ALS SPECTRUM

Advanced Light Source :: Facility Report :: 2012-2013

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What's Inside:

U.S. DEPARTMENT OF ENERGY
Office of Science

BERKELEY LAB

ALS ADVANCED LIGHT SOURCE

ALS COMMUNICATIONS

Lattice Upgrade Increases ALS Brightness

by Christoph Steier

A major upgrade of the ALS lattice was completed in 2013, increasing brightness by a factor of three at superbend and bend-magnet beamlines, and by at least a factor of two at insertion-device beamlines. The upgrade consisted mainly of the addition of 48 new sextupole magnets. With the completion of this upgrade, the horizontal emittance—a measure of beam spreading—was reduced from 6.3 to 2.0 nm (the smaller the emittance, the brighter the beam).

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On October 23, 2012, representatives of the ALS, Berkeley Lab, and the Shanghai Institute of Applied Physics celebrated the installation of the first of 48 new sextupole magnets into the ALS storage ring.

The Promise of Advanced Ceramic Composites

by Keri Troutman

Ceramic materials have been used for thousands of years as a basis for pottery, and today they have a myriad of other uses from biomedical implants to semiconductors. However, their structural use has been compromised by the fact that they are inherently brittle, making them a poor choice for certain high-heat applications, such as engines.

Berkeley Lab senior materials scientist Rob Ritchie, who also holds the H.T. & Jessie Chua Distinguished Professorship in UC Berkeley's Departments of Materials Science & Engineering and Mechanical

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Pseudo Single Bunch Expands Experimental Scope

by Keri Troutman

Initial tests of a new pseudo-single-bunch (PSB) operational mode at the ALS have shown promising results—PSB would expand the facility's capacity to carry out dynamics and time-of-flight experiments with a major reduction in sample damage. In PSB operation, a single electron bunch is displaced transversely from the other electron bunches circulating in the storage ring [C. Sun et al., *Phys. Rev. Lett.* **109,** 264801 (2012)].

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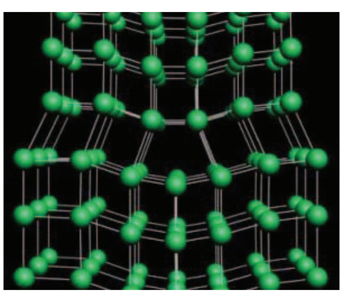
Science Roundup

Shedding Light on Nanocrystal Defects

B. Chen et al., *Science* **338**, 1448 (2012)

Nanocrystals have been the focus of much scientific interest lately. Their resistance to stress has had researchers proposing nanocrystals as a promising new protective coating for advanced gas turbine and jet engines. But recent studies at the ALS show that their tiny size does not safeguard them from defects. Researchers studied nanocrystals of nickel subjected to high pressure and found that dislocations—defects or irregularities—can form in the finest of nanocrystals when stress is applied. The results demonstrate that dislocation-mediated deformation persists to smaller crystal sizes than anticipated and that computer models used to predict nanocrystal behavior thus far have not given enough consideration to the effects of external stress and grain boundaries.

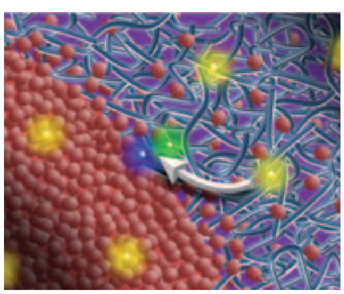


Stress-induced deformation of nanocrystalline nickel reflects the dislocation activity observed by researchers using a radial diamond-anvil-cell x-ray diffraction experimental station. (Image courtesy of NDT Education Resource Center).

A New Path to Higher Efficiency in Organic Solar Cells

B.A. Collins et al., *Adv. Energy Mater.* **3**, 65 (2013)

The efficiency of polymer/organic photovoltaic cells hinges on excitons—electron/hole pairs energized by sunlight—getting to the interfaces of donor and acceptor domains quickly, before recombining. At the interfaces, they become free charges that must then reach device electrodes. With the discovery of mixed domains of donor and acceptor molecules, many have pictured the excitons' journey as easy but the charges' journey as precarious. Instead, using a combination of x-ray scattering and microscopy techniques, researchers have found that excitons may actually not fare so well in mixed domains but need access to pure aggregates to efficiently convert into charges. The smaller the aggregates, the better, allowing increased interfacial area and dramatic increases in device performance.

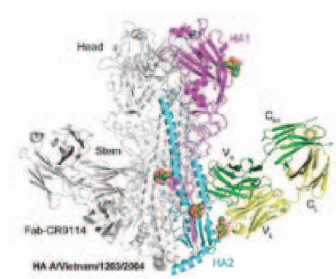


Molecular view of a polymer/organic solar film showing a donor/acceptor interface. Red dots are organic fullerene molecules and blue lines represent polymer chains. Excitons (yellow) need to reach the fullerene aggregates to be separated into electrons (purple) and holes (green).

Toward Design of a Universal Flu Vaccine

C. Dreyfus et al., *Science* **337**, 1343 (2012)

Worldwide, influenza causes substantial deaths and yearly economic burdens, but the highly changeable nature of the flu virus complicates the production of an effective vaccine. One factor in determining the vaccine's effectiveness is how closely related the viruses used in the vaccine are to the viruses circulating that year. An international team of researchers, working at synchrotron facilities including the ALS, has solved the crystal structures of antibodies that protect against broad classes of influenza strains. Greater understanding of these antibody structures may aid in the eventual development of a universal vaccine, protecting against all types of influenza viruses and thus eliminating the guesswork that currently limits vaccine effectiveness.

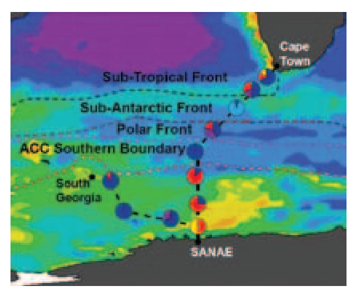


High-resolution crystal structure of the Fab (fragment antigen-binding) section of an antibody in complex with a key segment of the H5N1 virus.

Iron Availability in the Southern Ocean

B.P. von der Heyden et al., *Science* **338**, 6111 (2012)

The Southern Ocean, circling the Earth between Antarctica and the southernmost regions of Africa, South America, and Australia, is notorious for its high-nutrient, low-chlorophyll areas, which are rich in nutrients, but poor in essential iron. Sea life is less abundant in these regions because the growth of phytoplankton—the marine plants that form the base of the food chain—is suppressed. A STXM study done at ALS Beamline 11.0.2 suggests that it is not just a lack of iron, but a lack of iron in an easy-to-use form, that is affecting the ecosystems. The researchers sampled two north-south corridors across the Southern Ocean. Along the way they collected particles containing solid iron from a series of ocean systems with different characteristics.



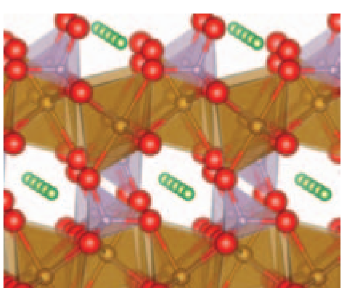
Map of the part of the Southern Ocean that was sampled, showing chlorophyll concentrations in milligrams per square meter. There is a rough tendency for soluble iron regions to show greater chlorophyll concentrations.

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

Two Studies Reveal Details of Lithium-Battery Function

X.S. Liu et al., *J. Am. Chem. Soc.* **134**, 13708 (2012); Y.-C. Lu et al., *Sci. Rep.* **2**, 715 (2012)

Our way of life is deeply intertwined with battery technologies that have enabled a mobile revolution. As conventional lithium-ion batteries approach their theoretical energy-storage limits, new technologies are emerging to address the long-term improvements needed for mobile systems, electric vehicles in particular. Battery performance depends on the dynamics of evolving electronic and chemical states that, despite advances in material synthesis and structural probes, remain elusive and largely unexplored. At the ALS, researchers studied lithium-ion and lithium-air batteries using soft x-ray spectroscopy techniques. Detailed information about the evolution of electronic and chemical states will be indispensable for optimizing better battery materials.

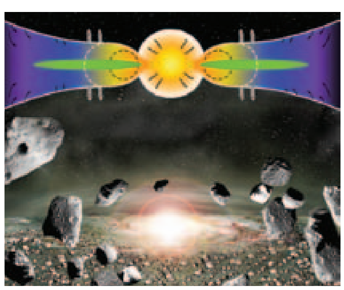


Crystal structure of $LiFePO_4$. Lithium ions (green) diffuse into and out of the olivine framework through a one-dimensional channel.

Studying the Solar System's Chemical Recipe

S. Chakraborty et al., *PNAS Early Ed.*, doi: 10.1073/pnas.1213150110 (2013)

To study the origins of different isotope ratios among the elements that make up today's smorgasbord of planets, moons, comets, asteroids, and interplanetary ice and dust, a team of scientists used Chemical Dynamics Beamline 9.0.2 to mimic radiation from the protosun when the solar system was forming. The researchers are using Beamline 9.0.2 to see if photochemistry can explain the differences in isotope ratios between elements on Earth and what's found in meteorites and interplanetary dust particles. The beamline generates intense beams of vacuum ultraviolet light that can be precisely tuned to mimic the radiation from the protosun. It is powerful enough to dissociate gas molecules like carbon monoxide, hydrogen sulfide, and nitrogen, providing information about gas-phase photodynamics.

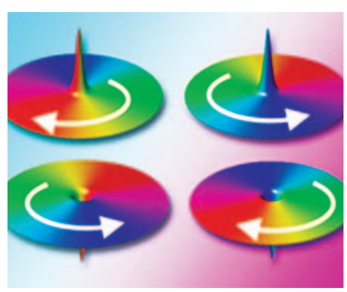


The protosun evolved in a hot nebula of infalling gas and dust. Visible and ultraviolet light poured from the sun, and numerous chemical reactions contributed to the isotopic ratios seen in relics of the early solar system today.

Reversing the Circulation of Magnetic Vortices

V. Uhlíř et al., *Nat. Nanotechnol.* **8**, 341 (2013)

In magnetic media, information is stored in binary form, one or zero, depending on which way the electronic spins are aligned in a given section of the medium. Recently, however, magnetic vortices have drawn scientists toward a new possibility: multibit storage in which each logic unit has four states instead of two and can store twice the information. Each tiny magnetic whirl has a polarity that can point up or down and a circulation that can be oriented clockwise or counterclockwise. Previous studies have shown that the polarity can be flipped on command. Now, using time-resolved magnetic soft x-ray microscopy at the ALS, researchers have shown for the first time how to use pulsed magnetic fields to reverse the circulation.



Magnetic vortex structures establish a 4-bit magnetic storage/logic unit, represented by two levels of polarity (up/down) and two levels of circulation (clockwise/counterclockwise).

Flipping Photoelectron Spins in Topological Insulators

C. Jozwiak et al., *Nat. Phys.* **9**, 293 (2013)

Inherently strange crystalline materials called 3D topological insulators are all the rage in materials science. This new phase of condensed matter is an insulator in the bulk but behaves like a metal on the surface, even at room temperature. The spin of a surface electron is locked to its momentum, perpendicular to the direction of travel. These electronic states already promise many uses, but researchers working at Beamline 4.0.3 have discovered that, when hit with a photon beam, the spin polarization of the electrons they emit can be completely controlled in three dimensions, simply by tuning the polarization of the incident light. This strong effect was not what had been assumed about photoemission from topological insulators, or any other material.

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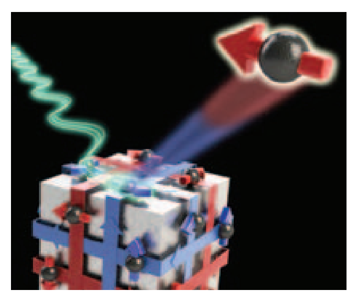

Researchers have discovered that the spin polarization of photoelectrons emitted when a topological insulator is struck with high-energy photons is completely determined by the polarization of the incident light.

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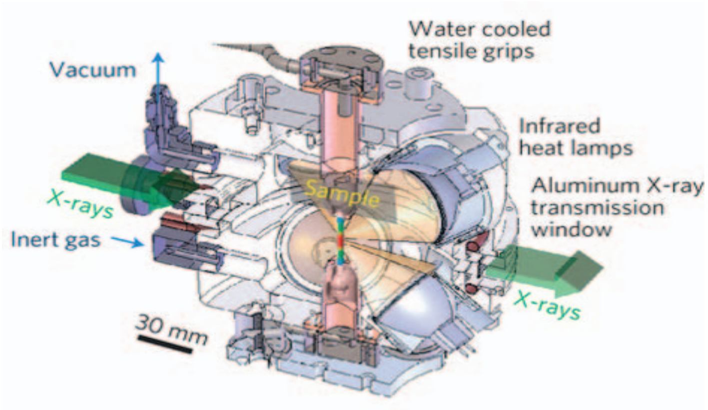
EXPERIMENTAL SYSTEMS GROUP: THREE TECHNIQUES FOR NEW MATERIAL INSIGHTS

by Howard Padmore and Alastair MacDowell

Synchrotron technology and associated techniques have constantly evolved and refined over the years. Here we focus on three techniques over three spatial ranges that are undergoing new developments leading to new insights into the broad range of materials typically studied at synchrotrons.



The tensile test rig used at Beamline 8.3.2 can pull or compress a small, cylindrical sample in ultrahigh-temperature environments (up to 2000 °C). The rig can also operate in vacuum or in atmospheres that are inert or oxidizing.

X-Ray Microtomography—New Environments. Similar to the 3D imaging capabilities of a CAT scan in a hospital, x-ray microtomography on Beamline 8.3.2 has become a widely used tool to image samples in 3D on the micron scale. Using the hard x-ray flux from an ALS superbend magnet, the high x-ray flux density allows for imaging small objects in only a few seconds of scan time. Thus, the strength of Beamline 8.3.2 is in performing time-resolved experiments.

In collaboration with beamline users, ALS staff developed a unique and novel chamber that is able to image a sample in 3D while the sample is under a load of up to 2000 newtons and at a temperature of up to 2000 °C. This opens up a new range of experiments on the behavior of materials under load and at high temperature (see also "The Promise of Advanced Ceramic Composites" on p. 1). The information obtained is in digital form and is used to validate computer models that predict the critical performance characteristics of the materials being developed. In this way, development cycles are accelerated and new materials are brought to market on a shorter time scale.

Other user groups are developing programs with this novel high-temperature tomographic cell to answer questions in difficult but realistic environments. NASA is studying the effect of atmospheric reentry on the degradation of heat shields to validate computer models of heat-shield defects and damage for incoming spacecraft. Another group is examining the cracking behavior of molten rock to simulate its behavior inside a volcano. The crack-propagation behavior of magma determines the explosive properties of volcanoes, which (in keeping with the aeronautical theme) can have important implications for air traffic control, as was evidenced by the closure of European airspace by the eruption of a volcano in Iceland back in the spring of 2010.

Future generations of spacecraft may benefit from high-temperature microtomography studies of advanced materials for use in fuselage and control-surface applications.

X-Ray Ptychography—Higher-Resolution X-Ray Microscopy. X-ray ptychography is an advanced imaging technique that takes full advantage of the soft x-ray brightness of the ALS and can extend the achievable spatial resolution well beyond the limitations of x-ray optics. The technique is similar to standard scanning

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ESG: Material Insights

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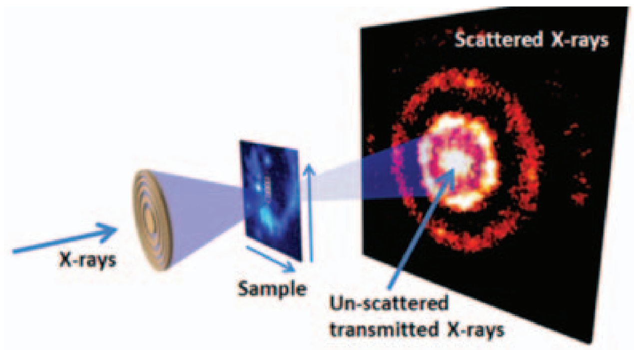
transmission x-ray microscopy (STXM), as the sample is also scanned through a focused x-ray beam. However, at each sample position, a full coherent diffraction pattern is measured rather than just the total transmitted intensity. These diffraction patterns are typically sampled on a 1000 x 1000 grid, so the data volume and rate increase by up to six orders of magnitude. This enormous increase in information can be exploited by state-of-the-art nonlinear optimization algorithms to calculate the sample structure at a spatial resolution limited, in principle, only by the illuminating wavelength.

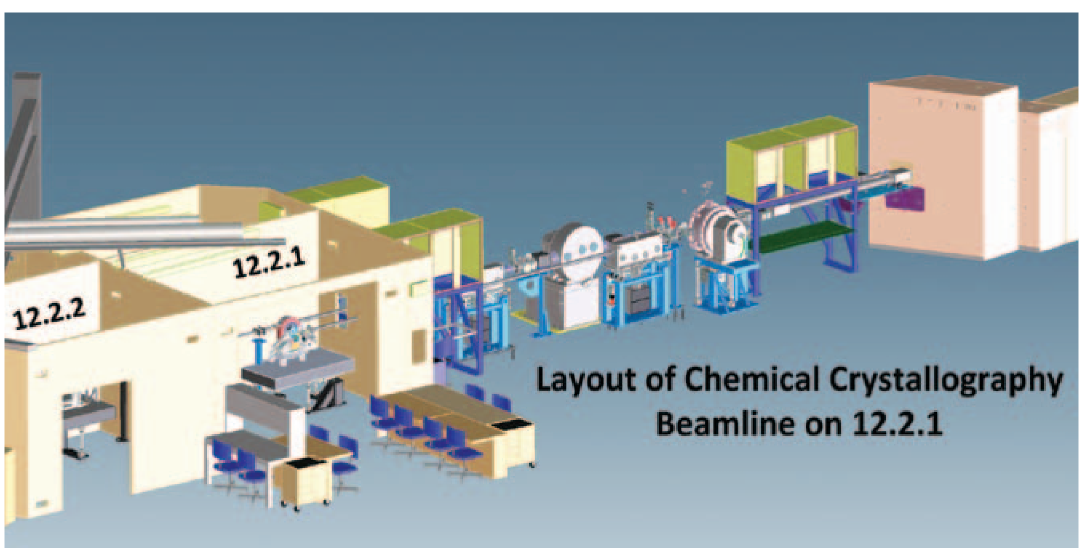
Leveraging previous developments in precision scanning systems, fast-readout CCD detectors, and high-performance reconstruction algorithms, the ALS has developed three ptychographic microscopes as part of the R&D effort leading to the new COSMIC beamline. These advances and the high coherent flux available at COSMIC will allow for the study of mesoscale materials with unprecedented spatial resolution and spectroscopic content. Prototypical ptychographic measurements performed at the Beamline 11.0.2 STXM endstation have already demonstrated the highest spatial resolution ever achieved in an x-ray microscope.



In ptychography, a sample is scanned and a diffraction pattern is measured at each sample position. The sample structure can then be calculated at a spatial resolution limited, in principle, only by the illuminating wavelength.

Chemical Crystallography—Brighter and Faster on a Superbend. Chemical crystallography on Beamline 11.3.1 has been remarkably productive, with the user community consistently generating publication numbers on par with the most productive ALS beamlines, despite being on a regular bend magnet with low flux. To expand on this success, chemical crystallography will be moving to the superbend Beamline 12.2.1. Intensity will increase by ~100 and the energy range extended from the current 7–17 keV to 7–25 keV, allowing for higher reciprocal-space coverage and thus improved data refinement. The wider usable energy range also allows for resonant scattering experiments, which exploit the absorption edges of elements to increase or decrease their effective scattering power.

The harder x-ray energies of the new beamline will also extend the range of samples and environmental cells that can be used, since pressure- and gas-environment experiments require the crystals to be placed in cells that attenuate both incoming and exiting x-rays. Moving to the superbend source will allow for diffraction from difficult samples like actinides and samples in a gas or diamond-anvil cell to be collected in a fraction of the time currently required. The user community is increasingly drawn toward these in situ experiments that give valuable insight into the functionality of materials in realistic conditions. The superbend upgrade will compensate for the increasing complexity of experiments and materials to ensure the continued high productivity of the ALS chemical crystallography program. ■



The chemical crystallography program will move from regular bend-magnet Beamline 11.3.1 to superbend Beamline 12.2.1.

SCIENTIFIC SUPPORT GROUP: STATE-OF-THE-ART INSTRUMENTS FOR NEW SCIENCE

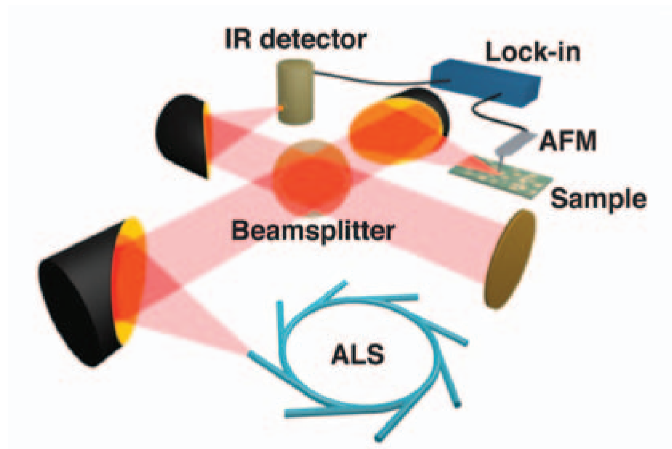
by Zahid Hussain, Jinghua Guo, Mike Martin, Hans Bechtel, Sung-Kwan Mo, and Elke Arenholz

Advanced Materials Beamline for Energy Research. There has been a rapid increase in user proposals from energy science researchers over the past six years. To meet this need, the ALS has proposed construction of the Advanced Materials Beamline for Energy Research (AMBER), with in situ soft x-ray spectroscopy and nanospectroscopy tools fully optimized for energy science. This new facility is now the highest priority in the ALS Strategic Plan.

AMBER would comprise a high-throughput monochromator with three branchlines/endstations, for in situ photon-in/photon-out soft x-ray spectroscopy (PIPOS), ambient-pressure x-ray photoemission spectroscopy (APXPS), and scanning transmission x-ray microscopy (STXM). An undulator with complete polarization control (EPU) would be its source, one of a chicaned pair in the straight section of Sector 8.

AMBER would provide in-depth understanding and ultimate control of processes at the atomic and molecular level in several fundamental research areas, including artificial photosynthesis, energy efficiency with designed catalysts, and energy storage and batteries. A recent example is from Braun et al., *J. Phys. Chem. C* **116,** 16870 (2012), where chemical reactions induced by illumination with visible light can be seen directly in the absorption spectrum. In that experiment, an external voltage was applied to a photoelectrochemical cell, and spectroscopy was performed on the device during operation. Two extra peaks were observed under illumination, but not in the dark, and only at specific bias voltages. They were identified as two types of holes induced in the valence band by photochemical reactions, one of them appearing only after the application of a 600-mV anodic bias but not at 200-mV bias, demonstrating that surface electronic states are altered under operating conditions.

The proposed AMBER facility will provide the characterization and interpretation tools needed to develop science and technology for renewable energy applications.



Schematic layout of the Beamline 5.4 AFM-based scattering scanning optical microscope.

Broadband Infrared Nanospectroscopy. Characterizing and ultimately controlling chemical processes, material heterogeneity, energy flow, complex dynamics, and emergent behavior on length scales larger than single atoms but smaller than the macroscopic world is a grand challenge for mesoscale science and many next-generation and bio-inspired technologies. Chemical fingerprinting and optical spectroscopies with true nanoscale spatial resolution will be key tools in addressing these grand challenges.

At ALS Beamline 5.4, we have combined the high-brightness broadband synchrotron infrared source with an atomic-force microscope (AFM)-based scattering scanning optical microscope. With this new tool, we image and spectroscopically probe deeply subwavelength, sub-50-nm features on a variety of samples. Topography, overall infrared images, and spectra covering 750–6000 cm^{-1} are rapidly acquired employing asymmetric Fourier-transform infrared (FTIR) interferometry where both magnitude and phase are recovered. The wavelength rage is limited only by the beamsplitter and detector. We find the phase signal is closely related to far-field absorbance, meaning nanospectroscopy can take advantage of the wealth of FTIR reference libraries available for spectroscopic identifications. Early uses of this novel technique include probing the surfaces of battery

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SSG: State-of-the-Art

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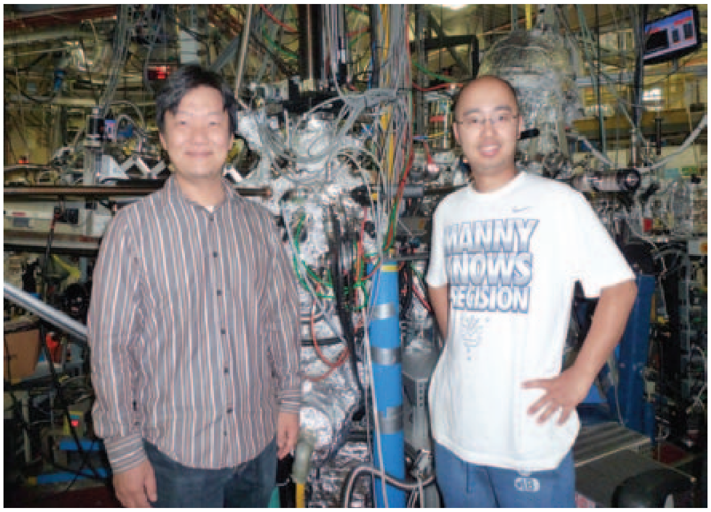


Sung-Kwan Mo and Yi Zhang at the MBE station at Beamline 10.0.1.

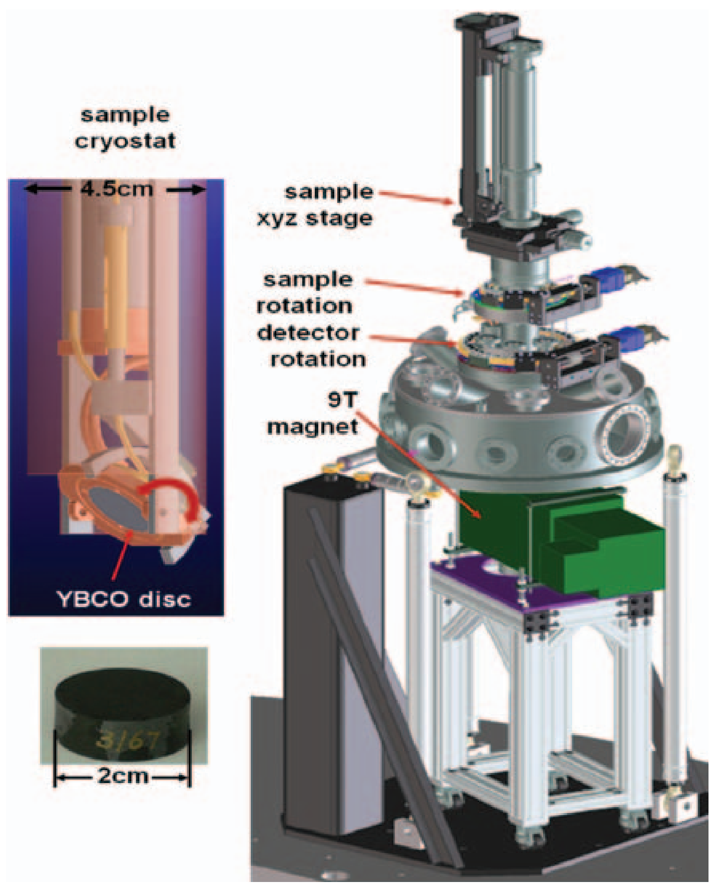
materials, patterned proteins, peptoids, and biominerals.

MBE Thin-Film Growth at Beamline 10.0.1. The molecular-beam epitaxy (MBE) chamber for growing thin-film samples is now fully commissioned and in operation at Beamline 10.0.1. The growth chamber is equipped with a four-channel e-beam evaporator with separate flux monitors, four effusion cells optimized for different temperatures, a silicon evaporator, a crystal thickness monitor, a reflection high-energy electron diffraction (RHEED) system, an argon sputter gun, a low-energy electron diffraction (LEED) system, and a sample stage capable of heating up to 1500 °C. Thin-film samples grown in this chamber can be easily transferred, in ultra-high vacuum, to the ARPES system for in situ characterization and investigation of electronic structure.

The current setup is geared toward growing thin-film samples of topological insulators and metal dichalcogenides. Recent data show successful growth of these films with precise layer-by-layer thickness and doping controls, and concomitant novel changes in electronic structure.

New Endstation for Soft X-Ray Scattering at Beamline 4.0.2. Intriguing novel phenomena occur at interfaces: the discontinuity in chemical composition can lead to ferromagnetism between antiferromagnets or superconductivity between insulators. Moreover, the coupling of order parameters present in the constituent materials can be achieved at an interface and allow, for example, control of magnetic order through electric fields or strain. A fundamental understanding of the physics behind these interface phenomena will be essential to developing the principles for directed materials design and to exploit these remarkable properties for applications.

Periodically spaced interfaces represent meso- and nanoscale phenomena that can ideally be probed using resonant soft x-ray scattering, an x-ray diffraction technique where constructive interference of x-rays occurs only in selected geometries determined by the nanometer periodicity of the of interfaces and photon energies characteristic of a particular electronic (charge, spin, or orbital) state. To allow the study of interfaces in high magnetic fields using resonant soft x-ray scattering, an ALS team has developed a new endstation now installed at Beamline 4.0.2. In this system, a superconducting disc is situated behind the sample. The disc is cooled through its transition temperature in an applied external magnetic field. When the external field is ramped down to zero, a persistent current induced in the superconductor creates persisting magnetic fields of up to 4 T at the sample while still allowing full access to the sample for scattering experiments. ■



Schematic of the resonant soft x-ray scattering endstation at Beamline 4.0.2.

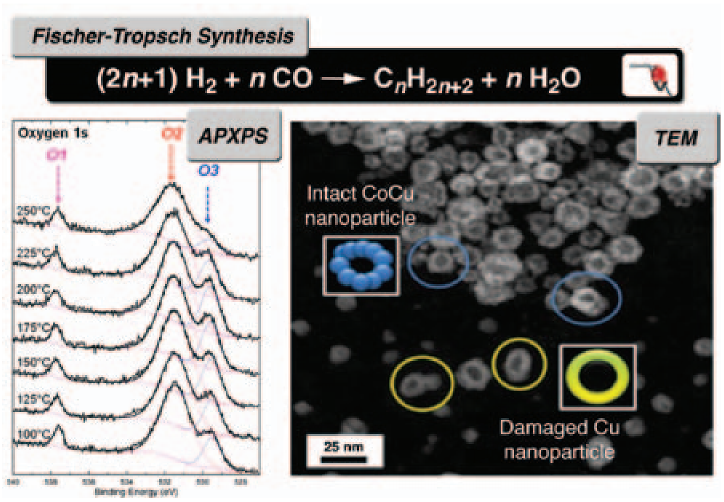
BEAMLINE 11.0.2: FROM NANOCATALYST STRUCTURE TO ANTHROPOGENIC IRON

by Hendrik Bluhm and Mary K. Gilles

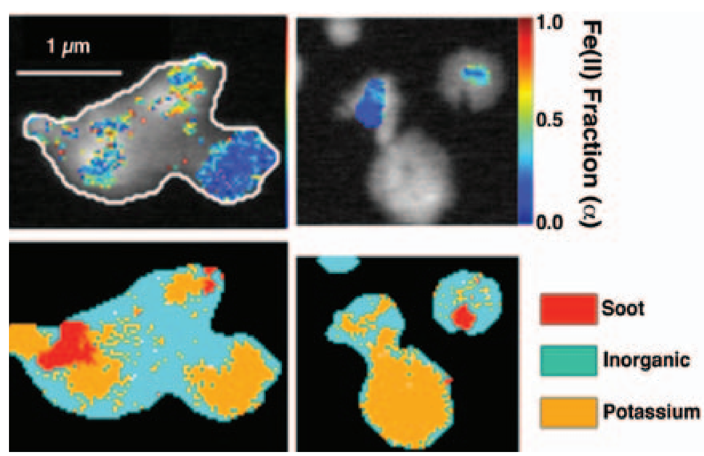
The Molecular Environmental Science Beamline (11.0.2) is scientifically productive while continually improving existing and new endstations. Scanning transmission x-ray microscope (STXM) experiments investigate soils, catalysts, star dust, actinides, atmospheric aerosols, and magnetic properties of materials. Experiments at the ambient-pressure x-ray photoelectron spectroscopy (APXPS) endstation focus on environmental science, catalysis, and energy science.

The APXPS endstation was used to study the evolution of mixed cobalt–copper nanocatalysts during a model Fischer-Tropsch reaction. In this reaction, raw sources of carbon and oxygen (coal, biomass, etc.) are converted into high-purity liquid fuels of various chain lengths and oxygen content. An in-depth understanding of the nanocatalyst structure and its evolution is critical for controlling the reaction products. APXPS monitored the surface chemistry of CoCu nanoparticles exposed to CO and H_2 gas. APXPS revealed that the active nanoparticles were metallic, but it also showed changes in the CoCu surface composition upon reaction. This insight was combined with chemical mapping of the nanoparticles before and after the reaction, using transmission electron microscopy (TEM). TEM further showed the consequence of extended reaction time on the nanoparticle structure: Co was found to migrate out of some CoCu nanoparticles. The combination of APXPS and TEM unraveled the mechanism leading to nanoparticle degradation during prolonged reaction, suggesting new routes to improve Fischer-Tropsch nanocatalysts that in turn promote the use of cleaner fuels. [S. Carenco et al., *J. Phys. Chem. C* **117**, 6259 (2013).]

Bioavailable iron is an important ocean nutrient that impacts the global carbon budget. Hence, understanding Fe sources and their oxidation states is important. Major iron sources for atmospheric particles include dust storms and anthropogenic combustion. The partitioning of Fe into Fe(II) and Fe(III) is often determined by the ratio between two peaks in the NEXAFS spectra—707.8 eV for Fe(II) and 709.5 eV for Fe(III). Examining this ratio for calibration standards showed the best agreement at intermediate values of Fe(II) fraction (~0.2–0.5). However, even for the standards, significant uncertainty arises for low fractions of Fe(II) while at large Fe(II) fractions (>0.65), the standards deviate significantly from one another. Speciation experiments using STXM examined the ratio, α, where α = [Fe(II)/[(Fe(II) + Fe(III)], in atmospheric aerosols transported from China and collected in Okinawa. These experiments showed an insignificant enrichment of Fe(II) compared to dust standards. However, the iron-containing aerosols contained sulfur (typically from power plants), contained Fe distributed as submicrometer inclusions, and also frequently contained soot. The combination of iron with sulfur is important because sulfuric acids could promote conversion of Fe(III) to Fe(II), increasing its bioavailability. Hence, it appears that power plants were the source and there was not a significant enhancement of bioavailable iron. [R.C. Moffet et al., *J. Geo. Res. D: Atmos.* **117**, D07204 (2012).] ■



The Fischer-Tropsch synthesis reaction. Left: In situ APXPS spectra of the nanoparticles during reaction. Right: Ex situ TEM observation of the nanoparticles after reaction.



Top: Maps of Fe(II) fraction, α, on individual atmospheric particles. Bottom: Carbon map of the particles indicating soot, inorganic components, and potassium. However, the collocation of Fe with soot and the submicrometer size of the Fe inclusions indicate a combustion source.

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BEAMLINE 9.0.2: LASERS, LASERS, LASERS

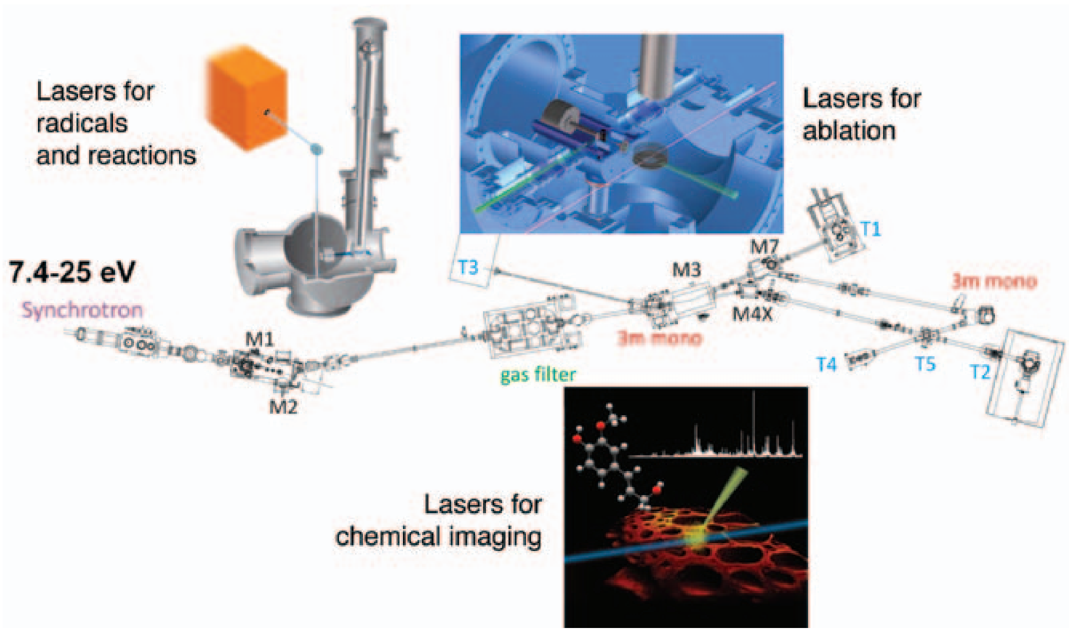
by Musa Ahmed

The use of lasers forms a common thread in three highlights, summarized below, from the Chemical Dynamics Beamline (9.0.2).

Criegee intermediates (CIs) are the main intermediates in atmospheric reactions between ozone and organic molecules, resulting in the formation of free radicals, organic acids, carbonyl compounds, and organic aerosols. Such reactions contribute to local photochemical smog and global climate change. Advances in understanding the chemistry of CIs long remained elusive because these compounds could not be observed directly. However, this changed recently as the Sandia Combustion Research group, in collaboration with atmospheric scientists from the University of Manchester, used photoionization methods at Beamline 9.0.2 to detect these intermediates and measure their kinetics of reaction with various molecules relevant to atmospheric and environmental chemistry. The reactions were initiated by pulsed laser photolysis. Following their earlier study of the smallest CI (CH_2OO), they moved on to the next larger CI, CH_3CHOO. They detected two distinct CH_3CHOO conformers, syn- and anti-, both of which react readily with SO_2 and with NO_2. The anti-CH_3CHOO is substantially more reactive toward water and SO_2. Reaction with water may dominate tropospheric removal of Criegee intermediates and determine their atmospheric concentration. [Taatjes et al., *Science* **340**, 177 (2013).]

The activation of methane is a long-standing goal of catalysis due to its potential industrial utility as an inexpensive hydrocarbon feedstock. It is also the most fundamental C-H bond activation reaction. In a collaboration between beamline staff and Ricardo Metz (University of Massachusetts, Amherst), the energetics of two key species involved in methane activation by a platinum atom—the H-Pt-CH_3 insertion intermediate and $PtCH_2$ + H_2 products—have been determined by photoionization studies using a laser ablation apparatus. Coupled with kinetics experiments and high-level electronic-structure calculations, these findings provide unprecedented insight into the mechanistic details of this reaction. Comparison of the potential energy surfaces for the reactions of Pt and Pt^+ with CH_4 shows that charge has surprisingly little effect on reactivity. [Perera et al., *Angew. Chem. Int. Ed.* **52**, 888 (2013).]

Soil organic matter is important because its decay drives life processes in the biosphere. However, analysis of organic compounds in geological systems is difficult because of their intimate association with mineral surfaces. In a collaboration between beamline staff, colleagues from Berkeley Lab's Chemical and Earth Sciences Divisions, and Markus Kleber (Oregon State University), laser desorption synchrotron post-ionization (synchrotron-LDPI) mass spectrometry has been introduced as a novel analytical tool to characterize the molecular properties of organic compounds in mineral–organic samples from terrestrial systems. When combined with secondary ion mass spectrometry (SIMS), it can provide complementary information on mineral composition. The combination of synchrotron-LDPI and SIMS shows that the energetic conditions involved in desorption and ionization of organic matter may be a greater determinant of mass spectral signatures than the inherent molecular structure of the organic compounds investigated. The latter has implications for molecular models of natural organic matter that are based on mass spectrometric information. [Liu et al., *Anal. Chem.* **85**, 6100 (2013).] ■



Schematic of Beamline 9.0.2, where a wide variety of chemical dynamics experiments can be performed with the help of lasers. Three examples are depicted in the insets.

CXRO: A SHARPER IMAGE OF THE FUTURE

by Patrick Naulleau

The strong collaboration between the Center for X-Ray Optics (CXRO) and the ALS has continued to flourish this past year. A new industry-funded EUV full-field microscope has been commissioned and progress has continued on the development of a new 8-nm resolution nanopatterning tool for advanced photolithography materials research. Pushing the boundaries of x-ray/EUV optics and instrumentation remains central to our mission, and much progress has been made in the past year.

In June 2013, the world's most advanced EUV microscope went online at Beamline 11.3.2, and demand outstripped supply from day one. The much-anticipated SHARP microscope (SEMATECH High-NA Actinic Recticle review Project) provides semiconductor companies with the means to push their chip-making technology to new levels of miniaturization and complexity by providing unparalleled views into the advanced nanolayered masks used as the circuit master in the computer-chip manufacturing process. The microscope allows us to characterize the mask at the same wavelength and optical conditions at which it will be used, providing direct insight into exactly how damaging each defect will be to the final circuit-patterning process.

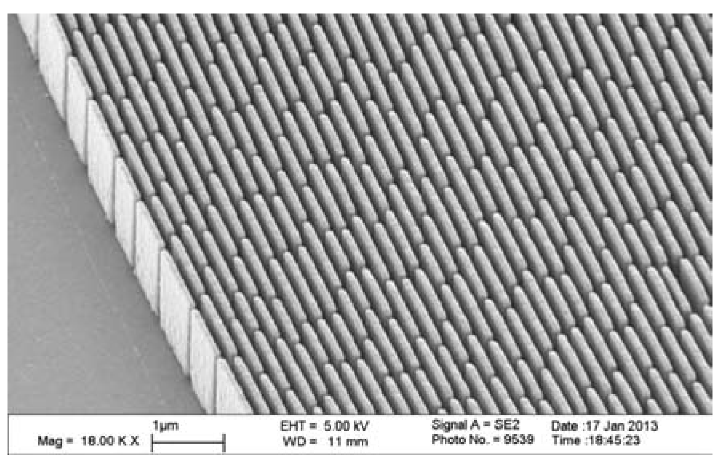
Right next door to SHARP is the SEMATECH Berkeley Micro Exposure Tool (MET) on ALS Beamline 12.0.1, providing ultrahigh-resolution EUV printing capability for developing next-generation photoresist materials. The MET drives resist innovations in the areas of resolution, sensitivity, and line-edge roughness, with nearly 3000 new lithography materials being tested over the past year.

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. It is both a cornerstone of the Magnetic Materials program in the Materials Sciences Division and continues to support a vibrant ALS user program beyond nanomagnetism. XM-1 has made important contributions to the study of magnetic vortex structures over the past year (see "Reversing the Circulation of Magnetic Vortices" on p. 3).

In the area of zone-plate optics, we have concentrated on high-aspect-ratio structures for hard x-rays. We continue to push for both higher resolution and increased thickness. On the ultrahigh-resolution soft-x-ray front, we continue to work directly with lithographic materials suppliers to push the limits currently imposed by photoresist materials on the highest achievable resolution. This work leverages our activities in EUV lithographic materials research.

CXRO has also put considerable effort into the reduction of stress in multilayer optical coatings for the soft x-ray regime. This issue of stress becomes critically important as we push toward thinner substrates and even free-standing mirrors. Progress in this area has allowed us to fabricate a wavefront-preserving EUV beamsplitter for use in scanning microscopy systems. The narrow-band, multilayer, freestanding beamsplitter is designed to select a single harmonic from a high-harmonic-generation EUV source. The new low-stress process results in a film with much less distortion compared to one produced by conventional multilayer processes. ■

Members of the SHARP team (left to right): Markus Benk, Arnaud Allezy, Takeshi Katayanagi, Ahmet Pekedis, Will Cork, Carl Cork, Kenneth Goldberg, Jeff Gamsby, Rene Delano, Senajith Rekawa, Jason DePonte, Weilun Chao, Iacopo Mochi, Michael Dickinson, and Daniel Zehm. Not shown: M. Gideon Jones.



A 100-nm outer-zone-width, 1.5-μm-tall gold zone plate fabricated by CXRO and now in use.

STRUCTURAL BIOLOGY: A THOUSAND STRUCTURES SOLVED AT 5.0.2

by Corie Ralston

The Berkeley Center for Structural Biology (BCSB) manages both the industry-funded Beamlines 5.0.1, 5.0.2, and 5.0.3, and the HHMI-funded Beamlines 8.2.1 and 8.2.2. Beamline 5.0.2 recently passed a milestone, with its 1000th structure solved this year; this was a study led in part by Genentech, one of the participating research team (PRT) members for Beamlines 5.0.1 and 5.0.2. In fact, Beamline 5.0.2 had its best year ever, with 100 structures solved in one year. This is especially significant considering that the largest portion of the beam time is allocated to pharmaceutical companies who mostly conduct proprietary research, leading to far fewer published structures than have actually been solved at the beamline. Not be outdone, the HHMI beamlines had 30 high-profile publications between them in the last year alone.

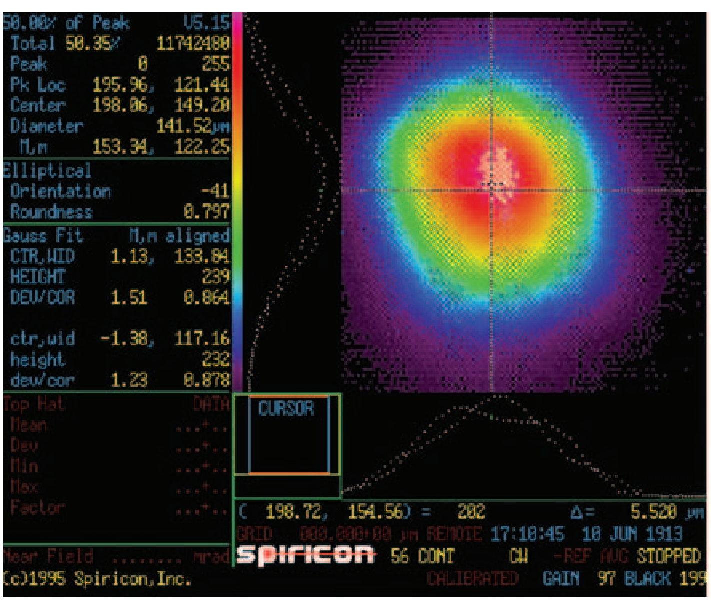
What has enabled this high level of productivity? Improvements across all the BCSB beamlines in the previous year have resulted in greater beam stability, brightness, and new capabilities. These upgrades and features have become more important as users bring smaller and more weakly diffracting crystals to the beamlines.

The primary x-ray mirrors for 5.0.1, 5.0.3, 8.2.1, and 8.2.2 were removed during the recent shutdown to allow for installation of several upgrades. A new cooling system has been installed on the 5.0.1 and 5.0.3 mirrors; these mirrors are subject to very high thermal loads and the improved efficiency of the new cooling system has reduced thermal distortion and improved beam focus. All four x-ray mirrors also had newly developed position-encoder systems installed. The new encoders improve the accuracy and reproducibility of the mirror-positioning system and enable more aggressive optimization of the beam focus. In combination with the ALS lattice upgrades, this has increased the brightness of Beamlines 8.2.1 and 8.2.2 significantly.

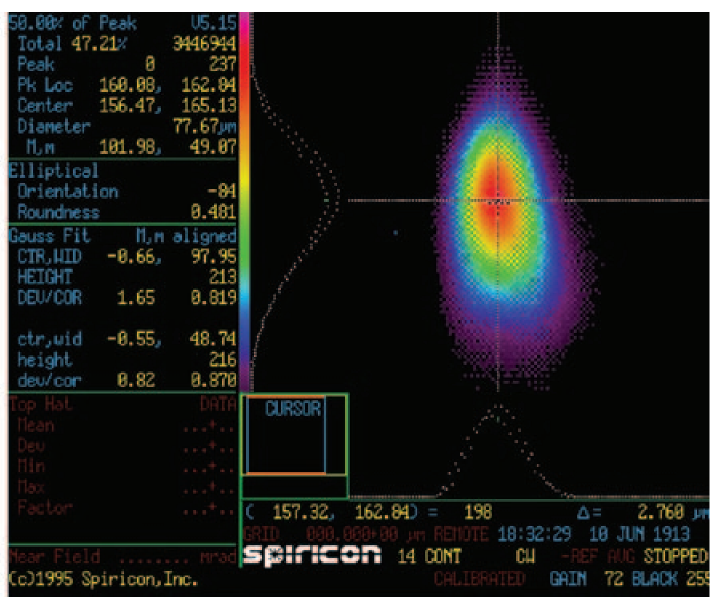
A measure of the improvement is the raw beam size on Beamline 8.2.1, which has decreased from 150 x 122 µm to 100 x 50 µm because of the ALS lattice upgrades and the new mirror encoder. Note that the same number of photons is now going into a much smaller spot size.

While the mirror upgrades are integral to the BCSB beamlines, another new and unique addition is a portable humidifier device that can be used on all the beamlines. This device allows users to precisely control the relative humidity of the environment surrounding the crystal. By slowly decreasing the humidity in a highly controlled pseudo-equilibrium manner, a crystal may be induced to undergo a phase change to a denser space group with increased crystallographic order. This can result in higher-resolution diffraction data, reduced mosaicity, or the crystallization of previously disordered domains.

Many such features and upgrades have contributed to the past and continued success of all the BCSB beamlines. ■



Beamline 8.2.1 raw beam size, old lattice (150 x122 µm).



Beamline 8.2.1 raw beam size, new lattice (100 x 50 µm).

THE ALS COMMUNITY

LAUNCHING THE NEXT TWO DECADES

by Roger Falcone

It's been a very busy and productive year for ALS staff and users. While there is reason to be concerned about, and plan for, uncertainties in government funding (this year we have been both "continued and sequestered" under the Continuing Resolution and Budget Control Act), we have been focusing on a host of great projects, as well as being extremely productive in supporting users and publishing outstanding science.

We have an amazing number of activities that are finishing up. For example, we closed out and are now utilizing some major "stimulus" (ARRA) projects—fast new CCDs for dealing with high-speed signals and data resulting from brighter x-ray beams, a new superconducting vector magnetometer for measuring magnetic properties of materials, and a new elliptically polarized undulator for our ultrafast beamline. An additional project, replacing magnets in the storage ring to create higher brightness (more than a factor of three on some beamlines), has had significant effects on focused spot sizes and stability of x-rays around the ring and is enabling science that we could not have done previously because of low signals with the old larger focal spots.

Additionally, we are in the midst of constructing new undulator-based beamlines—COSMIC and MAESTRO—visible to anyone who walks by Sector 7 with its busy construction activities. These new beamlines will propel our angle-resolved photoemission capability to the nanoscale and allow us to realize the full potential of techniques in coherent scattering and diffractive imaging that we have developed with years of demonstration experiments at suboptimal beamlines. Additional construction is happening at Beamline 12.2.1 as we move chemical crystallography to a superbend that will give 100 times the performance of the previous Beamline 11.3.1.

Other improvement projects, for storage-ring rf power supplies and controls, will insure a second 20 years of reliable and stable ALS operation, as will improvements to our heating and air conditioning, and fixing the roof—all infrastructure projects that we've recently "done" but of course are never really "finished."

We are in the early stages of a major upgrade to the Web interface used to access the ALS—our user portal project. With the help of the Lab's Computational Research Division, we are redesigning that interface for more efficient and safer processes, starting from the first time a potential user connects to the ALS, to getting new experiments onto the floor, and extending to the recording of publications.

Our involvement with major DOE energy research initiatives has never been greater, with dozens of experiments that are helping the JCAP Hub develop efficient artificial photosynthesis of fuels as well as helping launch the new JCESR Hub that is focused on developing new batteries with lower cost and higher capacity. It is because of

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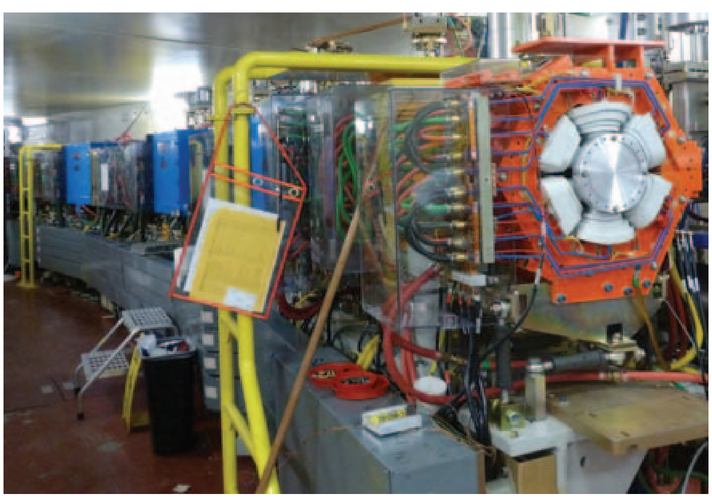

One of 48 new sextupole magnets installed in the ALS lattice as part of a major brightness upgrade.

Sector 7 after demolition of the old Beamline 7.0.1.

Falcone *continued from page 12*

U.S. Congresswoman Barbara Lee (D-CA) greeted Corie Ralston during a tour of the ALS on January 29, 2013, accompanied by Berkeley Lab Director Paul Alivsatos and ALS Division Director Roger Falcone.

X-ray microtomographic image of a spider head taken at Beamline 8.3.2. Recent visitors to the ALS mezzanine reception area were treated to a large-screen, high-definition, 3D-television version of this data.

previous investments by the ALS over many years in techniques that support the understanding of the science of sustainable energy systems that we are ready and able to support these critical new research missions for the DOE.

All this activity and our great productivity have not gone unnoticed by Congressional representatives, industries, schools, and other nations. The number of tours we have recently hosted has never been greater, and I am grateful to the whole ALS community for taking time to talk with the many visitors walking around the floor or asking questions. Personally, I greatly enjoy showing off all our activities—from 3D tomographic images of spiders, to structures of new miracle drugs, to microscopic images of cement drying, to helping make the next generation of microchips, to measuring the electronic structure of topological insulators—though as you might imagine, sometimes it's also important to have an answer as to why there is so much aluminum foil at the ALS, and explain how every year we serve 2000 users, operate 40 instruments 24/7, and publish 700 scientific papers, using beams of x-rays that are millions of times brighter than the sun and can look inside things.

As we look forward to the coming year, one of our goals will be explaining why it is a great investment for the nation to continue to upgrade the ALS. We are proposing a "diffraction-limited" or 100-times-brighter capability, which we believe we can accomplish by redoing the internal storage ring of ALS while keeping all of our beamlines. This would be a perfect project to keep the ALS the brightest soft x-ray synchrotron globally, enable important science that we are not able to do now, and launch the next two decades of continued forefront contributions to science and technology. ■

USERS' EXECUTIVE COMMITTEE UPDATE

by Corie Ralston, 2013 UEC Chair

The ALS Users' Executive Committee (UEC) has grown in recent years and now includes 15 members, including the past chair (Brandy Toner) and a student member. The UEC meets quarterly and represents the interests of all students, postdocs, and scientists who perform research at the ALS. The UEC provides a channel of communication between the ALS user community and ALS management, conveying the concerns and needs of the user community to management, and providing a means for management to update users on current and future plans for the facility. The UEC also facilitates communication between the ALS and other synchrotrons, and between users and federal funding agencies.

The UEC is very interested in hearing from users on all issues related to their research and beamtime at the ALS. For instance, one of the current topics of discussion within the UEC is the use of pseudo-single-bunch mode and how it compares to two-bunch mode. Another current discussion centers around whether beamline scientists should serve on the UEC. Please send any thoughts on this topic or any other topic of concern to individual UEC members or the entire committee at the alsuec@lbl.gov. We welcome all suggestions for the agenda.

One of the annual activities of the UEC is to organize the User Meeting. This year, the meeting will be October 7–9, and it is an especially exciting year since the ALS is celebrating

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THE ALS COMMUNITY

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UEC members who attended the January 2013 UEC meeting, from left: Yuri Suzuki, Jeff Kortright, Kevin McCarty, Peter Nico, Corie Ralston, Yves Idzerda, Gyorgy Snell, Mahati Chintapalli (student member), Adam Hitchcock, and Scott Classen.

its 20th anniversary. The chairs are Scott Classen (Physical Biosciences Division) and Peter Nico (Earth Sciences Division). With help from the outstanding ALS team, including Deborah Smith and Sue Bailey, they are organizing an impressive lineup of speakers and workshops for 2013.

Another activity of the UEC is to advocate on behalf of users at the National User Facility Organization (NUFO) annual meeting. NUFO represents the interests of users at national user facilities across the United States and organizes trips to Washington for this purpose. This year, the NUFO meeting was at Berkeley Lab, and so provided a perfect opportunity for ALS users to get involved in advocacy efforts and discussions. Yves Izerda (Department of Physics, Montana State University) and Sue Bailey (User Services Group Leader) are leading the efforts of the NUFO steering committee. Contact them for more information on how to get involved. ■

ALS COMMUNICATIONS: CONNECTIONS AND COLLABORATIONS

by Elizabeth Moxon

Over the past year, members of the ALS Communications Group have focused on developing new opportunities to expand and engage our diverse audiences. From the Open House to exciting new collaborations with local, national, and international scientific and communications groups, our new social, educational, and media activities have promoted the ALS scientific program—and the staff and users who make it such a success—in new and often fun ways.

The year kicked off with a brunch celebrating the 20th anniversary of the dedication of the ALS. Current and past employees squeezed into the conference room in the User Support Building to catch up with colleagues and view photo displays of years past. Former ALS Director Jay Marx, who oversaw the building of the facility, welcomed attendees and provided humorous anecdotes about some of challenges faced during construction. More celebratory activities—ALS timeline, T-shirt design contest, and a fall reception—are planned for the rest of the year.

A slide show of historical photos entertained staff during the brunch.

In June, the National User Facilities Organization (NUFO) held their annual meeting at Berkeley Lab. In the months preceding the event, the Communications Group was asked to develop a communications strategy to promote the meeting and to advertise an impending visit to Congress intended to show off the science produced at national user facilities. An impromptu collaboration quickly developed to tackle the problem, with fellow communicators Linda Vu (Computing Sciences Division) and Massie Ballon (Joint Genome Institute) rapidly expanding NUFO's reach through social media. This collaboration expanded to include staff from other national facilities who assisted us by providing input for two posters showcasing "The Faces and Places of National User Facilities," featured at the NUFO event in DC. Go to NUFO.org to catch up on the organization's activities and to see photos of the meeting and the Congressional visit.

An international collaboration of synchrotron communicators from the ALS, ESRF, and Soleil recently received a grant from the France-Berkeley Fund to develop a scientific educational program for high-school students that will introduce them to the opportunities and challenges of synchrotron research. Members of lightsources.org will also contribute ideas and materials so that the program can be reproduced at the more than 65 light sources throughout the world.

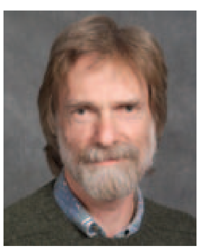
ALS users and staff are always invited to send us new ideas on how we can reach and expand our audiences. Email us at alscommunications@lbl.gov or visit us on the third floor of the User Support Building. ■

THE ALS COMMUNITY

HONORS AND AWARDS

Karim Benzerara, an ALS user from the Institut de Minéralogie et de Physique des Milieux Condensés, was awarded the Mineralogical Society of America Award for 2012 for his contributions to the understanding of mineral–microbe interactions and biomineralization.

Hendrik Bluhm, an ALS user from Berkeley Lab's Chemical Sciences Division, received the Friedrich Wilhelm Bessel Research award from the Humbolt Foundation for his outstanding research record. The award includes an invitation to spend up to one year cooperating on a long-term research project with specialist colleagues at a research institution in Germany.



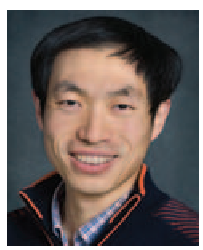
Warren Byrne, of the ALS Accelerator Physics Group, won a 2013 Director's Award for Exceptional Achievement for his "assistance in support of the Lab's mission and strategic goals provided in a spirit of cooperation and commitment to excellence."

Alexander Hexemer, an ALS staff scientist, received a DOE Early Career Research Program Award to create a "High Performance Toolkit for Photon Science." The "toolkit" is designed to accelerate the rate of scientific discovery by enhancing the rate at which the enormous amount of data, generated at light sources like the ALS, can be analyzed.

Howard Padmore, an ALS staff scientist, was presented with the Albert Nerken Award by the American Vacuum Society for his "sustained contributions to the design, development, and application of novel synchrotron x-ray instrumentation used to study a range of scientific problems from biology to materials and solid state science."

Rob Ritchie, an ALS user from Berkeley Lab's Materials Sciences Division (MSD), was awarded the 2014 Acta Materialia Gold Medal Award from The Minerals, Metals & Materials Society for his development, along with fellow MSD researcher Tony Tomsia, of a lightweight ceramic material that has an unprecedented combination of strength and toughness.

Gabor Somorjai, an ALS user from Berkeley Lab's Materials Sciences Division, was one of 18 individuals recognized by the National Academy of Sciences for outstanding scientific achievement in the physical, biological, and social sciences. Somorjai received the NAS Award in Chemical Sciences for his "groundbreaking experimental and conceptual contributions to the understanding of surface chemistry and catalysis at a microscopic and molecular level."



Wanli Yang, an ALS staff scientist, was part of a team of Berkeley Lab researchers who won an R&D 100 Award for their invention of a new material for use in rechargeable batteries. Termed the Conducting Polymer Binder, the material can boost battery storage capacity by 30% and is made from a type of flexible plastic glue that holds electrode materials together while facilitating the shuttling of electrons and positively charged lithium ions. The team also included Berkely Lab scientists Gao Liu, Lin-Wang Wang, and Vincent Battaglia and postdoctoral fellows Sang-Jae Park, Mingyan Wu, and Shidi Xun.

This year's American Physical Society (APS) Fellows include three scientists from the ALS: **John Byrd, Howard Padmore,** and **David Robin.** Only half of one percent of APS members are elected by their peers to be Fellows in any given year for exceptional contributions to the physics enterprise, including outstanding research, important applications, leadership or service to physics, and significant contributions to physics education.

THE ALS COMMUNITY

OPEN HOUSE: ADVENTURES IN SCIENCE

by Shauna Kanel

In the biggest Open House in recent history, more than 6000 people came up the hill last October to see what goes on at Berkeley Lab. More than 1500 of them made their way even further up the hill to visit the ALS for tours, talks, and hands-on activities to help them understand how we use electrons, magnets, microscopes, and computers to conduct research at the ALS. At the X-Ray Café on the ALS patio, staff and scientists spoke one-on-one with guests about how the ALS works, why and how scientists use it, how it is funded, and plans for the future. For nearly five hours, visitors took guided tours of the ring, talking directly with beamline scientists about their research, reading and asking questions about posters, and learning how the facility runs from the many staff and volunteers stationed around the ring. Again this year, guests had the opportunity to have souvenir family photos taken in the high bay of the User Support Building. At the end of their tour, visitors received a buildable model of the ALS as a keepsake of the Open House. Thanks to the many volunteers who worked tirelessly to make the ALS's participation in the Lab's "Adventures in Science" Open House such a huge success! You can see photos on the ALS flickr page (http://www.flickr.com/photos/advanced-lightsource). ■

Ingrid Peterson (Physical Biosciences Division) leads a hands-on demonstration at the "Make Your Own Protein Crystals" exhibit.

Alastair MacDowell engages a large crowd of visitors at the "Adventures in Energy" exhibit.

FACILITY UPDATES

FACILITY FOCUS: THE NEW X-RAY OPTICS LABORATORY

by Valeriy Yashchuk

Construction of a new optical metrology laboratory in the User Support Building, with comprehensive control of environmental conditions, was completed in late summer 2013. Because the lab's capabilities now extend far beyond classical optical surface metrology to include the entire spectrum of in situ and ex situ metrology, and because it also supports the design and fabrication of x-ray optics and optical and mechanical systems, the new lab has been renamed, from the Optical Metrology Laboratory (OML) to the X-Ray Optics Laboratory. Headed by Valeriy Yashchuk, the lab is a unique facility that will serve the needs of several West Coast DOE research organizations that lack dedicated on-site optical metrology capabilities, including the Linac Coherent Light Source (LCLS) at SLAC and the Center for X-Ray Optics at Berkeley Lab.

In October 2009, a white paper was submitted to the DOE on radically improving the efficiency, precision, and reliability of the old OML. The plan pointed out that the OML had been operating since 1995, using instrumentation with an accuracy of ~1 µrad (rms) for measuring a surface slope, adequate for the x-ray optics existing at that time.

Now, however, optics with a slope variation of better than 0.1 µrad (rms) and a shape accuracy of < 1 nm are vital for third- and fourth-generation synchrotrons and free-electron laser facilities. The upgrade plan called for (1) radical improvement of environmental and operational conditions; (2) replacement, upgrade, and extension of existing instrumentation; and (3) a comprehensive R&D program to keep capabilities adequate to the constantly increasing demand for state-of-the-art instruments and measurement and calibration methods.

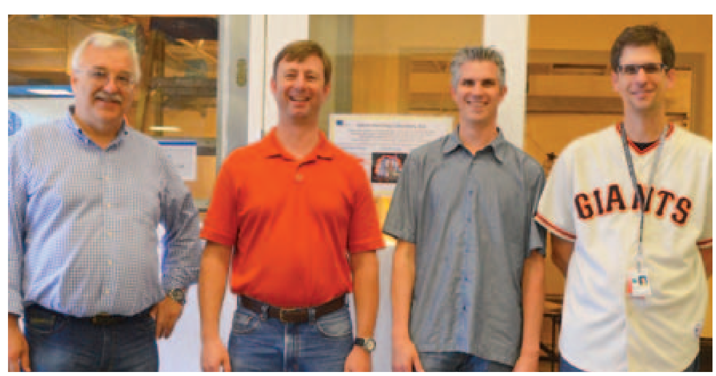
A clean, stable environment will enable efficient operation and the highest possible precision during routine operations with minimal repeatable cross-check measurements, as was needed in the old lab for suppression of instrumental drift and systematic errors. The new lab will be in a class 10,000 cleanroom, a standard that permits only 10,000 particles of 0.5 µm or larger per cubic foot of air.

The instrumentation upgrade and R&D program have been inseparable parts of the plan to radically improve the OML. Starting in 2004, a comprehensive R&D program was directed at improving surface metrology at long spatial wavelengths. Specifically, the OML's long-trace profilers (LTP-II and DLTP) have been significantly upgraded. However, much of the precision of the ALS OML, comparable to that of the world's best synchrotron metrology labs, is due to the development of original methods for the suppression of systematic and drift errors as well as a method for calibrating the modulation transfer function (MTF) of surface profilometers. The latter method was invented and developed by Valeriy Yashchuk and Wayne McKinney in collaboration with Peter Takacs (Brookhaven National Laboratory). The calibration allows for significantly more reliable simulation of beamline performance using the metrology data for the inspected x-ray optics. Recently, the invention was awarded a Proof of Concept Commercialization Gap Grant from the University of California, Office of the President.

To fully realize the advantages of the developed methods, the instrumentation must be mounted on heavy granite tables (to increase temperature stability and decrease vibration) with air-bearing translation systems capable of precision 2D scanning over the surface under test. An example of an extremely carefully designed granite table with the option for 2D slope metrology is the table used for the HZB/BESSY-II NOM (Germany). Now, such systems are also in use at DIAMOND (UK), ALBA (Spain), and SPring-8 (Japan). A similar system has been purchased for our new lab as well. During the moving process, we also plan to replace the OML atomic-force microscope and 17-year-old ZYGO GPI interferometer with modern high-performing systems.

It is important to note that the ultimate performance of the lab's long-trace profilers is limited by systematic errors that increase when the entire angular range is used for metrology of significantly curved x-ray optics. The profilers' systematic errors will be reliably reduced with a sophisticated calibration method suggested at the ALS and based on a universal test mirror (UTM). Work to develop a UTM system is in progress as a collaborative project of the ALS, HZB/BESSY-II, and PTB (Germany) metrology teams. ■


A multidisciplinary team of designers, engineers, and final users worked on the construction project. First row: Ross Schaefer, Valeriy Yashchuk, and Nikolay Artemiev. Second row: Lito Magbitang, Brett Young, Thomas Chytrowski, Daniel Merthe, and Erin Wood.



Valeriy Yashchuk, Ross Schaefer, Steve Rossi, Jeffrey Troutman, and Charles Taberski (not in photo) carefully supported and monitored the construction project to ensure the lab's consistency and long-range functionality.

FACILITY UPDATES

ACCELERATOR AVAILABILITY AND RELIABILITY

by Christoph Steier, David Robin, and David Richardson

The last year saw many changes to the accelerator aimed at maintaining and improving the availability and performance of the ALS. While implementing these major upgrades, the ALS continued to deliver well over 5000 hours of user operations. The bulk of the upgrades were accomplished within the usual annual shutdown. The length of the shutdown periods is not fixed and depends on the scope of the work, which has been larger recently to accomplish the upgrade goals of the ALS strategic plan. Despite the much larger scope and complexity of the work, the average number of user hours has remained very high and teething problems due to the upgrades have been reasonably small.

One of the most important performance parameters of a user facility that is compared to all similar facilities is availability. It is defined as the ratio of delivered versus scheduled user time. While the DOE goal for availability is >90%, the ALS internal goal is >95%. Maintaining a high availability as the facility becomes more mature and complex remains a challenging task. The table below, which gives the availability of the ALS for the last four fiscal years, shows that we continue to exceed both the ALS and DOE goals.

Over the last years, the distribution and length of shutdowns has varied, based on the timing of major pieces of equipment (rf, power supplies) being ready for installation. There were nearly 6000 user hours scheduled in FY10, with the ALS running the entire year without a major shutdown. This resulted in two major shutdowns falling into FY11, resulting in just below 5000 scheduled user hours. FY12 has been more in line with historic averages with just below 5500 scheduled user hours. Also, the ALS continues to deliver periods of unscheduled light in addition to the scheduled periods: an additional 136 hours (2.3%) in FY10, 80 hours (1.6%) in FY11, and 76 hours (1.4%) in FY12.

Reliability is another important performance parameter, distinct from availability. A good measure of reliability is the mean time between failures (MTBF). The table also shows the MTBF numbers for the last four fiscal years. In FY12 we achieved an MTBF of 38 hours, somewhat below our goal, which remains to increase the MTBF to be reliably above 48 hours (less than 1 fault per two days).

Hours Scheduled and Delivered

	Scheduled hours	Delivered hours*	Availability	Mean time between failures (hours)
FY09	5471	5278	96.5%	37.4
FY10	5980	5707	95.4%	32.4
FY11	4939	4836	97.9%	46.6
FY12	5483	5283	96.4%	38.0

*Does not include unscheduled hours delivered to users.


Left: New storage-ring bend-magnet supply, operational since March 2013. Right: One of 48 new sextupole magnets installed in the storage ring as part of the brightness upgrade.

As explained in previous years, there are ongoing programs to analyze every single fault, analyze fault patterns and develop improvement programs. Major areas responsible for the reduced MTBF in FY12 were AC line glitches, cooling-water problems, control-system problems, and power-supply faults. The first two problems have been long-standing and the resolution is complex, with improvements to be implemented in FY13 and FY14. Fixes for the other two areas were implemented in FY12 and in early FY13. Another contribution to reduced MTBF in FY12 was the above-mentioned teething effects from the installation of new equipment. After resolution of those issues, however, the new systems have been reliable, trading off a slightly worse MTBF performance in the short term to achieve improvements in the long term.

In addition to upgrades aimed at improving reliability and energy efficiency, the last year also saw the implementation of the brightness upgrade, aimed at improving ALS performance. The upgrade was successfully completed in FY13. Also, new beam-position monitors for improving the orbit feedbacks and orbit stability are being developed under the leadership of NSLS-II in collaboration with ALS and SPEAR and have been tested at the ALS. Finally, new insertion devices (elliptically polarizing undulators) with tailored period lengths are being built for several new (and one existing) beamlines as part of the ALS strategic plan. ■

FACILITY UPDATES

USER DEMOGRAPHICS AND PUBLICATIONS

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–FY12). 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 "high-impact" label includes 20 journals so designated by the DOE: *Science, Nature, Proceedings of the National Academy of Sciences USA, Physical Review Letters, Nature Physics, Applied Physics Letters, Journal of the American Chemical Society, Nature Chemistry, Nature Chemical Biology, Angewandte Chemie International Edition, Cell, Nature Structural and Molecular Biology, EMBO Journal, Nature Materials, Advanced Materials, Nano Letters, Nature Nanotechnology, Nature Geoscience, Environmental Science & Technology,* and *Nature Photonics.* ■

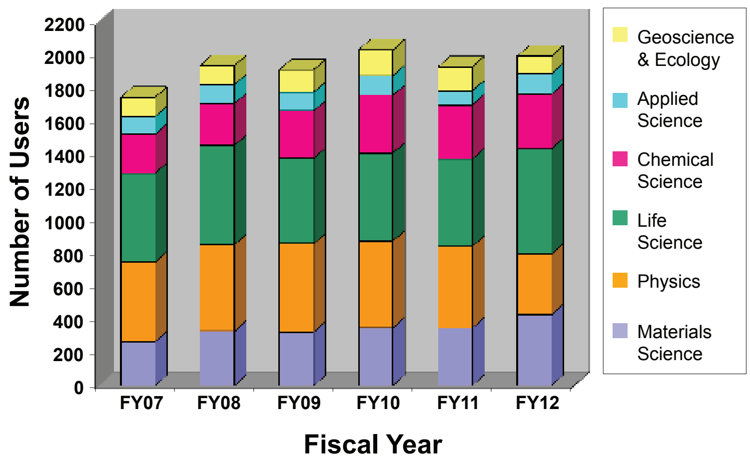


Figure 1. Bar graph showing the relative numbers of users in various areas of science.

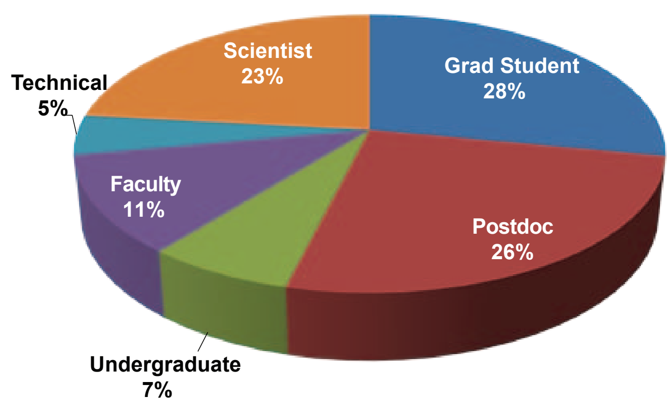


Figure 2. Pie chart showing the different categories of researchers at the ALS.

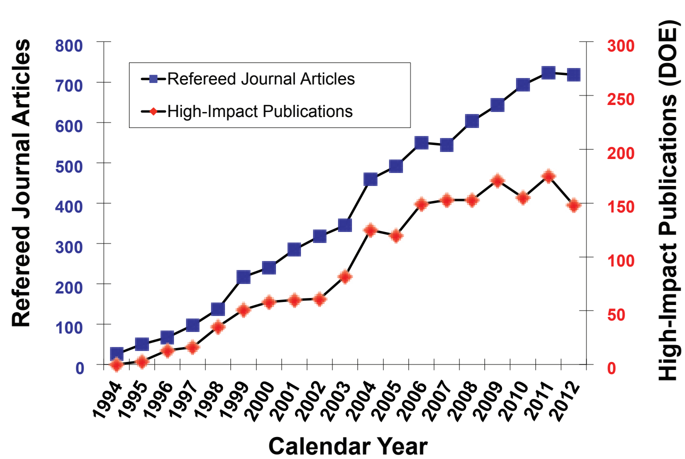


Figure 3. Graph of growth in refereed journal articles (blue), and high-impact publications (red).

Brightness Upgrade

continued from page 1

The ALS now has one of the smallest horizontal emittances of all operating third-generation light sources. Initial user operation has been very successful, with most beamlines benefitting significantly from the upgrade.

The new sextupole designs were completed in 2011 by a collaboration between Berkeley Lab and the Shanghai Institute of Applied Physics (SINAP). Three different designs were needed because of space constraints. One of the designs is optimized for small hysteresis and fast time response. These are used as the primary correctors in the fast orbit feedback. All new sextupoles also contain skew quadrupole coils, improving the vertical beam-size stability by providing an effective correction of the skew quadrupole errors of the planar insertion devices.

Magnet production, carried out at SINAP, started with prototype magnets just after the design reviews in early 2011. The pole shapes were manu-

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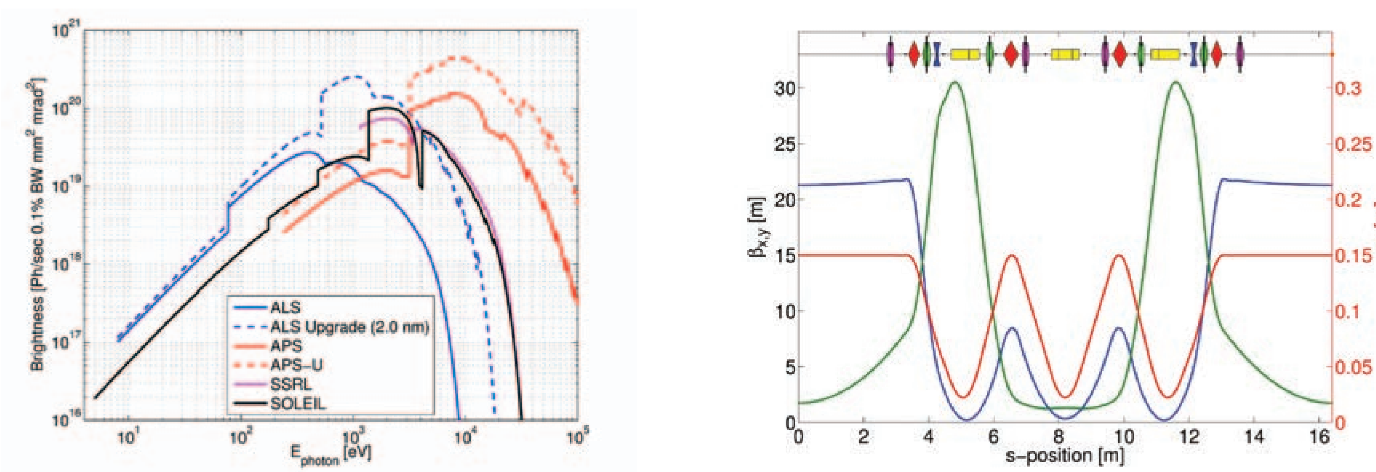
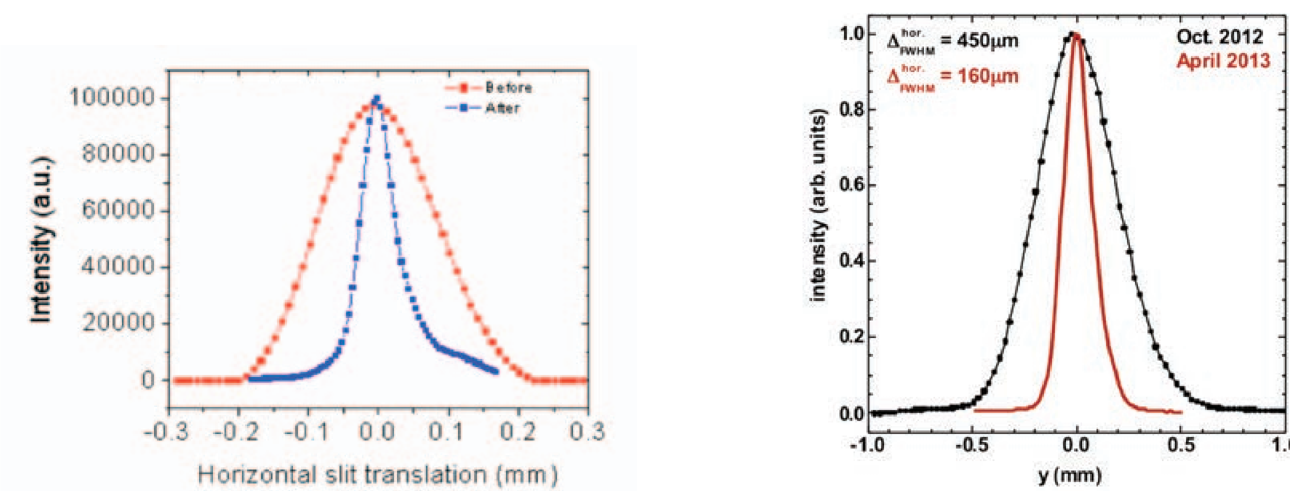


Left: Comparison of ALS brightness after top-off upgrade (solid blue) with the brightness after the low-emittance upgrade (dashed blue) and several other light sources. Right: ALS upgrade lattice with 2.0-nm natural emittance at 1.9 GeV.



Comparison of the horizontal beam profile before and after the upgrade measured at Beamlines 12.3.2 (left) and 6.3.1 (right) showing the factor of three improvement in brightness (vertical scale is renormalized). Courtesy of M. Kunz, N. Tamura, and K. Jenkins.

Brightness Upgrade

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factured by wire electric-discharge machining on fully assembled magnet cores to achieve excellent field quality. Manufacture was completed in summer 2012, on time to achieve the project installation milestones. During construction there was a detailed quality assurance program and all magnets were fully qualified by electrical, mechanical, and magnetic measurements. Precise fiducialization was carried out both mechanically and with the help of magnetic measurements. All magnets exceeded the field-quality requirements.

To create sufficient space for the new magnets, several vacuum-chamber and stand modifications were completed in 2012. Installation began in late 2012 during short maintenance shutdowns, with 13 of the 48 new sextupoles being installed ahead of time. This allowed for testing their corrector functionality (time response, hysteresis) and incorporation into slow and fast orbit feedback. The remaining magnets were installed during the spring 2013 shutdown. At the same time, all new power supplies and equipment-protection systems were installed, and the top-off interlock ranges were enlarged.

Migration to the new lattices was quick (a few hours), after verification of all magnet polarities and magnet transfer functions in the old lattice with the beam on. Simulations beforehand had predicted excellent dynamic and momentum aperture as well as lifetime for the optimized upgrade lattice. These predictions were quickly confirmed during initial commissioning. During the two weeks of commissioning, the harmonic sextupole settings were optimized, the insertion-device feed-forward (tune, beta beating, coupling) was updated, the new dispersion bump for the fs-slicing facility was implemented, and the top-off interlocks were retested with new ranges.

The dispersion bump was refined after final lattice optimizations. The dynamic aperture and momentum aperture, with the fs-slicing lattice insertion included, are similar to the bare lattice results and commissioning went quickly. The new lattices also provide a larger intrinsic horizontal separation of the sliced electron beam. We are currently in the process of evaluating how to make best use of this and expect that it will eventually allow a much better signal-to-noise ratio for the slicing facility. Optimizing photon beamlines with new beam spots progressed quickly, and user beamlines were able to resolve the brightness increase. The dynamic aperture and momentum aperture for the upgrade lattice were confirmed to be very close to the expected ones and, as predicted, the Touschek beam lifetime after the upgrade, despite the smaller horizontal and slightly smaller vertical emittance, is larger than before the upgrade, due to the larger dynamic momentum aperture. ■

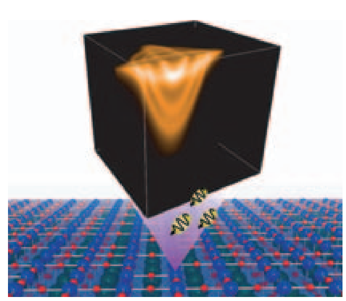
Science Roundup

continued from page 3

A Step Forward Toward Oxide Electronics

W. Meevasana et al., *Nat. Mater.* **10**, 114 (2011); P.D.C. King et al., *Phys. Rev. Lett.* **108**, 117602 (2012)

Two-dimensional electron gases (2DEGs)—narrow conducting channels at the surfaces and interfaces of semiconductor materials—are the bedrock of conventional electronics. The startling 2004 discovery that 2DEGs could be engineered at the interface between insulating transition-metal oxides initiated a worldwide effort to harness the functionality of oxide materials for advanced electronic applications. Now, using only intense synchrotron light, scientists have been able to create and control 2DEGs at the bare surfaces of insulating oxides. As well as suggesting a potential methodology to spatially pattern 2DEGs in a wide variety of complex oxides, this discovery opens a new avenue for spectroscopic investigation of these novel electronic systems.

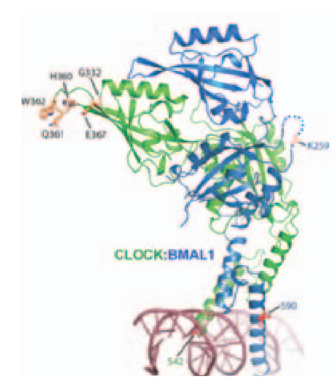


A multi-orbital 2D electron gas created at the surface of transition-metal oxides upon exposure to intense UV synchrotron light.

Central Activator Keeps the Circadian Clock Ticking

N. Huang et al., *Science* **337**, 189 (2012)

Most living organisms have adapted their physiology and behavior to match the daily cycle of light and dark generated by the rotation of the earth, operating with a period of approximately 24 hours. Molecular machines in cells ultimately control such rhythmic behavior, the details of which—the "circadian clock"—are largely conserved. To understand the inner workings of the circadian clock, researchers from the University of Texas Southwestern Medical Center and Howard Hughes Medical Institute used ALS Beamline 8.2.1 to determine the three-dimensional structure of the transcriptional activator CLOCK:BMAL1 complex, the central positive component of the mammalian circadian clock.



Model of CLOCK:BMAL1 in complex with E-box DNA.

Advanced Ceramic Composites continued from page 1

Engineering, is now studying a new generation of ceramic materials—ceramic-matrix composites—that are most noteworthy for their resistance to fracture at temperatures that would literally melt current state-of-the-art engine materials [*Nat. Mater.* **12,** 40 (2013)].

"Since the jet was first developed around World War II, the efficiency of the engine—its capabilities and fuel consumption—has literally been controlled by how hot you can run it," says Ritchie. "In my mind these materials represent one of the biggest structural-material breakthroughs in a long, long time. They're offering the potential of a 200- to 300-degree engine-temperature increase, whereas currently we'd kill for a 5-degree increase!"

The heat-resistant properties of advanced ceramic materials have been known for a long time, but the materials' poor resistance to fracture has always been the major drawback to their use as structural materials. This new generation of ceramic composites shows promise due to being reinforced with ceramic fibers to form microstructures, similar to natural materials such as teeth and bone. Because of the complexity of their design, they have the toughness to resist cracking at ultrahigh temperatures in extreme environments.

Jet or gas-turbine engines made from ceramic composites would weigh considerably less than today's engines and operate at much higher temperatures, which translates into far greater fuel efficiencies and reduced pollution. The materials' potential has industry players heavily interested in techniques for characterizing damage and fracture in these materials at extremely high temperatures. Ritchie's current ALS research is part of a collaborative research project with Teledyne, funded by NASA and the U.S. Air Force.

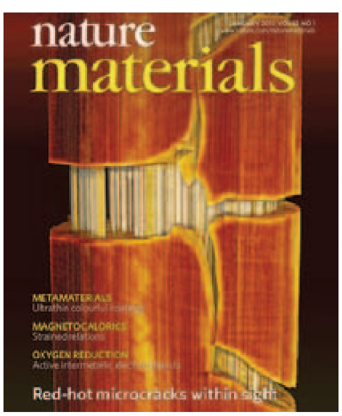
Considerable research on ceramic-composite materials was done in the 1980s and 90s, but the use of these materials was deemed to be too much of a risk. Recently, some engine companies, with GE at the lead, have made a decision to use these materials because they offer so many potential benefits. Ritchie notes that Boeing recently ordered new GE engines that will utilize a significant number of ceramic-composite components for their latest 777 series planes, which will go into service in 2019. "These materials are a reality, even though there still are many unanswered questions about their performance," says Ritchie.

The key to the materials' safety is how the microstructure can contain and impede the growth of numerous small cracks that are created when loads are applied at high temperatures. "Metals bend before they break, so you can see if something's going to go wrong," says Ritchie. "These new ceramic-composite materials have this same quality in that the fibers hold them together so that they don't break catastrophically, which gives designers a safety cushion."

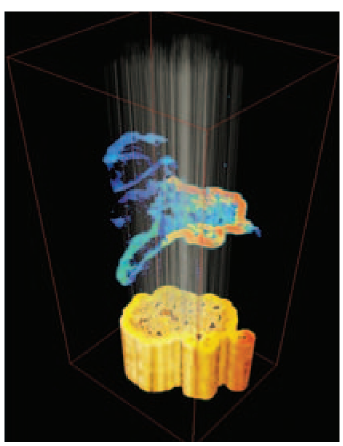
The real-time analysis of the mechanical properties of these space-age materials at ultrahigh temperatures has always been a challenge, but the microtomography facility at Beamline 8.3.2, developed by an ALS team including Alastair MacDowell and Abdel Haboub, allows Ritchie and his colleagues to study this process in 3D with a spatial resolution below one micrometer, under load, and at temperature (see also "X-Ray Microtomography—New Environments" on p. 4). Ritchie and his team have used these capabilities to fully resolve sequences of microcrack damage as cracks grow under load at temperatures several hundred degrees higher than previously possible. The observations are key ingredients of the high-fidelity simulations used to compute failure risks under extreme operating conditions.

Ritchie foresees ceramic-composite engines on the commercial market by the end of the decade, mainly in the aerospace industry. But there are other applications for these materials as well, notably in cleaner energy production: gas turbines made from ceramic-composite materials would run hotter and faster, making for cleaner energy production.

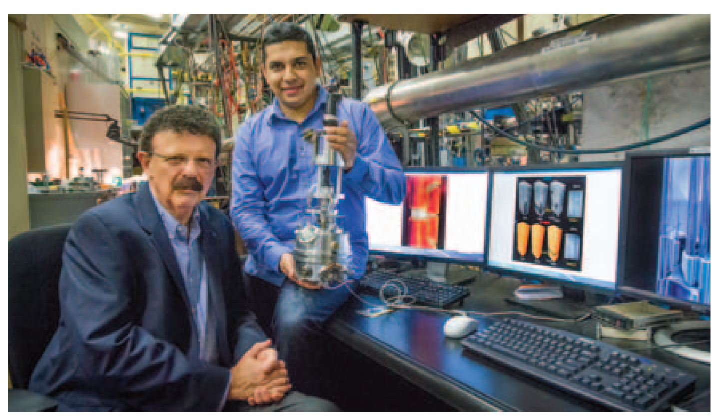
"We're getting measurements of the mechanical properties at temperatures that are literally unprecedented, coupled with wonderful 3D images and quantitative data of the damage under load, all results that can be accurately used to provide future predictions of the structural integrity and safe lifetimes of these exciting new materials," says Ritchie. ■



This cover of *Nature Materials* shows the cracking of the material as it is pulled apart at ~1750 °C. The internal structure of the composite is revealed showing strands of silicon carbide holding the cracked ceramic matrix together.



The power of 3D microtomography is revealed in this image of the breaking ceramic. The figure shows the ceramic matrix (in yellow below, transparent above), the silicon carbide fibers (white), and cracks in the matrix (multicolored, blue to red). The colors indicate the crack width (blue = 0 microns, red = 100 microns). Cracks in individual fibers can also be shown as the temperature and load changes.



Rob Ritchie and postdoc Hrishi Bale, holding the high-temperature cell.

Pseudo Single Bunch

continued from page 1

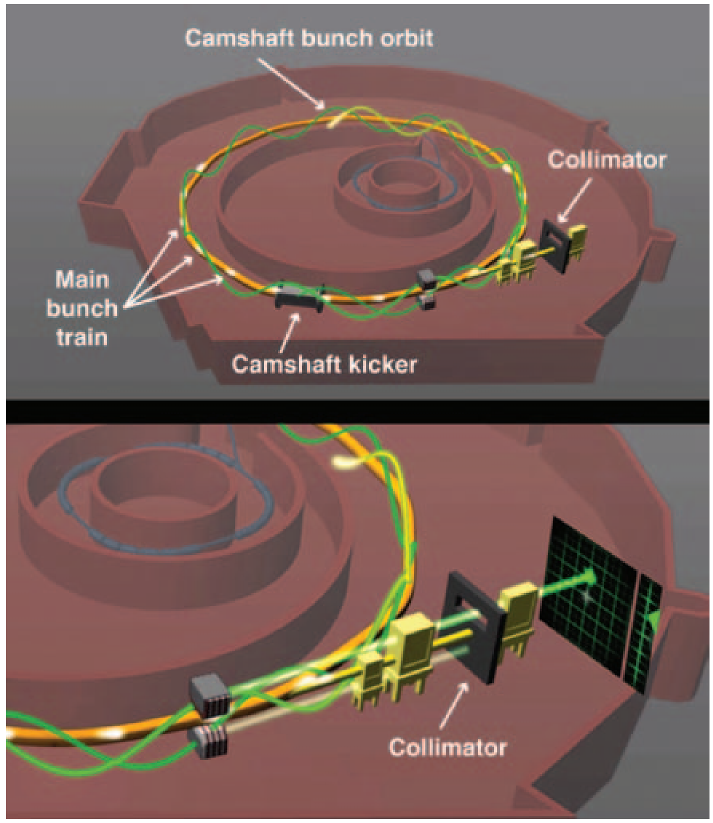
Experiments that require light emitted only from a single bunch can stop the light emitted from the other bunches using a collimator. Other beamlines would only see a small reduction in flux due to the displaced bunch. As a result, PSB complements the scheduling of multibunch and timing experiments—currently, the ALS only offers these capabilities for four weeks out of its yearly operating schedule. With PSB operation, the ability to conduct time-of-flight experiments could be available year-round.

The ALS has been successful in serving multiple users with a diverse set of requirements such as high-photon flux and brightness, a large range of wavelengths, variable polarization, and relatively short pulses. However, a major limitation of the ALS and other synchrotron light sources is the inability to serve two other classes of experiments simultaneously—brightness or flux-limited experiments and timing experiments. Typically, storage-ring light sources operate with the maximum number of bunches possible, with a gap for ion clearing. By evenly distributing the beam current, the overall beam lifetime is maximized. The ALS normally operates with a train of 296 bunches out of a possible 328, with a single "camshaft" bunch in the middle of a 60-nanosecond gap.

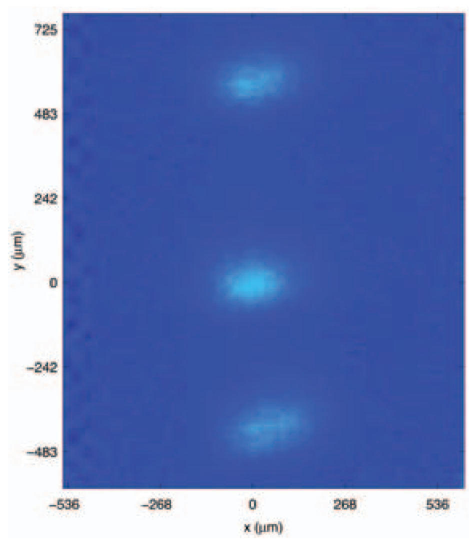
The concept of using a camshaft bunch in multibunch operations started many years ago and originated out of the desire for some timing researchers to operate during multibunch mode. However, most timing users cannot use the camshaft due to the short 60-ns gap. The ones that do must use gated detectors or expensive mechanical choppers to reduce the background from unwanted bunches. These choppers are challenging to fabricate and operate, and for beamlines that operate without a monochromator, they have to absorb about a kW of power while rotating at high speeds. Furthermore, the rotating frequencies of choppers constrain the repetition rate of the external laser.

The idea behind PSB operation is to use a high-repetition-rate (MHz), short-pulse (< 100 ns) magnet to vertically kick the camshaft bunch relative to the bunch train. Then, by blocking the light from the multibunch train in the beamline, only light from the camshaft bunch reaches the experiment. Putting this bunch in the middle of the ion-clearing gap reduces the required bandwidth of the kicker magnets.

PSB operation has been tested at the ALS in various forms. For example, using one kicker magnet running at the ring repetition rate (1.5 MHz), PSB can be operated in a fixed high-repition-rate mode by permanently putting the camshaft bunch on a different orbit. It can also operate with an adjustable repetition rate, from milliseconds to microseconds, with a novel "kick-and-cancel" (KAC) scheme. By adjusting the ring tune and the PSB kick pattern, the camshaft bunch can be first displaced to a different orbit and then kicked back to its original one within a few turns. This kick-and-cancel process can be repeated on demand, thus creating single-bunch pulses with adjustable repetition rates. This KAC scheme can significantly alleviate the complications of using high-power choppers and substantially reduce the rate of sample damage. It allows the use of non-gated detectors, increasing the variety and quality of experiments that can be done. ■



Top: Image shows the ALS storage ring filled with a train of bunches (white) and a single vertically displaced camshaft bunch on a two-turn closed orbit (green). Bottom: Light from the main bunch train is blocked at the beamline, leaving only light from a camshaft bunch.



Beam image measurement at the ALS's diagnostic beamline (3.1). For this measurement, the storage ring was filled with a 5-mA single camshaft bunch, and the beam was kicked at the maximum kick-and-cancel frequency.

Happy
20th Anniversary
ALS!


ALS staff photo circa 1993


ALS staff photo 2003


ALS staff photo 2013

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ALS Advanced Light Source

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