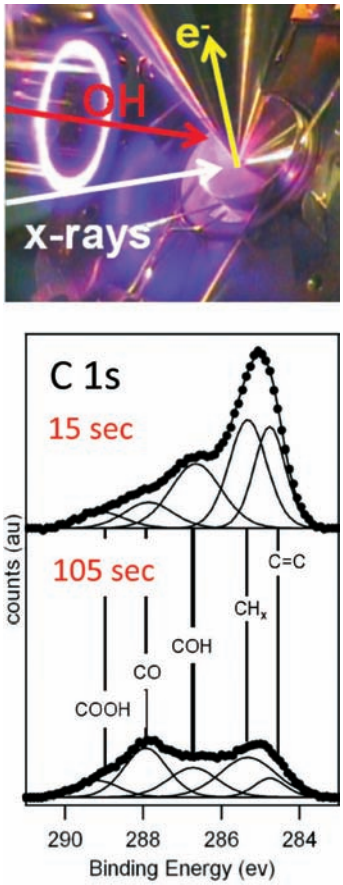
Mapping arsenic in bacteria using x-ray fluorescence (XRF). STXM has excellent spatial resolution and speciation capabilities but can be limited in sensitivity for minority species. By placing the detector extremely close to the sample, we can significantly increase the solid angle and sensitivity, thus enabling, for the first time, practical XRF-yield spectromicroscopy in a low-energy STXM. Using this capability, researchers [A.P. Hitchcock et al., *Microsc. Microanal.* **16**, 924 (2010)] investigated the arsenic distributions in cell-mineral aggregates formed by anaerobic, nitrate-reducing, iron-oxidizing proteobacteria isolated from the sediments of Lake Constance in Germany and cultured in the presence of arsenic [C. Hohmann et al., *Environ. Sci. Technol.* **44**, 94 (2010); A. Kappler et al., *Geobiology* **3**, 235 (2005)]. XRF spectra at 1315 eV (magnesium) and 1340 eV (arsenic) as well as x-ray absorption data



(a) STXM image of the sample showing a number of bacteria and associated biomineral deposits. (b) XRF spectra recorded from circled hot spot in sample. (c) X-ray absorption signal (blue) from hot spot area extracted from an image sequence (1308–1356 eV) compared to XRF-yield signal.

(1300–1360 eV) were measured at an arsenic-rich hot spot. The results clearly demonstrated that x-ray fluorescence is significantly more sensitive to arsenic (and magnesium) than x-ray absorption. Such a capability could be a valuable tool for investigations of anaerobic iron-oxidizing bacteria with the potential to significantly influence the environmental behavior of arsenic. Given that more than 100 million individuals worldwide are exposed to arsenic-contaminated water, the investigation of arsenic mobility in aquatic systems is of utmost importance.

Oxidation of PAHs by hydroxyl radicals. Organic compounds are known to constitute a significant fraction of anthropogenic pollution, but substantial uncertainty remains as to how these pollutants are transformed once they are released into the biosphere. To learn more about these transformations, researchers [E.R. Mysak et al., *Phys. Chem. Chem. Phys.* **13**, 7554 (2011)] have used APPES to monitor the oxidation and volatilization of a thin film of coronene ($C_{24}H_{12}$), a polycyclic aromatic hydrocarbon (PAH), with both ozone and hydroxyl radicals. Coronene monolayers were vapor deposited onto a silver substrate. By measuring the attenuation of the Ag signal as well as the C and O 1s peaks of the film, the thickness and composition of the film was obtained as a function of exposure to ozone and hydroxyl radicals. Detailed elemental and functional-group analysis of the spectra revealed that there is a competition between the addition of oxygenated functional groups (functionalization) and the loss of material (volatilization) to the gas phase. When the oxygen-to-carbon ratio (O/C) of the film is small (~0.1), the addition of functional groups dominates changes in film thickness, while for more oxygenated films (O/C > 0.3), carbon loss is an increasingly important reaction pathway. Decomposition of the film occurs via the loss of both carbon and oxygen atoms when the O/C ratio of the film exceeds 0.5. The results imply that chemically reduced hydrocarbons, such as primary organic aerosols, age in the atmosphere by forming new oxygenated functional groups, in contrast to oxygenated secondary organic aerosols, which decompose by heterogeneous loss of carbon and/or oxygen.



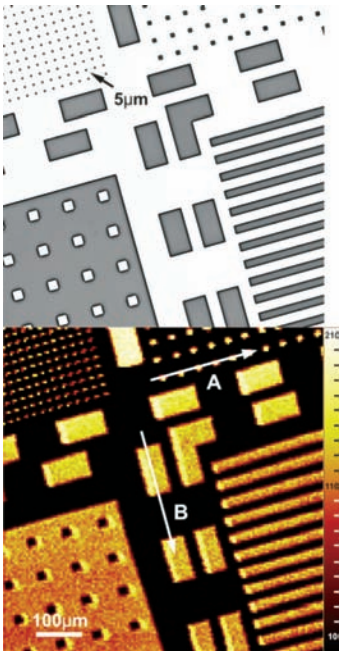
Top: Coronene/silver sample during reaction with hydroxyl radicals inside the APPES chamber at Beamline 11.0.2. Bottom: C 1s spectra as a function of time in the reaction between coronene and hydroxyl radicals.

STUDIES WITH TUNABLE VUV AT THE CHEMICAL DYNAMICS BEAMLINE 9.0.2

by Musa Ahmed

Vacuum ultraviolet (VUV) post-ionization. Biological systems are now seen as possible sources of alternative energy (plant-based lignocellulosic material) and environmental bioremediation (bacteria). Interestingly, the molecule types found on the surfaces of these biological systems, such as fatty acids, long-chain linear and branched hydrocarbons, and a variety of oxidized hydrocarbon species, are also thought to be present on the surfaces of tropospheric aerosols and in soil organic matter. It would be beneficial to have a set of tools for analyzing these surface molecules while maintaining information about their spatial distribution with respect to underlying biological structures. A convenient method for obtaining molecular information with high spatial resolution is imaging mass spectrometry, in which the molecules are laser-desorbed from the surface and ionized for subsequent mass spectrometry. However, the internal energy deposited within molecules upon desorption from a surface into the gas phase can lead to increased fragmentation, complicating analysis.

At the Chemical Dynamics Beamline 9.0.2, it has been shown that the internal energy gained during laser desorption leads to minimal fragmentation in a number of organic molecules (DNA bases) if ionization is decoupled from desorption, and this "post-ionization" is done using tunable VUV radiation. Using this method, 5-µm features in an organic polymer were observed using a 30-µm laser spot size and 7-ns duration [O. Kostko et al., *Chem. Asian J.* (in press)]. Recently, it has also been used to study the effect of antibiotics on bacterial biofilms [G.L. Gasper et al., *Anal. Chem.* **82**, 7472 (2010)], and it is anticipated that chemical composition and change within real-world environmental samples can be visualized with molecular specificity. Indeed, molecular information from bacterial biofilms, cellulose, lignin, melanin, soil extracts, and fossil feathers have been realized at the beamline in the last few months.



Photoresist features on a silicon wafer imaged using an optical microscope (top) and laser-desorption VUV post-ionization (bottom).

A "chemical reactor" for studying the formation of PAHs. To understand the formation of polycyclic aromatic hydrocarbons (PAHs) from the "bottom up" in combustion flames and in carbon-rich circumstellar envelopes in space, the energetics and dynamics of the reactions of aromatic radicals such as the phenyl radical (C_6H_5) are of paramount importance. Due to their inherent thermodynamic stability, aromatic radicals can reach high concentrations in flames; this makes them important reactants in the formation of PAHs and carbonaceous nanostructures. Recently, researchers using Beamline 9.0.2 obtained the first evidence that the PAH, indene (C_9H_8), together with its acyclic isomers, can be formed via "directed synthesis" through the reaction of the phenyl radical with propyne and allene.

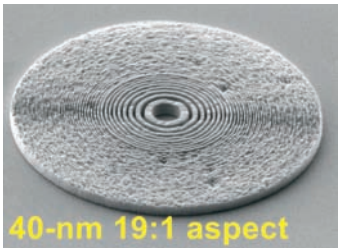
The centerpiece of the experiment was a resistively heated high-temperature "chemical reactor" incorporated into the beamline that allows the simulation of combustion-relevant chemical reactions and conditions (temperatures, pressures). Utilizing the reactor, the researchers synthesized the indene isomers via the reaction of pyrolytically generated phenyl radicals (C_6H_5) with two C_3H_4 isomers—allene (H_2CCCH_2) and propyne (CH_3CCH)—followed by isomerization and/or fragmentation of the initial C_9H_9 collision complexes. For distinct structural isomers, the adiabatic ionization energy and the corresponding photoionization efficiency curves can differ dramatically. By measuring these curves via tunable VUV radiation, individual indene isomers present in the supersonic beam were identified and their branching ratios established [F. Zhang et al., *J. Phys. Chem. Lett.* **2**, 1031 (2011)].

CXRO: X-RAY OPTICS AND BEYOND

by Patrick Naulleau

Although the Center for X-Ray Optics (CXRO) is part of the Materials Sciences Division (MSD), the Center has a tight and multifaceted relationship with the ALS. We develop beamlines and instruments, serve as beamline scientists, provide nanofabrication and coatings expertise, and are major users and collaborators.

We have expanded the scope of our nanofabrication activities beyond soft x-rays, applying the same double-patterning technology that enabled us to fabricate world-leading 10-nm-resolution zone plates (see feature story, "Exquisite X-Ray Optics") to the fabrication of high-aspect-ratio zone plates applicable to hard x-ray use. We have also developed a new process for the fabrication of phase-shift diffractive optical elements for the extreme ultraviolet (EUV) re-



Zone plate with 40-nm outer zone width and an aspect ratio of 19:1, produced using double-patterning methods.

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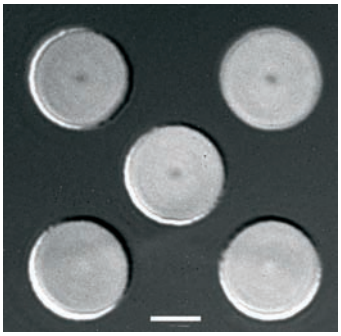
CXRO *continued from page 9*

gime. This process enables a threefold increase in efficiency compared to conventional amplitude structures. In the area of optics coatings, CXRO has continued making progress in the development of specialized aperiodic multilayer structures. Most recently, a narrow-band EUV multilayer with 25% reflectivity has been produced, enabling the use of compact high-harmonic-generation sources with diffractive optics.

The full-field soft x-ray microscope, XM-1, at Beamline 6.1.2 is a unique analytical tool for nanoscience research, combining excellent spatial and temporal resolution (70 ps) with a rich set of contrast mechanisms: elemental, chemical, topological, and magnetic. As part of the magnetic materials program in MSD, nanomagnetism research here focuses on a basic understanding of magnetic properties at fundamental length and time scales. One of our most exciting recent achievements was time-resolved microscopy of coupled vortex dynamics. This work represents an important step toward the realization of ultralow-power-consumption memory devices based on the physics of magnetic vortex structures. In addition, XM-1 also continues to support a vibrant ALS user program including activities beyond nanomagnetism. On the instrument side, the past year has seen the completion of a critical upgrade to our micro-zone-plate (MZP) manipulation stage. The new hexapod provides enhanced control over positioning and alignment, improving imaging performance and stability.



High-resolution soft x-ray image of a magnetic vortex core in the center of a permalloy disk. Watching these cores in time-resolved mode allows the interaction of multiple vortices to be studied.

CXRO's EUV lithography research tools provide the semiconductor industry with imaging capabilities generations ahead of their current processes. The SEMATECH Berkeley Micro Exposure Tool (MET) on Beamline 12.0.1 provides the world's highest-resolution EUV printing capability for evaluating photoresist materials and for mask research. The MET drives resist innovations in the areas of resolution, sensitivity, and line-edge roughness, with hundreds of new resist formulations being tested every year. At neighboring Beamline 11.3.2, the SEMATECH Berkeley Actinic Inspection Tool (AIT) is an EUV zone-plate-based full-field microscope dedicated to reflective photomask research. As the highest-performing tool of its kind, the AIT serves researchers from the leading semiconductor companies, providing fundamental learning not available anywhere else. Defects on EUV masks are one of the most crucial challenges facing the industry. Understanding how these defects are reproduced from the mask to the wafer is one of the most important activities on the AIT. This past year has also brought about an expansion of the EUV program in the form of a large enabling-technology development program for Samsung.

STRUCTURAL BIOLOGY: ONE THOUSAND AND COUNTING

by Paul Adams and Corie Ralston

The Howard Hughes Medical Institute beamlines at the ALS recently celebrated a milestone: their 1000th Protein Databank structure deposition. The winning protein structure is an endonuclease (PDB ID 3SIS), a protein important in protection against viruses because it is able to cleave foreign DNA. This is just one of many important macromolecular structures solved at ALS crystallography beamlines over the past decade. Solved structures extend from ion-channel proteins to receptors to nucleic-acid-binding proteins, illuminating steps in a diverse range of biological processes from cancer pathways to DNA decoding to prion disease and aging.

The steady increase in functionality and brightness of ALS beamlines over the past few years has contributed greatly to their popularity and use. Beamline 8.2.1, for instance, recently received an extensive x-ray optics overhaul, with upgraded focusing mirrors and a new liquid-nitrogen-cooled monochromator, now in the final commissioning stage. The new monochromator housing also includes a multilayer mirror that generates a tenfold boost in flux for specific experiments that do not require the higher energy resolution of the standard double-crystal monochromator. Software is being developed to allow for rapid transitions between the multilayer and the double-crystal monochromator, increasing the versatility of the beamline.

Several recent endstation improvements have also greatly increased the functionality of the beamlines. A variable-size collimator has been developed in-house and installed on most of the structural biology beamlines. The collimator size is adjustable from 10 to 200 microns and is user-controlled through a simple interface integrated into the standard beamline operating software. A larger robot dewar has been designed and installed on Beamline 5.0.1, doubling the current sample capacity. This allows for a con-

The 1000th protein structure deposited into the Protein Databank by the ALS's Howard Hughes Medical Institute beamlines.

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RING AROUND THE RING AROUND THE RING AROUND THE RING **AROUND THE RING**

Exquisite X-Ray Optics *continued from page 1*

tion ranges from 0.5 to 1 times the outermost zone width depending upon the illumination characteristics.

The Center for X-Ray Optics (CXRO) is a world leader in fabricating soft x-ray Fresnel lenses using advanced nanofabrication techniques. In nanofabrication, the biggest challenge is in the fabrication of features with extremely fine pitch. In general it is much easier to fabricate very small features with a larger pitch. Making use of this fact, CXRO has developed a double-patterning method that recently enabled the fabrication of Fresnel lenses with a 12-nm outer zone width.

The method works by first patterning every other zone such that, at the first step, a 12-nm zone with 48-nm spacing is produced. A second fabrication step is then used to insert the complementary zones, again having finest dimensions of approximately 12 nm with 48-nm spacing. This must all be achieved on thin membranes allowing for adequate x-ray transmission at the operational wavelength. Key to making this intricate process work is extremely precise alignment between the two fabrication steps. In general, the alignment is required to be a relatively small fraction of the outer zone width. Using unique pattern correlation techniques, CXRO has achieved alignment capabilities of 2 nm and better on their Nanowriter electron-beam lithography tool.

The ultimate proof of the fabrication and alignment capabilities of these Fresnel lenses is in imaging performance. The recently fabricated 12-nm Fresnel lens has been tested on the XM-1 full-field soft x-ray microscope at Beamline 6.1.2. Imaging resolution down to 10 nm has been obtained, consistent with expected performance given the partially coherent imaging conditions. A second lens optimized for scanning transmission x-ray microscopy (STXM) has also been fabricated. Because of other constraints on STXM lenses, current fabrication limits for these lenses is 17 nm. Using the latest CXRO-fabricated lens and test sample, the STXM microscope at ALS Beamline 11.0.2 has demonstrated imaging resolution down to 9 nm. The higher gain relative to the outer zone width compared to full-field imaging is a result of the scanning imaging process effectively being fully incoherent.

For x-ray spectroscopy, linear gratings are the optical components of interest. One way to improve performance is to employ high spectral order, as is done in ultrahigh-resolution optical spectroscopy. In the absence of optical aberrations, resolution is proportional to spectral order and line density; therefore, operating with high density and high order is a way to achieve the highest resolution. In the optical domain, spectral orders of a few 100 are often used. In the soft x-ray domain, however, we have always used first order as well as relatively modest line densities, in the mistaken belief that high spectral orders and high line density lead to low efficiency.

In fact, numerical simulations show that optimally designed gratings can diffract to very high efficiency, even in high orders, if the gratings are made with near atomic perfection. In collaboration with colleagues in CXRO and the Massachusetts Institute of Technology, ALS researchers have developed techniques for producing such gratings based on the anisotropic etching of silicon and multilayer deposition.

In this technique, a silicon wafer is etched in a hot KOH solution that acts anisotropically, etching a few hundred times slower along the <111> direction. The resulting exposed, sawtoothed facets are

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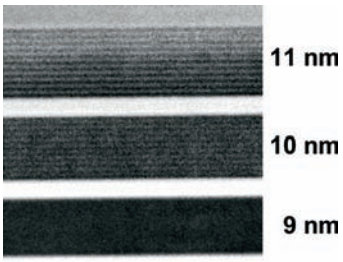
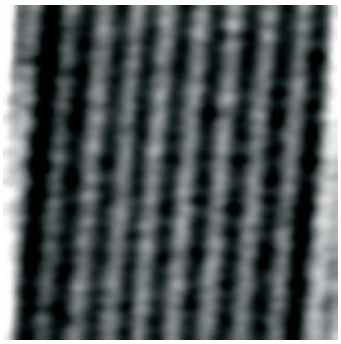


A CXRO zone-plate lens delivers 10-nm resolution as shown by full-field microscope images taken at Beamline 6.1.2.

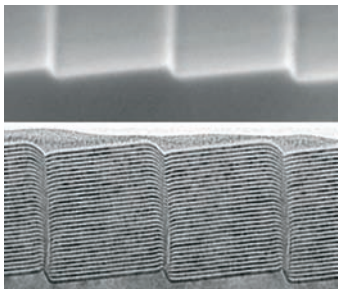


Scanned image of 10-nm half-pitch pattern at STXM Beamline 11.0.2.

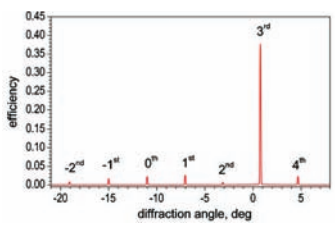
A Thousand Protein Structures *continued from page 10*

tinuous run time of up to 24 hours without the need to open the hutch. The new dewar design is scheduled for installation on the other Sector 5 beamlines within the next year. At Beamline 8.2.2, an upgraded CCD detector with faster readout and improved sensitivity is currently being installed. Two new data-collection modes, raster and vector collection, have been integrated into the beamline software, allowing analysis of very small or locally disordered protein crystals that might previously have been discarded.

All these improvements lead to faster and more reliable data collection and increase the feasibility of solving ever more complex structures. Thus the ALS crystallography beamlines are well positioned to solve another thousand structures in the years to come.



Top: SEM image of a 200-nm grating after KOH etch. Bottom: Cross-section TEM image of the grating coated with a Mo/Si multilayer.



Diffraction from a Mo/Si multilayer-coated grating measured at an incidence angle of 11° and a wavelength of 13.6 nm.

THE ALS COMMUNITY

HONORS AND AWARDS

Stephen Leone, director of Berkeley Lab's Chemical Sciences Division, professor of chemistry and physics at UC Berkeley, and director of the Chemical Dynamics Beamline 9.0.2, received the 2011 Irving Langmuir Prize in Chemical Physics from the American Physical Society "for his pioneering use of soft x-rays in probing ultrafast dynamics in atomic and molecular systems."

Gabor Samorjai, a senior scientist with Berkeley Lab's Materials Sciences Division (MSD), has won a "Frontiers of Knowledge Award" from Spain's BBVA Foundation. The Basic Sciences award recognizes Somorjai for "his pioneering contributions to the understanding of surface chemistry and catalysis at a microscopic and molecular level."

James Berger, of Berkeley Lab's Physical Biosciences Division and UC Berkeley, has won the 2011 National Academy of Sciences Molecular Biology Award for "elucidating the structures of topoisomerases and helicases and providing insights into the biochemical mechanisms that mediate the replication and transcription of DNA."

Ting Xu, a faculty scientist with MSD and UC Berkeley assistant professor of Chemistry and Materials Science and Engineering, was awarded the Camille Dreyfus Teacher-Scholar Award.

Chuck Fadley, also a faculty scientist with MSD and Distinguished Professor of Physics at UC Davis, was elected a Fellow of the American Association for the Advancement of Science.

Scott Mullin, a student of Nitash Balsara of UC Berkeley, was awarded the Padden award at the APS meeting in March 2011, for his contribution of "Electric Field Induced Ordering of Battery Electrolyte" using data from Beamline 7.3.3.

Several members of the ALS community have received the honor of being named APS Fellows for 2010, including **Harald Ade, Musahid Ahmed, Pupa Gilbert, Z.Q. Qiu,** and **Craig Taatjes.**

Falcone: Looking Forward

continued from page 1

tion. Our additional roles include the following:

- Communicating to different communities what we do, why it is unique, and why it is important. This is why we ask our users and staff to work with the ALS Communications Group to articulate research highlights and make sure we know about all publications in a timely way. For example, these highlights are sent to Washington and the stories are posted on Web sites and other social media that present the work of the DOE Office of Science, promoting our contributions and helping secure the investment that the nation is making in us.
- Training the science and technology workforce. At the ALS, we invest several percent of our budget in student training, from hosting high school students in the summer, to engaging undergraduate, post-baccalaureate, and graduate students in research, to mentoring postdoctoral students. We partner with users to leverage resources and mentoring by our staff, and we ensure that the nature of student training is relevant to DOE missions.
- Responding to the needs of industry and driving economic growth, jobs, and societal benefits. While our mission-driven work, as one of the DOE's Office of Science user facilities, is dominantly basic science, we also have an important role in

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Graduate students, such as ALS doctoral fellow Lynelle Takahashi (Chemical Dynamics Beamline 9.0.2), find many opportunities to talk about their research and network with other scientists at the ALS.

USERS' EXECUTIVE COMMITTEE UPDATE

by David L. Osborn,
2011 UEC Chair

It is my pleasure to serve the users of the ALS as chair of the Users' Executive Committee (UEC) in 2011. The ALS continues to serve a growing community of users, increasing by ~10% in the past year. The combined research of users and beamline staff has opened new doors in fields as diverse as battery degradation, magnetic refrigeration, and flame structure, to name just a few areas. In a year of highly uncertain funding for Department of Energy user facilities, ALS management and staff have continued to provide an efficient and safe environment for groundbreaking discoveries.

The 11 members of the UEC represent the interests of users to both the Department of Energy and ALS management. The ALS management turns to us whenever decisions that impact users are being considered. We work closely with them to ensure that the needs and perspectives of users are always at the forefront. Whenever user issues arise, please feel free to contact me or the other UEC representatives.

At the core of all ALS experiments lie the photons that this 20-year-old accelerator produces. Aging equipment gave the Accelerator Operations and Development Group an uphill battle to continue delivering light to users. Despite significant challenges, their careful planning and execution has led to exceptionally reliable operation, with a record of 250 hours of light delivered without a fault in May of 2011. On behalf of all users, I'd like to express our appreciation to David Robin and his staff for their achievements this year.

The User Services Building (USB) is now fully operational, providing major improvements in experiment staging and assembly, new staff offices, user drop-in cubicles, lockers for user storage, increased meeting space, and additional parking. I encourage all users to learn more about how this new asset can leverage your scientific productivity at the ALS.

Every year, the planning and execution of the ALS Users' Meeting is a major activity of the UEC. This year, program co-chairs Jeff Kortright and Gyorgy Snell, together with Sue Bailey, Deborah Smith, and their team at the ALS User Office, have organized an exciting meeting. We hope you can join us October 3–5, 2010, to experience the accomplishments of the past year and contribute to new research directions though the many topical workshops.

David L. Osborn

As we strive to solve the scientific needs of our nation and world, I wish you the best for a productive year of research.

The MERLIN beamline (4.0.3) attracted visitors all day.

2010 OPEN HOUSE

by Elizabeth Moxon

The Advanced Light Source once again welcomed the public to tour the facilty as part of Berkeley Lab's Open House in October 2010. Early Saturday morning, cheerful ALS volunteers donned tie-dyed yellow shirts and spread themselves around the experiment floor, ready to welcome visitors and answer their questions as they walked around the ring. What started out as a brief trickle of visitors turned quickly into an unending torrent of enthusiastic, scientifically curious guests who read every poster and took turns peppering staff with questions that covered all aspects of synchrotron science, from how an accelerator works to the results of experiments. Many were astonished at the sheer size of the ALS and at the fact that scientists from many disciplines come from around

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Sue Bailey of the ALS User Office points out the uses of macromolecular crystallography.

THE ALS COMMUNITY

ALS VISITORS COVER A WIDE SPECTRUM OF INTERESTS

The ALS remains a must-see stop for visitors to Berkeley Lab, with tour groups this past year representing a wide range of scientific, governmental, educational, business, and community interests. Following is a month-by-month sampling of the people who have walked through our doors in 2011.

January: President Ralph Eichler of the Eidgenössische Technische Hochschule (ETH) Zürich. ETH Zurich, founded in 1855, is renowned for its educational, technical, and basic research programs… Congressman George Miller, representing California's 7th Congressional District.

February: Staff members for Congresswoman Barbara Lee, representing California's 9th Congressional District.

March: Vivek Kundra, U.S. Chief Information Officer, and Bob Brese, Acting Deputy Chief Information Officer for the Department of Energy… Einstein Fellows—science and math teachers who share their expertise with Congress and other branches of the federal government.

April: Sebastian Klick and Mathias Kaufmann, twelfth graders at the Gymnasium St. Michael, a technical-sciences oriented high school in Bad Münstereifel, Germany, visited as guests of the Goethe Institute in San Francisco, part of their reward for their award-winning research projects… The steering committee for the East Bay Green Corridor—a collaboration between local communities to create a region of green technology and economic development.

May: Thirty visiting international MBA students at UC's Haas School of Business. The students, from the HEC Paris EMBA program, were participating in an executive program focusing on Corporate Social Responsibility, Global Citizenship and Sustainability at Haas, and were interested in Berkeley Lab's history of innovation and practical applications of scientific research in service to global needs.

June: Matthew Fedderson, 17, and Blake Marggraff, 18, from Acalanes High School were invited by ALS Director Roger Falcone to tour the ALS following their win at the 2011 Intel International Science and Engineering Fair with their research on treating simulated cancer cells with secondary radiation produced by Compton scattering… A group of 50 women from the Middle East and North Africa were brought to the ALS by TechWomen, an organization that pairs women in Silicon Valley with their counterparts in the Middle East and North Africa for a professional mentorship and exchange program at leading technology companies.

July: A group from Dow Chemical Company, led by Theresa Kotanchek, Vice President for Sustainable Technologies and Innovation Sourcing at Dow… California Public Utilities Commissioner Mike Florio.

TechWomen, a professional mentorship and exchange program that identifies 38 women who are emerging leaders in technical fields in Muslim communities, toured the ALS with Zahid Hussain (right) and Michael Banda (not pictured).

Paul Alivisatos, Michael Banda, Roger Falcone, and Senator Barbara Boxer discuss combustion research with Musa Ahmed at Beamline 9.0.2.

Florio was recently appointed commissioner by Governor Jerry Brown and is interested in the energy research capabilities of Berkeley Lab.

August: U.S. Senator Barbara Boxer of California… Congresswoman Zoe Lofgren, representing California's 16th Congressional District.

FACILITY UPDATES

ACCELERATOR AVAILABILITY AND RELIABILITY

by David Robin

The ALS operating schedule is divided into user beam shifts, accelerator physics/machine setup shifts, maintenance and installation, and vacation shutdown shifts. In a "typical year," there is usually one long shutdown for major servicing, large installations, and upgrades. This long shutdown and associated startup usually occurs in the spring and lasts about six weeks. Also, in a typical year the ALS schedules and delivers more than 5000 beam hours to users. Below is a summary of events during the previous three fiscal years and the availability and reliability of the facility.

Availability is one of the most important performance parameters of the facility. Availability is defined as the ratio of delivered versus scheduled user time. The Department of Energy goal for availability is >90% and the stretch goal is >95%. Maintaining a high availability as the facility becomes more mature and complex (e.g., the addition of new insertion devices, feedback and feed forward, injector upgrades and top-off injection) is a challenging task.

The number of hours scheduled each year was not uniform. In particular, there were nearly 6000 hours scheduled in FY10 because there was no major shutdown that year. Also, the availability was lower in FY08 mostly due to slower fill times in non-top-off operation. The explanation is as follows. At the end of FY07, the injector was upgraded to inject at full energy (1.9 GeV). This was in preparation for top-off operation, which began February 11, 2009. Prior to that, the ALS was still operating in decay (non-top-off) mode with three fills per day. In decay mode, we were allowing 18 minutes of fill time without penalty. However, just after the injector upgrade, the fill time was usually about 30 minutes, due to inconsistent fills resulting from problems with the newly installed controllers for the booster power supplies. This problem was subsequently resolved. Nevertheless, the additional fill time was designated as lost time and was responsible for about a 4% loss in time delivered to users.

Since February 11, 2009, the ALS has been in top-off operation. From that day through the end of FY10, the availability has been 96.8%. A further benefit of top-off operation is that there is no dead time between fills, so the total time available to users in every shift increased by roughly 5%. Also in FY10, an additional 136 hours (2.3%) of unscheduled light was delivered to users.

Reliability is another important performance parameter of the facility, which is distinct from availability. Providing good availability is necessary but not sufficient to ensure good reliability. For example, a user run with many unscheduled dropouts but fast recovery times would result in good availability but poor reliability. A good measure of reliability is the mean time between failures (MTBF). The MTBF was 31 hours in FY08 and increased to 37 hours in FY09 and 32 hours in FY10. These MTBF numbers are respectable; however, we would like to increase the MTBF to about 50 hours (less than 1 fault per 2 days).

Several of the systems that have been responsible for the larger fault numbers are being addressed, including faults coming from water clotting, power-supply failure, external power glitches, aging core controls, and the rf power system. We are already seeing the payoff: as of August 2011, the availability for FY11 is 97.5% and the MTBF is 42 hours.

Hours Scheduled and Delivered

	Scheduled hours	Delivered hours*	Availability	Mean time between failures (hours)
FY08	5087	4664	91.7%	31.2
FY09	5471	5278	96.5%	37.4
FY10	5980	5707	95.4%	32.4

*Does not include unscheduled hours delivered to users.

Open House *continued from page 13*

the world to work here.

As the enthusiasm and size of the crowds grew, more scientists, some running experiments that day, took time away from their beamlines to join in the event and talk about their research. At the end of the day, organizers estimated that more that 1200 people toured the ALS during the five hours that it was open. Some visitors stayed the whole time and were clearly disappointed when the doors closed. Guest reviews after the event were uniformly enthusiastic, many delighting not only in the fact that scientists were on hand to answer challenging questions, but that they were also able to see a working scientific facility where people were happy to showcase their work. Kudos to all ALS staff who made the event such a success!

David Hardy of Louisiana State University takes a moment away from his experiment on Beamline 10.0.1 to answer questions.

FACILITY UPDATES

USER DEMOGRAPHICS AND PUBLICATIONS

by Sue Bailey

As a national user facility, the ALS is required to report comprehensive user demographics and publication information annually to the U.S. Department of Energy. A sampling of that information is reproduced here. Figure 1 shows the overall user numbers in various scientific fields over time (FY07–FY10). Beginning in 2010, we began counting the growing numbers of remote users, primarily from the life sciences. These users save time, energy, and money by shipping their samples to the ALS and remotely operating the beamline to collect their data. Figure 2 shows the different categories of researchers at the ALS. Over half our researchers are young trainee scientists who will make key contributions to U.S. competitiveness in the future. Figure 3 shows the scientific productivity of the ALS as measured in refereed and high-impact journal articles since 1994. The designation "high-impact" includes journals with an impact factor greater than 7.2.

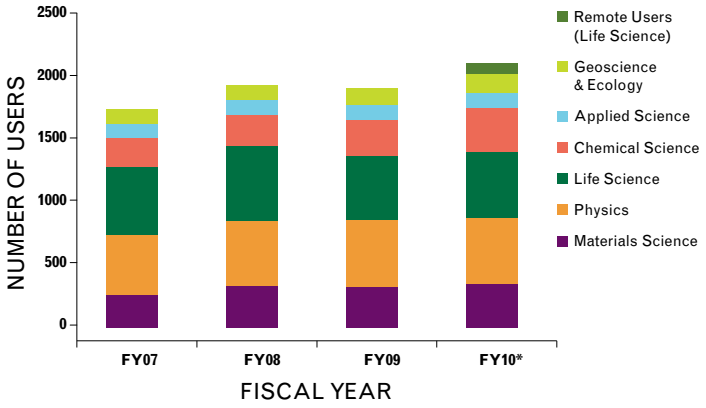

Figure 1. Bar graph showing the relative numbers of users in various areas of science.

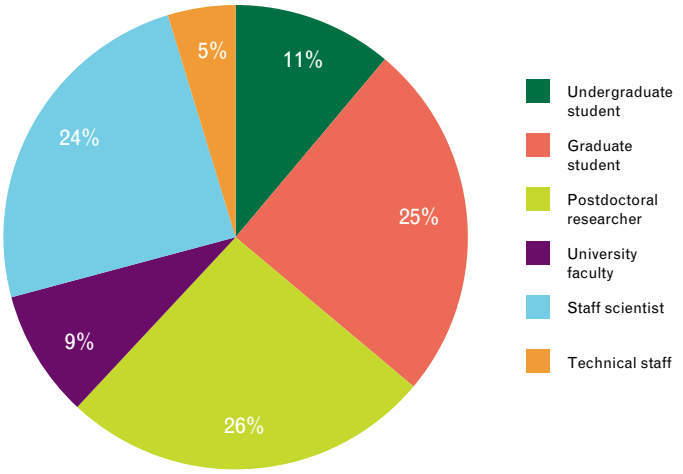

Figure 2. Pie chart showing the different categories of researchers at the ALS.

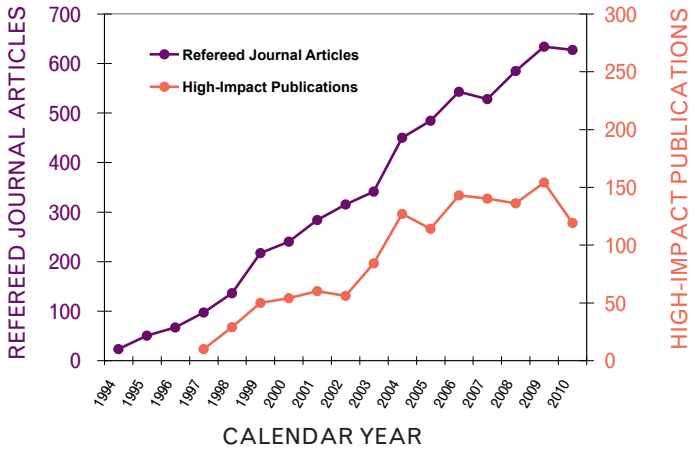

Figure 3. Graph of growth in refereed journal articles and high-impact publications.

Exquisite X-Ray Optics *continued from page 11*

extremely flat, with only single atomic steps over lengths of 200 nm. This atomically engineered reflecting substrate is almost perfect, a prerequisite for high reflectivity when multilayer coated.

Measurements of the diffraction efficiency of these gratings show that, as expected, all orders are suppressed except for the designed third order, where close to 40% efficiency was achieved at a wavelength of 13.6 nm. This result is equivalent to a grating with 15 times the line density of a conventional grating and perhaps 2 to 4 times the efficiency.

Future efforts will continue to find ways to increase the line density and spectral order. Presently, the researchers are working to extend the same techniques up to 1 keV or more, to cover the oxygen K-edge and the transition-metal 2p edges in particular. In 2012, tests in a real monochromator at Beamline 5.3.1 are being planned, possibly providing a basis for a new ultrahigh-resolution scattering facility at the ALS.

Publications about this work: D.L. Voronov et al., *Opt. Lett.* **35**, 2615 (2010); D.L. Voronov et al., *Opt. Express* **19**, 6320 (2011); and W. Chao et al., invited presentation, XRM 2010, Chicago, IL (August 2010).

FACILITY UPDATES

NEW ALS WEB SITE UNVEILED

by Elizabeth Moxon

The ALS Communications Group debuted a new ALS Web site this past year, built from the ground up. By interviewing users, staff, and management, we learned what people wanted to see, how they felt information should be prioritized, and what kind of navigation they considered the most efficient. The new pages were created using a peer-to-peer content management system that allows us to keep track of out-of-date stories while providing the ability to rapidly update the site with current information. Social media (Facebook, YouTube, and Twitter) have also been incorporated to further push ALS news and events to new audiences.

The site features a streamlined User Guide, which takes new and returning users through the proposal process from start to finish. The step-by-step process covers all aspects of becoming a user, including preparing a proposal, safety training and documentation, registration requirements, and housing and parking information. A new section, "Safety for Users," provides clear and succinct guidelines to prepare for and conduct experiments through its "How Do I?" series. Information about safety training, working with chemicals, laser authorization, personal protective equipment, and more is available here.

We continue to develop content for the site and are always looking for new ways to educate and inform our diverse audiences. In Fall 2011, we will debut "Beamline Highlights at the Advanced Light Source," a multimedia series of 90-second shorts featuring beamline scientists describing their research and the unique capabilities of their beamlines. And our science highlights will now include a multimedia "lay" version of the featured research, making it more accessible to a wider audience. Future projects include an interactive beamclock so that users can easily determine which beamline has the right capabilities for their experiments, an educational section for students and teachers, and a section on the history and future of light sources.

Redesigned home page of the ALS Web site.

What has really made the new site so successful is the input we have had from users and staff, and we continue to want to develop the site for all our audiences. Everyone is invited to send story ideas and recent science news and events to alscommunications@lbl.gov or drop by the ALS Communications Group offices in the User Support Building (Rooms 316–320).

FACILITY UPDATES

DOME GETS NEW ROOF

by Shauna Kanel

The 2011 ALS summer shutdown was quite productive. Highlights include renovation of the iconic ALS dome, installation of new equipment, and the performance of essential maintenance.

Asphalt shingles on the dome's facade were replaced with new "cool roof" technology. It took workers more than five weeks to reroof the 20,000 sq ft surface with Owens Corning Duration Premium Cool Shingles, which have a 25% solar reflectance and can withstand wind speeds up to 130 mph. Inside the dome, 30 feet of scaffolding was erected on top of the rotating crane, and a plastic sheet was put in place to catch falling debris while workers removed lead paint from 30% of the dome's interior. A "leadlock" coating was then applied to protect occupants from exposure to the remaining lead paint.

Crews work hard to put a "cool roof" on the ALS dome.

A wide range of work was done to restore and improve the ALS's electronic components, storage ring, power sources, beamlines, and more. The old sextupole and QFA power supplies were replaced with new, more reliable power sources. The new QFA will be less sensitive to line voltage sags that cause beam dumps.

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Falcone: Looking Forward *continued from page 12*

responding to the more near-term needs of industry. Two examples of this are in health (pharmaceutical companies are using the ALS to discover new drugs and understand the molecular basis of disease through structural biology) and electronics (the semiconductor industry is using the ALS to develop the next generation of chips so that they can sustain Moore's Law of increasingly powerful microprocessors); there are many other examples.

So how are we doing? Well, in March the DOE brought in a team of external scientists for the triennial review of the ALS. The results were very encouraging. Reviewers were extremely pleased by the culture of safety that not only keeps us working safely but is welcomed by staff and users as helping us all to work better. The report included some key phrases: "first-rate and trend-setting science," "state-of-the-art instruments," and "a reputation for excellence." Also included in the report were suggestions to make us even better, including consideration of a shorter proposal cycle and a more integrated Web-based system for handling the cycle of proposals, experiments, and publications; we are working on both these ideas. Echoing the review committee, I greatly appreciate the efforts of our entire community in making the ALS an outstanding facility, now and into the future.

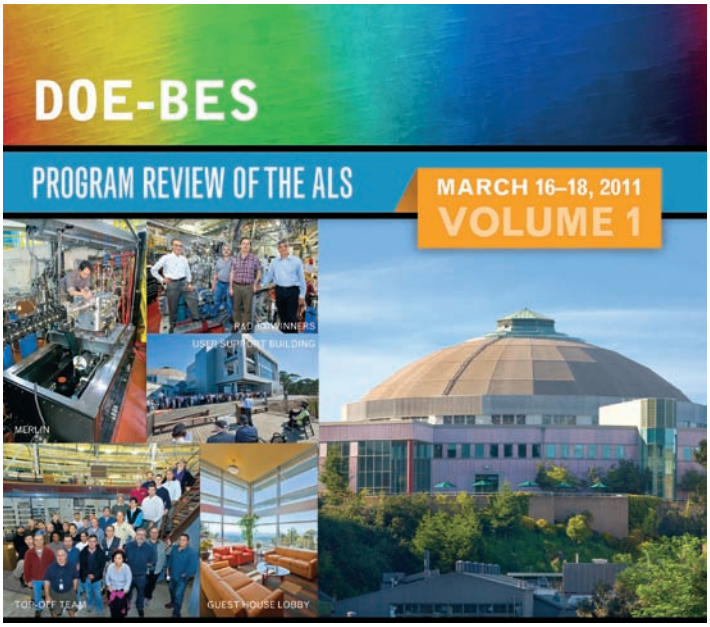
What else are we doing to maintain excellence? The ALS has a strategic plan, developed over time with users and staff. This plan was formally initiated in 2009 and jump started by federal stimulus funds. We used those funds to secure critically needed equipment and to develop new capabilities—mainly through purchases from companies—that will lead to an even more productive and effective facility, stimulate technological discoveries, and lead to further innovation.

Long-term, our strategic plan is aimed at keeping ALS brightness at the top of soft x-ray facilities globally, replacing aging and increasingly unreliable components of the machine, building new beamlines and instruments with greater capacity and capability, and increasing user support. It is a challenge to find the funding to accomplish our renewal plan, but we are prioritizing our requests to highlight the most urgent and relevant work (for example, new beamlines will serve the DOE's new Energy Hub focused on enabling artificial photosynthesis). Also, we seek cost-effective upgrades that will allow us to serve more users (for example, chicaning ALS's straight sections to allow two undulator sources, beamlines, and instruments, each with unique capabilities, where there was only one set before).

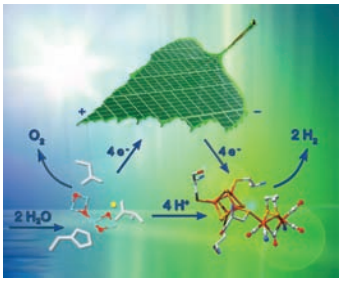
While budget challenges in Washington have recently slowed hoped-for growth in federal funding for research (remember the doubling plan?), our strategic plan is still being supported. Development of the MAESTRO and COSMIC beamlines is now underway in Sector 7. MAESTRO will enable, through angle-resolved photoemission at the nanoscale, the understanding of materials whose performance is dominated by correlated electrons. COSMIC will enable the characterization of fundamental fluctuations of material properties in space and time through coherent scattering techniques at the nanoscale, as well as forefront diffractive microscopy.

There are many ways we can accomplish our broader goals, beyond directly requesting new funds from Washington. These include partnering with other divisions at Berkeley Lab, other national labs, and industrial users as well as engaging with DOE's new Energy Frontier Research Centers and Energy Hubs. Our goals in communicating ALS's value to the nation are also served by reaching out to our local community, for example at the Lab's Open House (on October 15), where the ALS is always the center of the action, with thousands of visitors making their way around the ring. We are also keeping ALS infrastructure in good shape (you might have noticed that we replaced our leaking 20-year-old roof over the dome with a new "cool roof").

Returning to our "metrics," while our user and publication numbers are as high as ever (and let me not forget to mention the beam reliability record recently set by our accelerator staff and operators), we must continue to evaluate performance of all aspects of the ALS and focus on moving from less productive activities that should be concluded to initiating critical new activities that will keep us at the forefront of science and better serve users. This will be a challenge for all of us—users, staff, and management—and as always, I look forward to hearing from you on how we can continue to improve.



Highlights of the past three years were provided to members of the DOE's Basic Energy Science (BES) review committee.



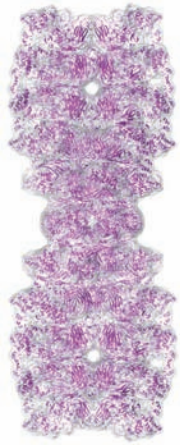
Fuel from sunlight: urgent and relevant topics such as artificial photosynthesis (the focus of a DOE Energy Hub) will help guide ALS strategic development.

Science Roundup *continued from page 3*

Giant Protease TPP II's Structure, Mechanism Uncovered

Chuang et al., *Nat. Struct. Mol. Biol.* **17**, 990 (2010)

Cholecystokinin (CCK) is a hormone in the brain and gastrointestinal system that helps stimulate the digestion of fat and protein and acts as a satiety agent, suppressing hunger and inhibiting food intake. Tripeptidyl peptidase II (TPP II) is known to partly regulate CCK-8 (a CCK with 8 amino-acid residues) by cleaving the hormone into 5- and 3-residue chains, inactivating it. To gain insight into TPP II's mechanisms of activation and proteolysis, researchers combined single-particle cryo-electron microscopy and x-ray crystallography at Beamline 8.2.2. Knowledge of the mechanism of action for this molecular machine would help researchers find TPP II inhibitors or CCK protective agents that might then be incorporated into obesity treatments.



Structure of the TPP II holocomplex. The protein "skeleton" of this giant protease is depicted in magenta. The gray enclosure represents a lower-resolution surface and is included to aid visualization of the complex.

Superconducting Topological Insulators

Wray et al., *Nat. Phys.* **6**, 855 (2010)

In 3D topological insulators (TIs), electrons near the surface move at high speeds in a "light-like" fashion. Quantum interactions cause individual electrons to be spin polarized even at room temperature and to strongly resist scattering from defects, naturally achieving some of the most desirable traits for computing components and next-generation "spintronics" technologies. Angle-resolved photoemission spectroscopy studies at Beamlines 10.0.1 and 12.0.1 have paved a way for these novel properties to be taken even further. The studies showed that by doping the TI, bismuth selenide, with copper, it's possible to make the topologically ordered electrons superconducting, drop-

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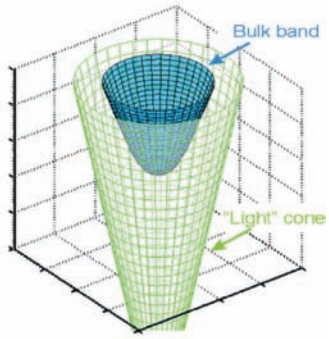


The energy vs. momentum relationship for electrons in topological insulators resembles either light (green mesh) or high-energy relativistic particles (blue mesh).

2011 Summer Shutdown *continued from page 17*

Both power-supply upgrade projects will aid in more reliable beam delivery, but are mainly to replace aging equipment and keep the ALS running smoothly.

A new Thales klystron was installed and major preparatory work was done so that during the next long shutdown it can replace the extant Phillips tube. Sector 6 began receiving major upgrades, including a new vacuum chamber and chicane magnet. These installations readied the downstream portion of the Sector 6 straight section for a new EPU, which will be installed over several two-day shutdowns in coming months. Other improvements include upgrading the low-conductivity water system to provide an additional 20% capacity.

Maintenance activities include the first full survey and alignment of the storage ring girders since top-off operation began in 2009. Additionally, the helium compressors on the ALS's superbend magnets were replaced. This involves warming the magnets from their sub-5K operating temperature, breaking the vacuum, swapping the compressors, reestablishing the vacuum, and cooling the magnets back down. It takes a minimum of 3 weeks and must be done every 18 months, making it a driver for an annual extended shutdown.

Crews remove the old sextupole power supply using a counter weight and a 4-ton crane.

A rare sight: this superbend magnet is open to receive a new helium compressor, or "cold head."

From a scaffolding set up on the radial crane, workers hang a large plastic sheet inside the dome to prevent stray pieces of hardware or lead paint chips from falling.

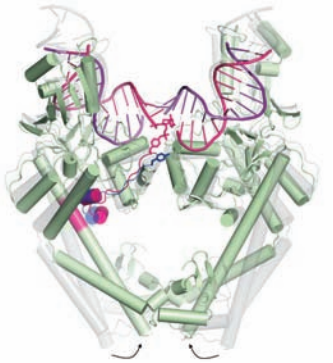
Science Roundup *continued from page 19*

ping electrical resistance in the surface states all the way to zero.

A Novel DNA Cleavage Mechanism

Schmidt et al., *Nature* **465**, 641 (2010)

Type II topoisomerases are molecular machines that regulate DNA supercoiling and separate interlocked chromosomes. These enzymes are also exploited clinically as targets of antibiotics and anticancer therapeutics. Researchers at Beamline 8.3.1 imaged type II topoisomerase's ordinarily short-lived state in which it is linked to a DNA's nucleic acid segment through its active-site tyrosine, cleaving the DNA. Details of this molecular model provide evidence for the chemical mechanism by which type II topoisomerases (topo IIs) and a related topo family (topo IA) accomplish DNA cleavage. The structure also reveals how the enzyme avoids dissociating when DNA is cleaved, preventing the aberrant formation of mutagenic genomic lesions.

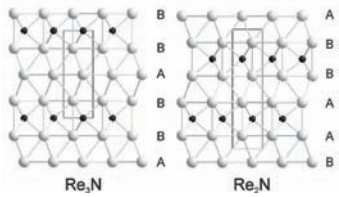


Cleavage of a DNA strand pulls on a linker element—like tugging on the drawstring of a latch—closing the dimer interface.

Two Novel Ultra-Incompressible Materials

Friedrich et al., *Phys. Rev. Lett.* **105**, 085504 (2010)

Some current challenges in aerospace engineering and fission/fusion applications require materials that are mechanically and chemically stable at extreme conditions. One such class of materials is ultrahigh-temperature ceramics, which are often binary transition-metal carbides, borides, or nitrides. It is therefore of great interest to understand how to synthesize new compounds of this type. A research team working at Beamlines 12.2.2 and 12.3.2 has now synthesized and characterized two novel bulk rhenium nitrides, Re_2N and Re_3N. Both phases are extremely incompressible, and Re_3N is better placed for potential applications than other incompressible transition-metal carbides and nitrides of the period-six elements because it can be formed at relatively moderate pressures and temperatures.



The incorporation of nitrogen (black) into the rhenium lattice at increasing pressure and temperature leads to the formation of Re_3N and Re_2N. The N atoms occupy interstitial sites between AA or BB layers only.

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ALS ADVANCED LIGHT SOURCE

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