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ADVANCED LIGHT SOURCE

ACTIVITY REPORT 1994

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August 1995
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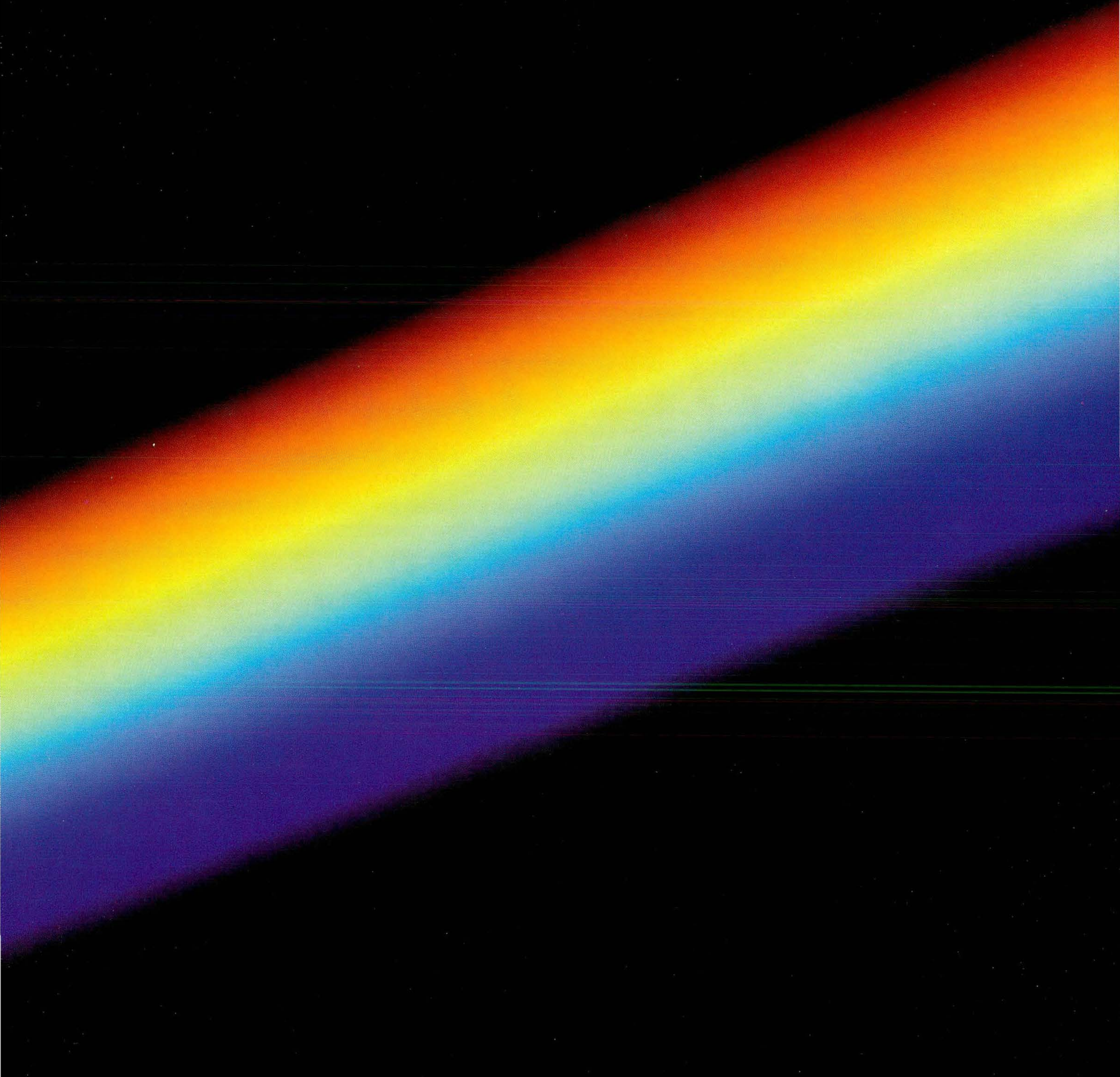

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Light—a yardstick,
a language, a tool
that can perform
a thousand tasks.

Whether defined in limited context as the range of visible wavelengths, or in the broader sense to include the entire electromagnetic spectrum from radio waves to gamma rays, it illuminates and unveils the world around us.

Almost all the knowledge we have about our universe is ultimately derived from the interaction of light with matter. The simple yet beautiful rainbow of colors produced by a prism, the iridescence of soap bubbles, and the perfect symmetry of the diffraction of light by a crystal—when observed and analyzed—reveal invaluable information about the nature of light and the materials illuminated. Now scientists have a new light source to explore the nature and structure of matter: the Advanced Light Source (ALS). The ALS is America's brightest light in the soft x-ray and ultraviolet range of the spectrum and one of the first third-generation synchrotron sources in the world.

The delivery of light to the first user experiments at the ALS in October 1993 signaled the beginning of a new era in synchrotron-based research. This success was echoed in 1994 as the ALS proved itself a robust and dependable performer, achieving a record of 94.5% reliability for user shifts (actual/scheduled). In addition to maintaining consistent, high-quality operations, we devoted considerable effort toward constructing and commissioning new beamlines, resulting in a substantial increase in the number of operational user beamlines (from three to eight) and the number of users working at the ALS.

Our high level of performance in operations and the quality of our engineering and R&D efforts have led to continued strong support from the scientific community, such as the decision by the Office of Health and Environmental Research to fund the start of the ALS Protein Crystallography Facility. We are hopeful that such levels of support will continue, and that the ALS will receive the additional funding required to expand our operations and services so that we can meet the ever-increasing demand for use of the facility. The high-quality scientific results from the first full year of user operations, some of which are highlighted in this report, are ample evidence that, given the opportunity, this facility can open new frontiers for science and technology.

ADVANCED LIGHT SOURCE

ACTIVITY REPORT 1994

The Advanced Light Source, a national user facility located at Lawrence Berkeley National Laboratory of the University of California, is available to researchers from academia, industry, and government laboratories. Its building incorporates the dome that once housed the 184-inch Cyclotron built by the Laboratory's founder E.O. Lawrence.

The ALS Activity Report is designed to share the breadth, variety, and interest of the scientific program and ongoing R&D efforts in a way that is accessible to a broad audience. Recent research results are presented within four areas of scientific investigation, and are designed to demonstrate the capabilities of the ALS in these areas, rather than to give a comprehensive review of 1994 experiments. Although the scientific program and facilities report are separate sections, in practice the achievements and accomplishments of users and ALS staff are interdependent. This user-ALS staff collaboration is essential to help us direct our efforts toward meeting the needs of the user community, and to ensure the continued success of the ALS as a premier facility.

August 1995 ▲ LBL-35912/UC-411
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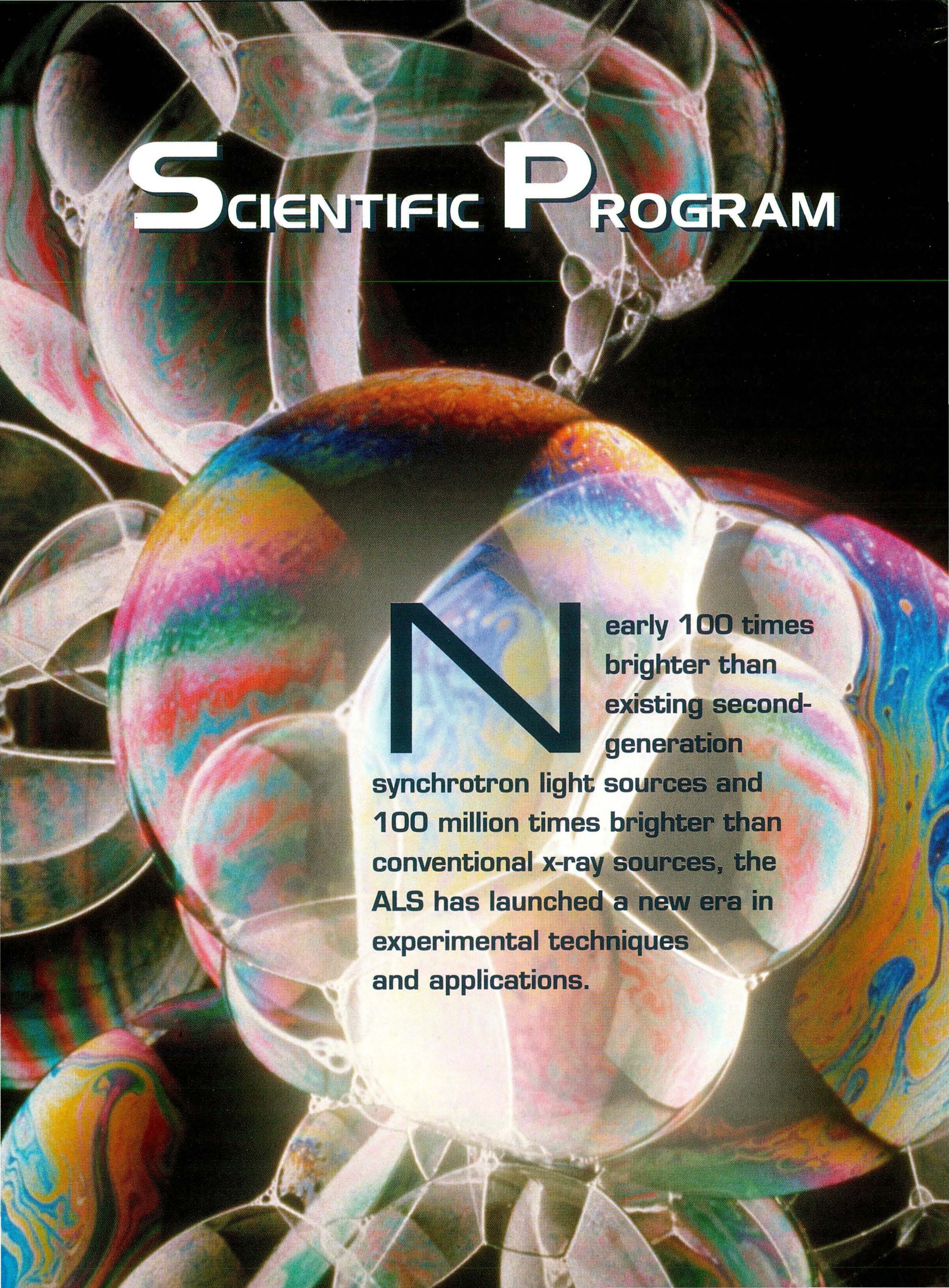
SCIENTIFIC PROGRAM



Nearly 100 times brighter than existing second-generation synchrotron light sources and 100 million times brighter than conventional x-ray sources, the ALS has launched a new era in experimental techniques and applications.

Scientists from around the world have used the ALS's soft x rays and ultraviolet light to probe matter at the atomic level, investigating structures, processes, and spatial features beyond the reach of visible light. Their explorations cover familiar areas, including those of environmental or medical interest, as well as experiments pushing the leading edges of pure science and developing technologies. The pages ahead give a sampling of what has already been achieved using this remarkable tool and provide a glimpse of the exceptional science to come.

About photo (left):

Soap bubbles derive their shimmering colors from the interference of light waves. When white light, which contains the full spectrum of colors, shines on a bubble, part of the light reflects from the outside surface of the bubble wall and part reflects from the inside surface. When the two sets of reflected light waves recombine, some colors are reinforced (constructive interference) while others are extinguished (destructive interference). The colors revealed vary with the bubble wall's thickness, producing mesmerizing rainbow effects.

Atomic & Molecular Science

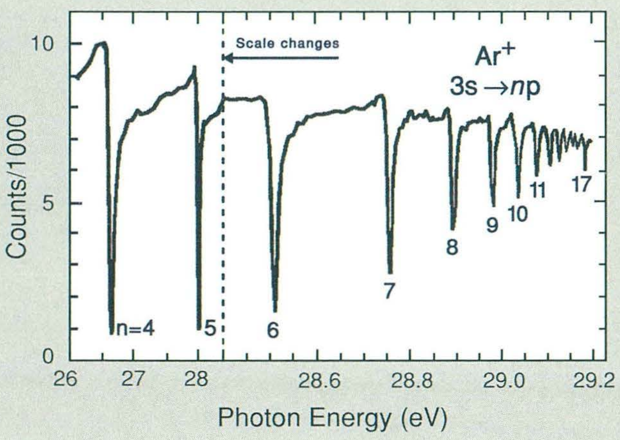
The properties of the matter comprising the world we experience trace back to its constituent atoms and molecules, making it important to understand the basics of atomic and molecular science in as much detail as possible. At the ALS, high flux, brightness, and energy resolution are allowing explorations of these disciplines using a rich variety of techniques, each of which yields information in great depth.

Atoms are the simplest physical systems for which we can test our understanding of the electronic structure of matter at its most fundamental levels. On the atomic scale, the prevailing theory is quantum mechanics, which describes particles' behavior using differential equations known as wave equations. These equations rapidly become complex when more than one particle is involved; indeed, equations describing mutual interactions among three or more particles cannot be solved exactly. To overcome this problem, atomic theorists have put forth a variety of approximations for use in solving wave equations for "many-body" systems. Validating and refining these approximations requires sensitive experiments that compare the actual behavior of systems with their behavior as predicted by theory.

The experimental techniques used to test the prevailing theories in atomic physics often must select a small fraction of the available signal in order to gain higher resolution in some variable. An example is angle-resolved photoemission: if researchers want to add electron

High-Resolution Photoelectron Spectroscopy

This photoelectron scan of argon 3s → *n*p autoionization resonances represents an advance over previous argon photoelectron studies because of its combination of high energy resolution and angular resolution (for more on autoionization resonances, see "What Are"). Previous experiments of this type had mapped argon's 3s → *n*p autoionization resonances (Rydberg series) only to *n* = 8, but this higher-resolution spectrum shows the Rydberg series all the way through *n* = 17. The angular distributions from this study have provided a test for previously untried theoretical calculations of photoelectron angular distribution; agreement of the measurements with theory (Taylor, 1977) was excellent.



The experimental setup took advantage of the high brightness and high degree of linear polarization of ALS undulator beams. The group's experimental chamber had two newly designed time-of-flight analyzers which rotated around the horizontally polarized beam, taking data at several angles relative to the electric field vector of the incoming photons. The photoelectron yield spectrum shown was taken at an angle of 0° with respect to the polarization of the synchrotron light.

Research performed by scientists from Western Michigan University, using their angle-resolved gas-phase electron spectrometry endstation at Beamline 9.0.1.

WHAT ARE... AUTOIONIZATION RESONANCES?

Autoionization resonances are the phenomena observed in spectroscopy when two processes, direct photoionization and a more indirect path involving autoionization, lead to the same final ionic state and thus interfere with each other in a quantum-mechanical sense. The two paths cannot be separated from each other experimentally since their final states are the same and they emit electrons with the same energy. In the case of the argon spectrum on p.6, the processes can be written as follows:

Direct photoionization $\mathrm{Ar(3s^2 3p^6)} + h\nu \rightarrow \mathrm{Ar^+(3s^2 3p^5)} + e^-$
Excitation/autoionization $\mathrm{Ar(3s^2 3p^6)} + h\nu \rightarrow \mathrm{Ar^*(3s3p^6}n\mathrm{p)} \rightarrow \mathrm{Ar^+(3s^2 3p^5)} + e^-$

where *n* represents an atomic energy level of 4 or more. In direct photoionization, the atom absorbs a photon ($h\nu$) and emits a 3p electron (e^-) directly. The other, indirect process has two steps: excitation, in which the atom absorbs a photon and one 3s electron is excited to an *n*p state, and autoionization, in which the excited state decays and emits an electron. Photoemission spectra show a separate autoionization resonance (on the spectrum, a characteristically shaped rise and dip in photoelectron counts vs. incoming photon energy) for each energy level *n* to which 3s electrons are excited.

Research Techniques, 1994–1996

	Beamlines
• Coincidence detection	9.0.1, 9.0.2*, 9.3.1*
• Crossed-molecular-beam dynamics studies	9.0.2*
• Fluorescence spectroscopy	7.0.1, 8.0, 9.0.1, 9.3.1*
• Photoabsorption spectroscopy	6.3.2, 9.0.1, 9.3.1*, 9.3.2
• Photodissociation	9.0.1*, 9.0.2*, 9.3.1*
• Photoelectron spectroscopy (incl. ZEKE)	9.0.1, 9.0.2*, 9.3.1*
• Time-of-flight spectroscopy	6.3.2, 9.0.1, 9.3.1*

*Techniques planned for use in 1995–1996 angular distribution information to the electron energy data they can measure by simpler techniques, they must use either a large position-sensitive detector (which detects electrons emerging at multiple angles simultaneously) or a scanning method (which measures electrons in one direction at a time). Either way, there is less signal in any one detector position than if a single detector element intercepted all electrons (as in angle-integrated photoelectron spectroscopy). Other techniques which must use smaller signals to gain more detailed data include coincidence detection, spin measurements, and near-threshold probes of photon-energy-dependent processes.

Researchers using these techniques benefit from very high flux (photons per second) on their samples in order to produce high-quality data within a reasonable amount of time. This is due not only to specialized techniques, but to the low density of their samples: atomic physics experiments generally use gas samples with less than one-millionth the number of atoms per unit volume found in normal atmosphere. Under such conditions, ALS beams' brightness (high flux combined with low emittance, or tight directional focus) is important for two reasons. High flux is required in order to excite enough atoms in the sample to produce an appreciable signal. Low emittance is important because high-resolution grating-based monochromators accept photons only over a narrow slit and a small angle. Many photons from a high-emittance source would be stopped by the monochromator slits before reaching the detector, reducing monochromator throughput and usable flux.

Using innovative experiment designs and detectors, scientists at the ALS are seizing the opportunities offered by its high-quality beams and going even further. One new

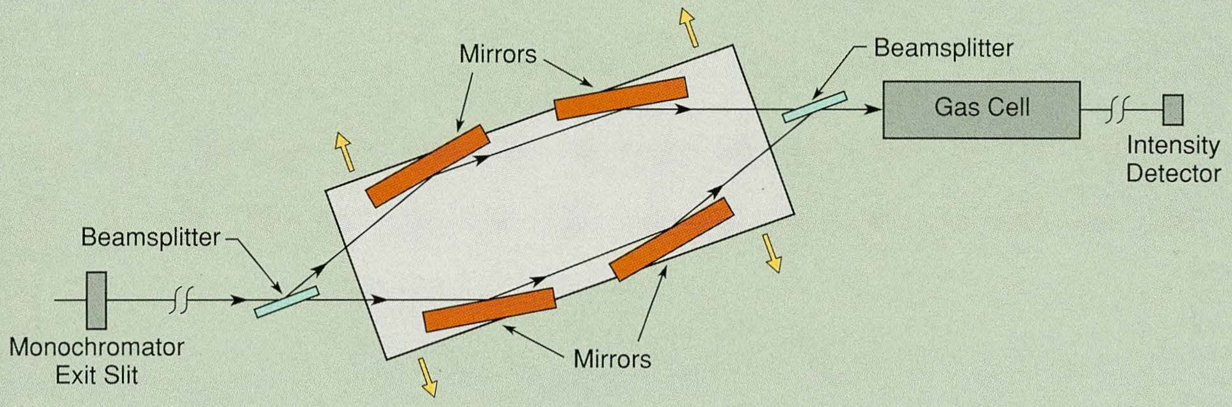
Challenging the Limits of Resolution

There are a number of problems in the physics of atoms and small molecules that challenge the resolution of the diffraction grating spectrometers that are traditionally used for vacuum-ultraviolet (VUV) and soft x-ray experiments at synchrotron radiation facilities. An interesting example is the helium absorption spectrum in the region from 60 to 80 eV, where both electrons are excited. With only two electrons, helium is a prototype three-body system in atomic physics and is important for the study of electron-electron correlation.

Although present technology continues to push the limits of resolution obtained in helium photoabsorption experiments (the current record is a resolving power of ≅ 60,000 achieved at the ALS in March 1995), it is still not sufficient to measure all the line-shape parameters and to resolve the extremely narrow resonances predicted by theory. The special position of helium in atomic physics sustains the interest of a large community of theoretical investigators and a continuing demand for higher-resolution experimental data.

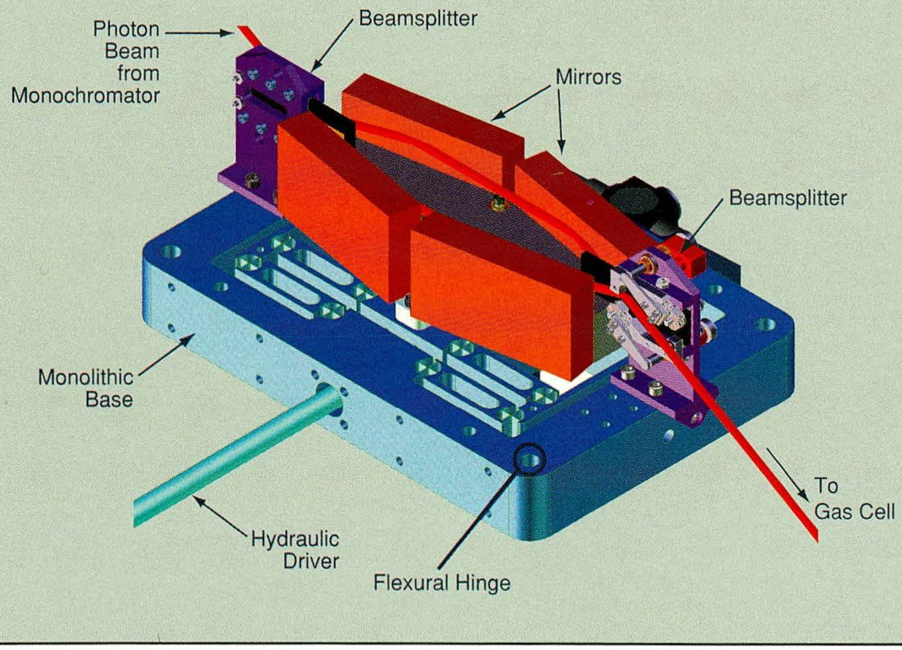
To address the desire for improved resolution, the ALS is developing a soft x-ray Fourier transform spectrometer with a resolving power target of 500,000 at 60 eV. Fourier-transform spectrometers, already used successfully in the infrared and ultraviolet regions, utilize a two-beam interferometer to measure the Fourier transform of the desired spectrum. The ALS is building a system to extend this technique into the soft x-ray region up to 100 eV. This would allow the principle of Fourier-transform spectrometry (FTS) to be applied to soft x-ray photoabsorption experiments with the goal of obtaining much higher resolution than is possible with a diffraction grating spectrometer.

The principle of FTS is to split a beam into two parts, delay the beam in one path relative to that in the other, then recombine the beams and measure the intensity of the reconstituted beam as a function of the delay. As with all spectroscopic instruments, the resolving power of FTS is essentially equal to the number of waves of path difference between the rays of greatest and least path through the instrument. The ALS design provides for a maximum path difference of 1 cm, corresponding to a potential resolving power of 500,000 at 60 eV. Commissioning of the soft x-ray Fourier transform spectrometer on Beamline 9.3.2 is scheduled for late 1995.



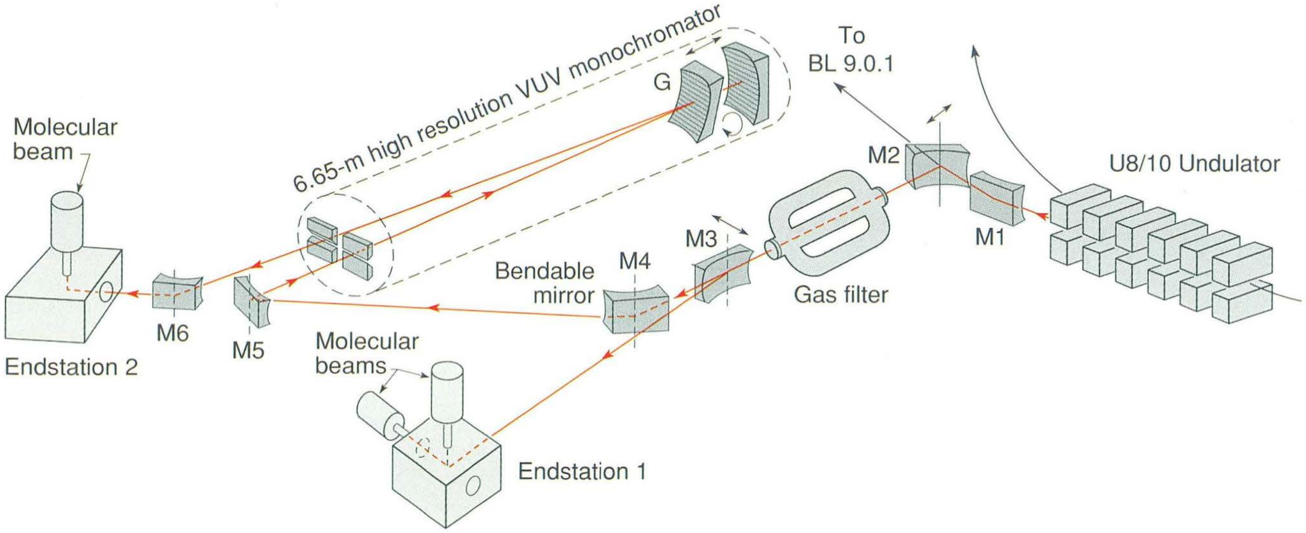
In an experiment using the soft x-ray Fourier-transform spectrometer, a sample in a gas cell is illuminated by a soft x-ray beam with a bandwidth of 0.1–1% from a low-resolution monochromator. The beam is split into two parts using a transmission-grating beamsplitter, and the optical path difference (x) between the upper and lower paths is varied by moving the table carrying the mirrors in the direction shown, the intensity (I) of the recombined beam at the detector is then recorded as a function of x. The absorption spectrum of the gas in the cell is finally obtained as the Fourier transform of I(x).

The achievement of high resolution is contingent upon the ability of the mechanical system to deliver 15 mm of motion while keeping tilts of the interfering wavefronts relative to each other below 1 µrad. To meet this stringent performance criterion, ALS designers had to ensure that all the optical surfaces would be flat within such a tolerance and that the linear motion machine would provide smooth movement without unintended rotations. To keep the latter below the required 1 µrad, a new type of flexural hinge was developed which allowed the machine to be built as a monolithic structure.



ALS-designed endstation has the potential to boost resolving power for photoabsorption spectroscopy to 500,000 using Fourier transform spectroscopy, a technique new to the soft-x-ray regime (see "Challenging the Limits," p. 9).

Opportunities for molecular science at the ALS will expand in 1995 as chemists inaugurate a new beamline to study the dynamics of elementary chemical processes. The chemical dynamics beamline will be equipped with special molecular-beam endstations and a selection of advanced infrared, visible, and ultraviolet lasers to complement the vacuum-ultraviolet beams of the ALS. Scientists at the beamline will probe the fundamentals of chemical processes including the reactions and photochemistry of transient radicals, important intermediates in most chemical systems. They will also investigate the possibility of selectively driving reactions to produce particular product molecules (bond-selective photochemistry). The energy and environmental payoff of this research, from more efficient combustion processes for example, could be immense.



Schematic of the chemical dynamics beamline, excluding lasers. The off-plane Eagle monochromator in Branchline 2 will provide resolving powers between 50,000 and 100,000. A time-sharing arrangement allows undulator light to reach Beamline 9.0.1 or either branch of Beamline 9.0.2, depending on the positions of mirrors M2 and M3.

Health & Environment

From the sub-cellular level to the planetary scale, information on how biological systems operate is essential to scientists fighting human disease and environmental degradation. Experiments performed using synchrotron light complement and extend the knowledge gained using visible-light and electron microscopes as well as laboratory x-ray sources, offering new opportunities in biological, environmental, and pharmaceutical research.

Scientists can use x rays to gain information on a variety of size scales, from the macroscopic to the molecular. For biologists working at the cellular level, experiments using x-ray zone-plate microscopy offer a means of moving beyond the resolution offered by visible-light microscopy. The first such investigations at the ALS have examined the structure of red blood cells infected with the malaria parasite and chromatin packing in the cell nucleus. Organization into chromatin is what allows billions of DNA base pairs to fit into a cell nucleus only microns in diameter, and learning the higher-order structure of chromatin has implications for the study of how genetic material functions in living organisms. Crucial for these cellular-scale studies is the ability to use samples much less elaborately prepared than is necessary for electron microscopy, often viewing cells in aqueous environments or without thin-sectioning or staining.

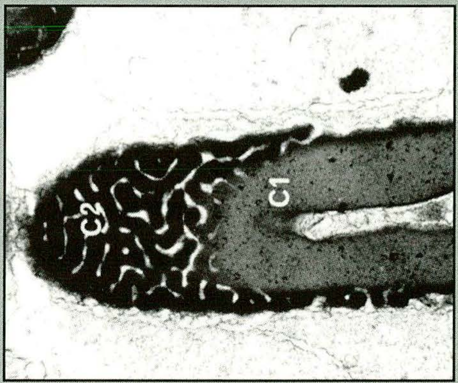
Structural biologists working on the molecular scale will gain a powerful tool at the ALS in 1996, when the protein crystallography beamline begins operation (see “Protein

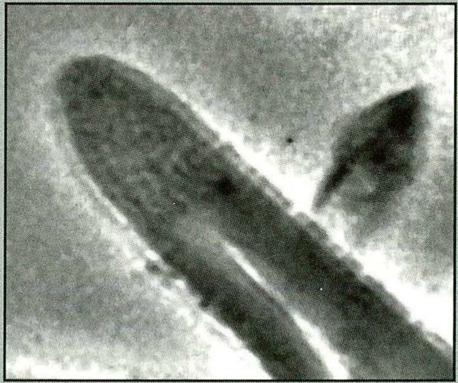
X-Ray Microscope Confirms Chromatin Observations

Images of chromatin in sperm cells of marsupial mice (*Sminthopsis*), taken using transmission electron microscopy (top) and x-ray microscopy (bottom). Researchers studying chromatin distribution had observed an inhomogeneity in the appearance of the chromatin in the transmission electron microscopy (TEM) image, but questioned whether this graininess could be an artifact of the staining process required to prepare the sample.

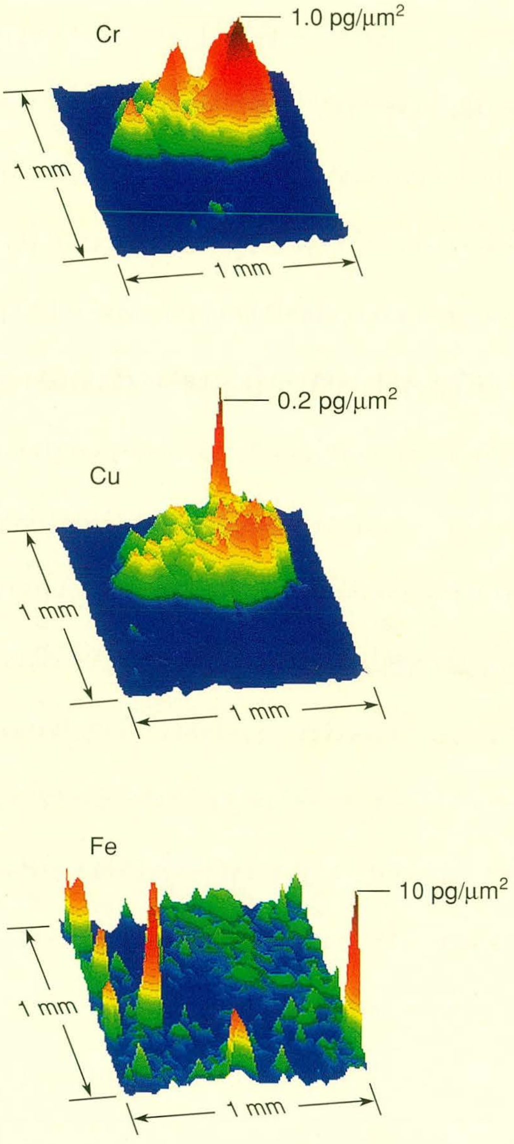
To answer this question, the researchers took advantage of the unique ability of x-ray microscopy to image thick (up to 10 μm) samples under conditions approximating their natural environments, without staining or thin-sectioning. The x-ray image showed the same grainy pattern, confirming that it is primarily related to chromatin mass distribution. The researchers can now work to explain the causes and consequences of this arrangement of chromatin, with the confidence that they are investigating a biological phenomenon rather than an experimental artifact.

TEM image from a study conducted by scientists at Lawrence Livermore National Laboratory and University of Adelaide (Australia). X-ray microscopy image taken on ALS Beamline 6.1 using the XM-1 microscope developed by LBNL's Center for X-Ray Optics.





Microprobe Characterizes Environmental Contamination



Photon energy in = 10 keV
Step size = 25 µm

Elemental maps showing concentrations of various heavy metals in a sediment sample taken from a contaminated wetland site. Sediment-contamination models often assume that pollutants precipitating from the water will coat sediment particles evenly. Here, however, the metals have precipitated heterogeneously, exposing less surface area, and are thus likely to re-dissolve into the environment more slowly than might otherwise be expected. This research improves the understanding of metal contaminants cycling in sediment environments, and therefore may help improve remediation efforts for these contaminants.

At the fluorescence microprobe used in this experiment, a high flux of hard x rays illuminates a spot less than 2 µm across, allowing detection of elemental concentrations of a few femtograms (1 femtogram = 10^{-15} g) per square micron area. All elements from potassium to zinc are detected simultaneously, with sensitivity to bulk as well as surface concentrations. These abilities make the microprobe a useful tool for applications in diverse fields of materials research including industrial quality control, archaeology, and forensics as well as environmental science.

Data taken on the LBNL Center for X-Ray Optics' hard x-ray fluorescence microprobe at Beamline 10.3.1, using a sample from the LBNL Earth Sciences Division.

Crystallography Beamline," p. 14). Protein crystallography, which requires a tightly focused beam of extremely bright x rays for high resolution and rapid sample turnaround, can reveal the molecular structures of viral protein coats, enzymes, and other biological macromolecules. Knowledge of the structures of these molecules is essential to the understanding of their biological function. Furthermore, pharmaceutical researchers can use this information to accelerate the pace of drug development, designing therapeutic agents specifically for the structures of the molecules with which they must interact.

Two other lines of research using ALS light show promise for studies of environmental degradation and remediation. The first uses x-ray fluorescence to perform elemental analysis, with sensitivity to as little as 10^{-15} g of an element in a micron-scale spot. This technique has been used to study the deposition of heavy metals in wetland areas, helping those focusing on environmental restoration to characterize the contamination and form strategies for its removal. The second avenue of research uses photoemission to provide spatial maps, with both elemental and chemical specificity, of minute radioactive samples. These experiments, with possible applications to nuclear waste monitoring and cleanup, require only micrograms of the material under study because of the brightness of the beam illuminating the sample. Radioactive material in amounts this small is relatively safe to handle because of its low total activity.

Research Techniques, 1994–1996

	Beamlines
• Fluorescence microscopy	10.3.1
• Fluorescence spectroscopy	4.0.1*, 9.3.1*
• Protein crystallography	5.0*
• Spectromicroscopy	6.1.2*, 7.0.1
• X-ray transmission microscopy	6.1.2
• X-ray holography	7.0.2*

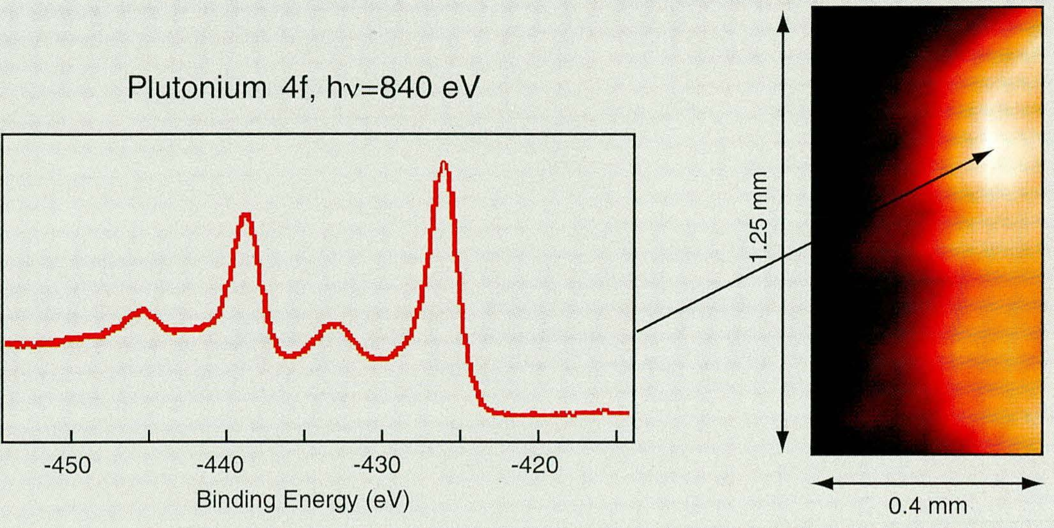
*Techniques planned for use in 1995–1996

Micro-Analysis of Actinide Samples

Elemental map and spectrum taken from a 4-microgram plutonium sample, demonstrating the wealth of information available from minute radioactive samples with low total activity (16 nanocuries for this sample). The image on the right gives the concentration of plutonium in a selected area of the sample. A high-resolution plutonium 4f photoemission spectrum, taken from the bright spot indicated on the elemental map, is shown on the left.

This series of actinide experiments, using synchrotron-based ultra-ESCA, has produced elemental maps and photoemission spectra from microgram samples of curium-248 and plutonium-242 (as curium oxide and plutonium oxide, deposited non-uniformly on platinum substrates). The researchers have taken plutonium spectra from the 4d and 4f core levels as well as the valence bands. A curium 4d ($N_{IV,V}$) absorption spectrum from these studies is the first absorption spectrum taken from a transuranic material in the soft x-ray regime.

Research performed at Beamline 7.0.1's ultra-ESCA endstation by a research group including scientists from University of Wisconsin-Milwaukee, University of Oregon, and LBNL.



Crystallography Beamline Gains Substantial Funding

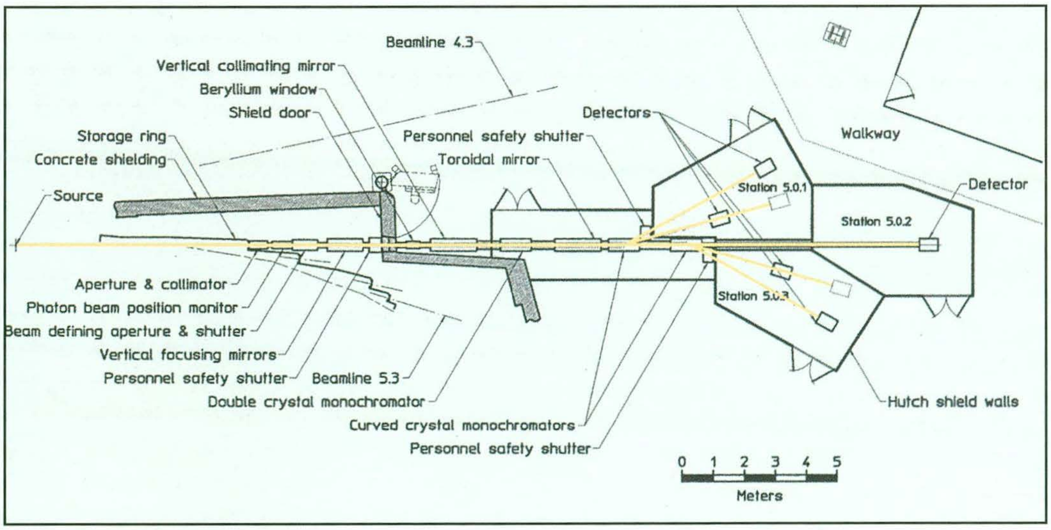
The high technical and scientific marks received by the ALS protein crystallography facility proposal during its extensive review process led to a commitment in November of $3.85M in capital and $250k per year in operating funds by the Department of Energy's Office of Health and Environmental Research (OHER). These funds, along with $500k from the University of California committed by LBNL Director Charles Shank, assure that a beamline with at least one branchline and endstation (of the three that are planned) will be built. The first endstation is scheduled to begin operation in May 1996; additional funding is now anticipated from West Coast biotechnology and pharmaceutical companies to build another endstation, based on their expressed interest.

The ALS Protein Crystallography Facility will offer a choice of crystallographic techniques with semi-automated operation and rapid sample turnaround, making it fully competitive with the best synchrotron sources in the United States. Its prime location guarantees a large base of potential users, including the west coast biotechnology industry, LBNL's Structural Biology and Life Sciences Divisions, and the crystallography groups at University of California Berkeley and UC San Francisco. The performance and reliability of the ALS, combined with the expertise and support facilities on site and nearby, offer the possibility for unprecedented productivity.

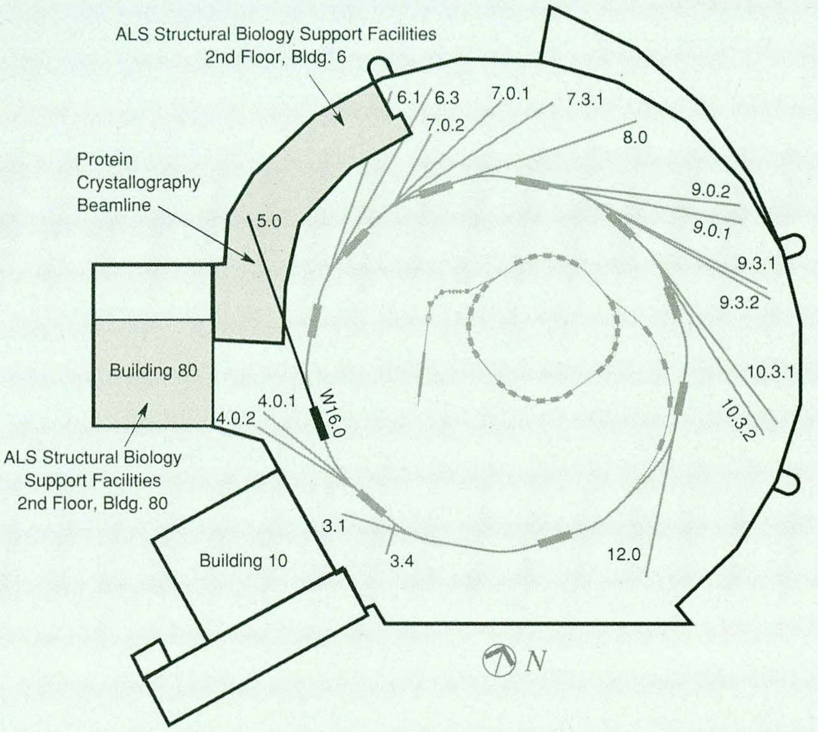
Facility Overview

Plans for the crystallography facility call for a multipole wiggler beamline with three automated endstations. The 38-pole, 2-tesla wiggler source will provide three distinct advantages over conventional x-ray sources for crystallography. First, its high flux (photons per second) will speed data collection by increasing data count rates, especially when the ALS operates at 1.9-GeV electron energy. Second, the wiggler radiation's high degree of collimation will make it possible to resolve diffraction spots from large unit cells (when matched with appropriate detectors) and to work with micro-crystals. Third, the wiggler provides x rays over a continuous synchrotron radiation spectrum (wavelengths planned for use are 0.9–3.0 angstroms). Broad bands of this spectrum can be used for time-resolved Laue diffraction, or precise tuning within the spectrum can facilitate multiple-wavelength anomalous diffraction (MAD) techniques.

The three planned endstations will primarily be used simultaneously. The central station (first to be



Schematic layout of the ALS protein crystallography beamline, including optical components and x-ray hutches. The two side-station detectors are shown at their positions for maximum and minimum x-ray wavelengths.



The Structural Biology Support Facilities will be located on the second floor of the ALS building above the crystallography beamline, and in the adjoining Building 80.

constructed) will offer monochromatic crystallography capability as well as rapid tunability for MAD phasing, and a white-light mode for Laue diffraction. This station will receive the on-axis, brightest portion of the wiggler light, while the two side stations will use off-axis light for monochromatic crystallography. Alternatively, in a time-sharing mode of operation, the side-station optics can be translated to intercept the brightest, on-axis light.

When the useful photon flux is as high as it will be in the crystallography beamline, data recording times become so short that detector readout time can cause a serious delay in the experimental process. The ALS facility will address this by using state-of-the-art matrix CCD detectors that have readout times of about 1.7 seconds, but which do not sacrifice other desirable qualities such as high quantum efficiency, high dynamic range, and small point-spread function. Since CCDs acquire an image of a crystal's diffraction pattern over a given exposure time, they are best suited to studying macromolecules in steady states. To study processes dynamically, a "pixel" detector is being developed for Laue diffraction at the central endstation. Individual counting electronics behind each pixel in this detector will allow continuous monitoring of time-dependent processes, without the time-averaging inherent in CCD and photographic-plate technologies.

Additional Support Facilities

The ALS Structural Biology Support Facilities, a fully funded $7.9M Department of Energy project scheduled for October 1996 completion, will provide an essential part of the infrastructure for the protein crystallography facility. Designed for ease of use and located directly adjacent to the beamline, the support facilities will provide space and equipment for laboratory and computing work to users of the crystallography beamline. Researchers will have access to equipment for biochemistry, spectroscopy, crystallography setup and testing, and computerized data processing with graphics capabilities.

Materials & Surface Science

The simple statement—structure determines function—expresses the fundamental importance of materials research, which strives to explore the structures of materials for an immense variety of applications in fields from medicine to microelectronics. The ALS, with its high-brightness beams, is uniquely equipped for new experiments linking structure to numerous properties of materials—thermal, optical, magnetic, electrical, and physical. Armed with an understanding of these links, scientists and engineers can manipulate structures to produce materials with specific, desired characteristics.

The key elements of structure that determine the characteristics of materials include local chemical properties, degrees and types of order, spin (magnetic) properties, and inhomogeneities. Local properties, such as the atomic bonding geometry or chemical states of a material, influence both the characteristics of the bulk material and its surface interactions with other substances. Information on local properties is accessible by a variety of x-ray-based techniques; those based on electron emission are effective surface probes, while those based on photon emission (fluorescence) can probe the bulk of a material, investigating subsurface layers and buried features. Both types of spectroscopy benefit greatly from the high brightness of the ALS, which allows spectra to be taken more quickly, with higher resolution and signal-to-noise ratios than with other sources. This is especially important for fluorescence techniques, because fluorescence is a low-yield process in the soft x-ray regime—typically, less than 1% of the incoming photons absorbed by a material excite fluorescence, while the rest lead to electron-emitting modes of decay.

Research Techniques, 1994–1996

	Beamlines
• Coherent scattering	7.0.2*
• Fluorescence microscopy	4.0.2*, 7.0.1*, 10.3.1
• Fluorescence spectroscopy	4.0.1*, 7.0.1, 8.0, 9.3.1*, 9.3.2*
• Infrared microscopy	3.4*
• Infrared spectroscopy	3.4*
• Magnetic microscopy	4.0.2*, 7.3.1*
• Magnetic spectroscopy	
• Faraday rotation	6.3.1*, 6.3.2, 7.0.1*
• Magnetic circular dichroism	4.0.1*, 4.0.2*, 7.3.1*, 9.3.2*
• Micro-diffraction	10.3.1*
• Photoabsorption microscopy	7.0.1*, 7.3.1*
• Photoabsorption spectroscopy (EXAFS, NEXAFS, XANES)	6.3.1*, 6.3.2, 9.3.2
• Photoelectron microscopy	4.0.2*, 7.0.1*, 7.3.1*, 12.0*
• Photoelectron spectroscopy	
• Angle-resolved valence spectroscopy	4.0.1*, 7.0.1, 9.3.2
• Core-level spectroscopy (ESCA, XPS)	4.0.1*, 7.0.1, 8.0, 9.3.1*, 9.3.2
• Diffraction/holography	4.0.1*, 7.0.1, 8.0, 9.3.1*, 9.3.2
• Soft x-ray interferometry	6.1.2*
• Spectromicroscopy	6.1.2*, 6.3.1*, 6.3.2*, 7.0.1, 7.3.1*, 12.0*
• Total reflection x-ray fluorescence	10.3.2*

*Techniques planned for use in 1995-1996

Today's Materials Science—Tomorrow's Computing Technology

The microelectronics industry has maintained a remarkable record of expanding the ratio of computing power to cost during the past 35 years. Memory chips, for example, have doubled in capacity every 1.5 years without a significant increase in price. The potential role of synchrotron radiation facilities like the ALS in continuing this pace of development is recognized by the semiconductor industry and detailed in their *National Technology Roadmap for Semiconductors* (1994), a plan for maintaining the competitive pace of U.S. industry in this global market.

U.S. semiconductor companies, now producing integrated circuits (ICs) with a minimum feature size of about 0.35 microns, aim to reduce that figure to 0.10 microns in less than 10 years. The decreasing minimum feature scale creates three major problem areas which research at the ALS can help to address:

Modification of material properties. As materials used in fabrication are confined to ever smaller areas, their electrical properties appear to change. Using spectromicroscopy, scientists at the ALS are attempting to understand why this change occurs; for example, whether it is due to a crystallographic change or a change in electronic structure due to confinement.

Reliability. Faultless fabrication of the electrical interconnects in ICs is a major challenge for reliable operation. Smaller gates mean smaller interconnects and higher current densities. As a high density of electron current flows through the aluminum wire conductor circuits, some of the electron momentum transfers to the aluminum atoms, and the atoms are moved in the direction of the current (electromigration). This flow results in the formation of defects which can become mobile and coalesce to form voids, that can then enlarge and break the interconnects. At the ALS, we are developing the technique of micro-diffraction to provide data on the relationship between strain and current density. This information will be used to model the basic process of electromigration voiding.

Contamination. Decreasing scale in IC manufacture demands lower levels of contaminants which impair IC function. These can be in the form of relatively large (0.1 micron) particles or dispersed material. Improved wafer cleaning and handling can accomplish the needed decreases in contamination, but they must be complemented by measurement techniques. In the case of ultra-trace amounts of contaminants dispersed on the silicon surface, these techniques must resolve elemental species, must be surface-sensitive, and must detect extremely small amounts of contaminants: 10^9 atoms/cm^2 is the current state of the art using high-power rotating-anode x-ray sources. The Semiconductor Roadmap indicates that by the end of this century, detection methods sensitive to 10^7 atoms/cm^2 will be required.

This need for extreme sensitivity is being addressed by the ALS using a variety of techniques. Researchers at the ALS working with Stanford Synchrotron Radiation Laboratory and Silicon Valley companies are doing experiments using synchrotron-based TXRF (total reflection x-ray fluorescence), a technique used to measure very small amounts of dispersed contamination. Other techniques being applied to the problem include the newly developed techniques of spectromicroscopy, such as micro-XANES (x-ray absorption near-edge structure), and spatially resolved micro-ESCA (electron spectroscopy for chemical analysis). Spectromicroscopy can identify small amounts of concentrated submicron-sized particles, and help determine the origin of the contamination in the fabrication process from the particle's chemical signature.

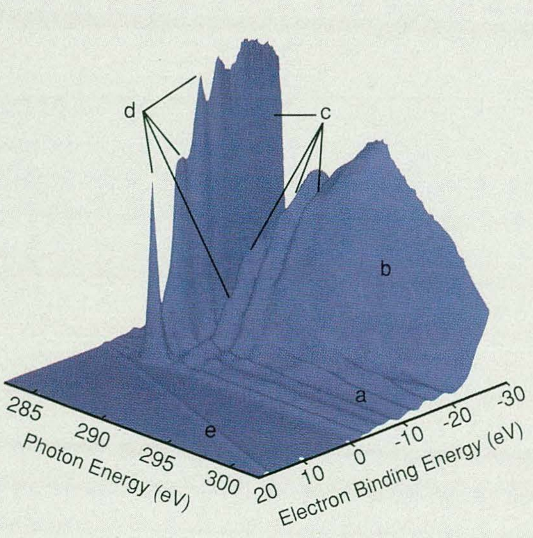
Information on atomic bonding is also available through infrared studies. The ALS produces infrared light at up to 1000 times the brightness of laboratory sources, giving access to weak vibrational spectra that serve as molecular "fingerprints." These molecular signatures can be used to study adsorbates on surfaces, forensic samples such as cocaine residues in hair, and polymer laminates such as color photographic film. Based on industry interest in synchrotron-based infrared techniques, the ALS plans to complete an infrared spectromicroscopy beamline in 1996.

Beyond their localized chemical properties, materials exhibit various degrees of order in their larger-scale organization. Degree of order ranges from perfect long-range order in single crystals, through short-range order in "highly oriented" or polycrystalline materials, to complete disorder in amorphous materials such as glass. In an ordered material, valence electrons take on properties distinct from those in single molecules or disordered materials, forming a band structure which dictates optical, electrical, thermal, and other qualities of the material (see "What Is Band Structure?" on p. 21). Features of this band structure are accessible through photoemission and fluorescence methods, in combination with a tunable synchrotron beam.

Also of interest in many materials are magnetic properties, notable for their applications in the microelectronics industry (see "Today's Materials," p. 17). Phenomena such as magnetic circular dichroism and Faraday rotation, which can be used to measure magnetic characteristics, depend on incoming beams with a high degree of polarization (circular and linear, respectively), which the ALS provides. Highly polarized beams are also used to study molecules with high symmetry, such as benzenes or "buckyballs" (spherical C_{60} molecules), probing the

Resonant Photoemission in Polymers

High photon flux, and thus high photoelectron yield, allowed the 101 poly(α-methylstyrene) spectra composing this surface plot to be taken in only 30 seconds each. The spectra, taken with 0.1 eV electron energy resolution at photon energies 0.2 eV apart, represent a far more richly detailed data set obtained in less time than previous studies of this type.



The bumps (d) on the plot specify the pairs of unoccupied and valence states involved in participant decay in the sample (for definitions of participant decay and other terms, see "What Are?". Because the energy transfers in participant decay are governed by rules of symmetry, localization, and/or spatial distribution, the plot can be interpreted to provide information on all these orbital attributes. Other features on the plot result from direct photoemission (a), normal Auger decay (b), spectator decay (ridges, c), and C 1s emission due to the second undulator harmonic (e). Researchers are already using the plots generated in these experiments to gain insight into polymer decay mechanisms and valence states, as the detailed data sets bring subtle features to light.

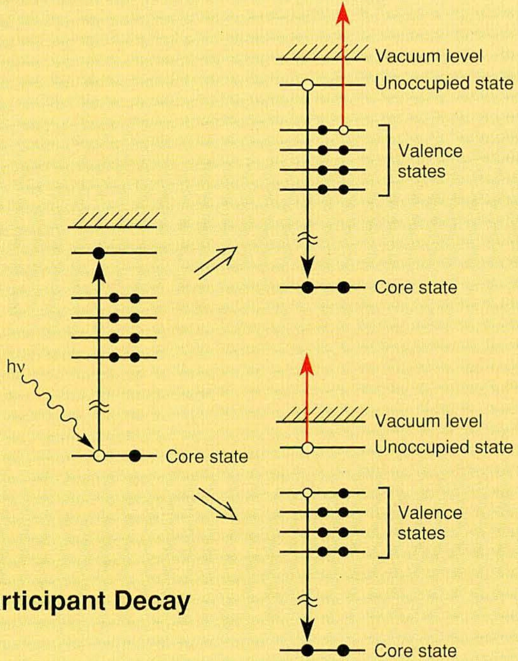
Experiment performed at the Beamline 7.0.1 ultra-ESCA endstation by scientists from University of Wisconsin-Milwaukee, University of Oregon, and LBNL.

WHAT ARE… PARTICIPANT, SPECTATOR, AND AUGER DECAYS?

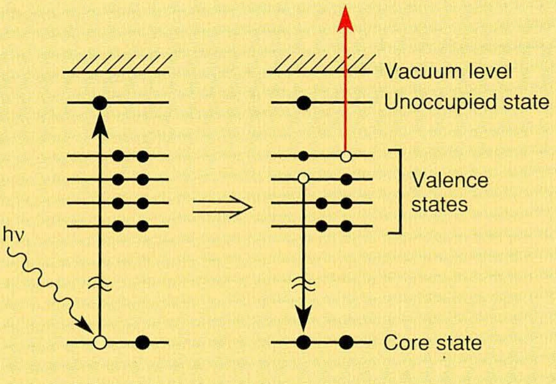
Participant decay and spectator decay are two processes an atom can undergo after one of its core electrons has been excited to an unoccupied state below the energy (called the vacuum level) required for the electron to escape from the solid. This definition of vacuum level, and several of the mechanisms explained below, assume that the atom in question is near the surface of the solid; electrons escaping from atoms deep within a solid are generally recaptured by interactions with other atoms before they break free of the solid's surface.

Participant decay involves the excited core electron and a valence electron; one of these immediately fills the empty core state and transfers its excess energy to the other, which is excited above the vacuum level and detected as photoemission (the red arrow in each diagram represents photoemission). The core-electron excitation and participant decay occur as a one-step process commonly called resonant photoemission, which because of its one-step nature can reveal information about the quantum states involved in the excitation and decay.

Spectator decay is an associated one-step process in which the excited core electron "watches" while one valence electron drops to fill the core hole and transfers energy to a second valence electron, which escapes and is detected as photoemission. Auger emission, a common non-resonant decay process to which participant and spectator decay are often compared, occurs when an initial excitation of a core electron above the vacuum level is followed by the same two-valence-electron decay process as in spectator decay. The absence of the spectator electron, and the fact that Auger emission is a two-step (decoupled) process, distinguish it from spectator decay.



Participant Decay



Spectator Decay

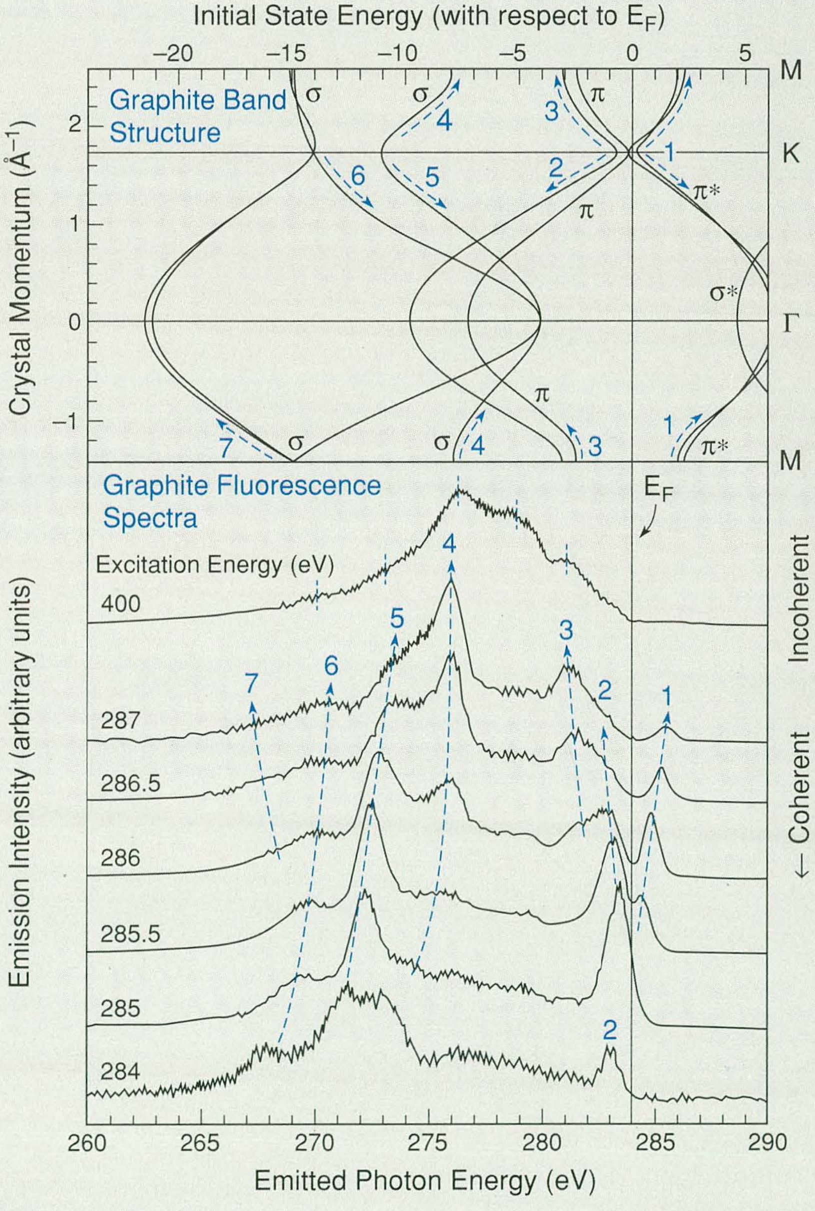
A New Way to Probe Band Structure

Resonant soft x-ray fluorescence spectra (lower panel) and calculated band spectra (upper panel) for graphite, with arrows showing the correlations between the two. These correlations form the basis for a new method of examining band structure in polycrystalline samples.

Experiment performed by scientists from Lawrence Livermore National Laboratory and University of Tennessee, working at the University of Tennessee soft x-ray emission spectrometer endstation on Beamline 8.0.1.

Band structure, a key predictor of a crystalline material's behavior, has previously been explored primarily using photoemission (see "What Is?"). Now it is possible to probe band structures with polycrystalline samples using resonant soft x-ray fluorescence, given a highly efficient spectrometer and an intense, bright source like the ALS. These reduce the time necessary to take fluorescence spectra by roughly an order of magnitude compared to second-generation x-ray sources.

In the fluorescence spectra shown, taken from highly oriented pyrolytic graphite, gradual displacements of the resonant fluorescence peaks with increasing excitation energy are labeled 1–7 with arrows. These arrows correspond to those in the upper panel, which shows the theoretically calculated band structure for graphite. M, K, and Γ refer to points of symmetry in the band structure, and E_F is the Fermi energy.



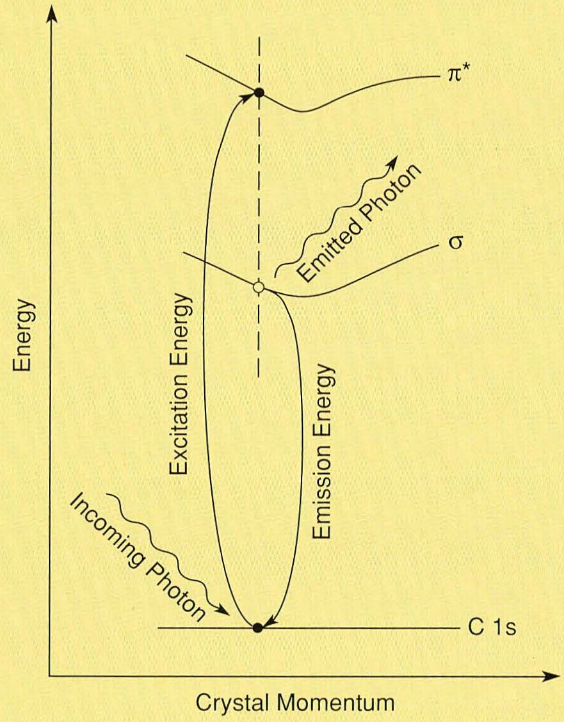
molecular orbitals and thus providing insight into the behavior of these materials.

The single most exciting advance made possible by the ALS's high brightness is the ability to add spatial resolution to all of the spectroscopic techniques described above, forming a class of techniques known as spectromicroscopy. This allows for the exploration of materials whose properties vary from one part of the sample to another, since high-brightness, low-emittance beams can direct enough photons into a small area of a sample to generate appreciable signal in scanning or imaging modes. This microfocusing capability is used in some experiments to look at the properties of individual

WHAT IS... BAND STRUCTURE?

The starting point for determining many of a material's properties (e.g., electrical and thermal conductivity, specific heat, and optical reflectivity) is its band structure. In a solid with an ordered arrangement of atoms (crystalline material), tightly bound core electrons are localized around atomic nuclei. The more loosely bound valence electrons that participate in chemical bonding, however, are not tied to any particular atom; instead they roam throughout the solid. They are characterized by a kind of momentum (crystal momentum) and an energy associated with each crystal momentum vector. Moreover, the energies of the valence states, which are quite discrete in individual atoms, broaden into sets of quasi-continuous bands in a solid, giving rise to the term *band structure* to describe the functional dependence of energy on crystal momentum.

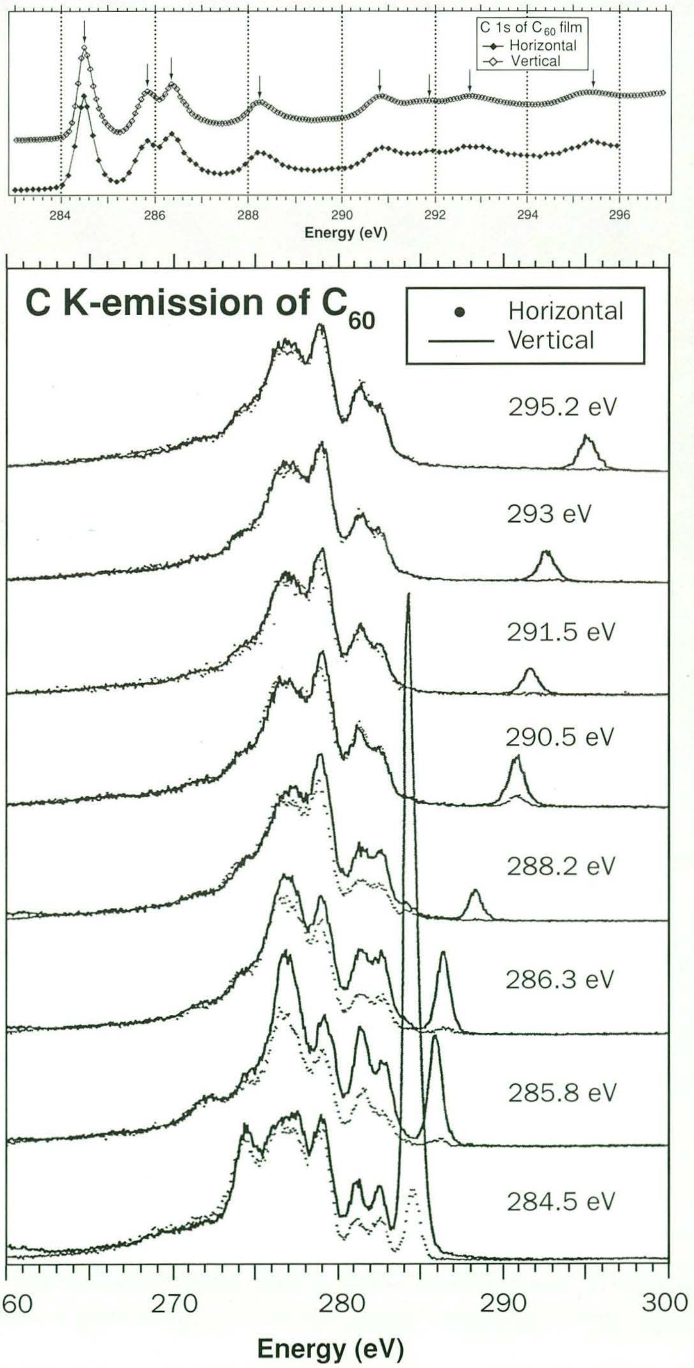
The figure at right shows why resonant fluorescence peaks shift in accordance with band structure. When a core electron is excited with a given excitation energy, the band structure determines the possible value(s) of crystal momentum (shown as horizontal position in the figure) for the electron's excited state. The electron that drops to fill the core hole has the same crystal momentum as the excited electron (dashed vertical line). A slightly lower excitation energy in this case would result in a lower emission energy, representing a shift of the same type as that labeled by arrow #4 in the figure accompanying "A New Way."



microcrystals in a polycrystalline material, where a macroscopic experiment would average out information from a large area. In other experiments, researchers may examine the chemical environment of local areas of materials. This is particularly productive with semiconductors, where the properties of potential fabrication materials can change when they are spatially confined on a fine-scale integrated circuit. Semiconductors are also prone to failure because of surface defects; one example is surface contamination resulting from problems with the fabrication process. Spectromicroscopy can identify these contaminants, helping manufacturers pinpoint the source of the contamination.

Soft X-Ray Fluorescence of Buckyballs

Fluorescence spectra (lower panel) from C_{60} "buckyballs," taken at photon energies corresponding to peaks in the C_{60} absorption spectrum (top). The high brightness and high energy resolution of the incoming beam were essential for producing a fluorescent signal strong enough to yield clear spectra and for resolving peaks due to orbitals with closely spaced energies.



The fluorescence transitions represented in these spectra are governed by selection rules based on the angular momenta and orbital symmetries of the molecular states involved. It is because of these selection rules that spectra taken with incoming photon energies near C_{60}'s ionization threshold (290.5 eV) differ so much from each other. Comparison of the data with *ab initio* theoretical calculations shows that the near-threshold spectra result from a one-step (resonant) fluorescence process; even an assumption of a two-step process (excitation followed by decay with fluorescence) with full selection rules in effect would not match these results. The distinction between one-step and two-step processes becomes even more clear when theoretical calculations are compared with observed polarization effects. The researchers observed polarization effects by placing the spectrometer in vertical and horizontal positions relative to the sample, using horizontally polarized incoming photons. At incoming photon energies farther from threshold, the spectrum ceases to change, indicating the dominance of non-resonant processes.

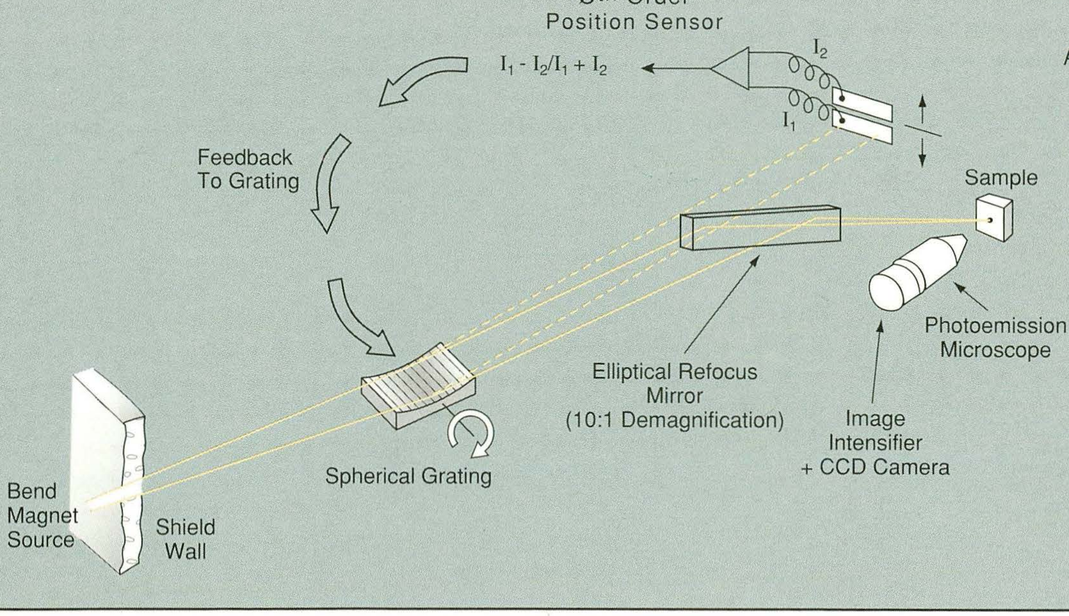
Experiment performed by researchers from Uppsala University, Sweden, using the soft x-ray fluorescence endstation at Beamline 7.0.1.

Application-Specific Beamlines—Superior Performance at Lower Cost

Experiments at a synchrotron light source sometimes demand conditions best provided by new, custom-designed beamlines. These new beamlines must be low-cost and quickly constructed to meet the needs of an active research environment. Accordingly, two beamlines now under development at the ALS are application-specific, designed with tightly defined purposes in mind. This approach optimizes performance, increases reliability, and significantly lowers the beamline's cost.

Beamline 7.3.1, designed for full-field photoelectron microscopy of magnetic surfaces, will be one such application-specific beamline. This bend-magnet beamline provides monochromatic illumination of a field of view on a magnetic sample, using right or left circularly polarized light from above or below the orbit plane of the ALS storage ring. The difference in absorption between the two helicities—called the x-ray dichroism—gives an indication of the magnetic state of the material. Imaging soft x-ray dichroism promises wide application in the study of magnetic materials; for example, in elucidating the electronic structure of artificially produced magnetic multilayers which are at the heart of the drive for ultra-high-density data storage media.

The goal of the beamline design is to maximize the photon flux density in the illuminated field of view of the microscope (typically 50 μm). This will allow the moderate spatial resolution of a simple electrostatic photoelectron microscope (2000 Å) to be utilized for experiments requiring fast data collection, or the high spatial resolution of an aberration-corrected photoelectron emission microscope (200 Å) to be fully realized. Beamline 7.3.1 maximizes flux density by directly imaging the source using only a vertically diffracting grating and a horizontal refocusing mirror, producing a monochromatic spot size of less than 40 μm (a feedback mechanism uses filtered "white" light, imaged by a pinhole in the central aperture stop, to compensate for any movement of the electron beam). A single monochromator grating can produce the desired energy range of ~300–1200 eV, covering the absorption edges of interest in magnetic materials. An energy resolution of 0.5 eV is sufficient for magnetic contrast measurements, so the grating can have a low line density (200 lines/mm). Such a grating has a high diffraction efficiency and uses only a small rotation angle to access the desired energy range.



Another application-specific beamline to be constructed at the ALS is Beamline 4.0.2 (magnetic microscopy with an elliptically polarizing undulator source). Beamline 7.3.1 is scheduled for operation in 1996, and 4.0.2 in early 1997.

Lithography

While the uses of synchrotron light are often discussed in terms of what it will allow us to see, there is another side to the story: synchrotron light can be used not only to examine materials, but to shape them. The techniques known as lithography, including the processes used for printing, use light to change the chemical structure of materials called photoresists. When a photoresist that has been exposed to a pattern of light and shadow is developed, usually in a chemical bath, the exposed and unexposed portions of the resist respond differently. Lithography has been used for technological applications with visible and ultraviolet light for some time, and now synchrotron light offers improvements and new opportunities using x rays and extreme ultraviolet (EUV) light. The manufacture of integrated circuits (ICs) for computer chips and of tiny, precise machine parts are two lithographic processes with aspects under development at the ALS.

One group at the ALS is developing a method of evaluating the tools for a new IC production process which could shrink the minimum size of circuit elements, providing for a future phase of IC miniaturization. The current generation of ICs is produced using ultraviolet (UV) lithography, in which the photoresist is a thin layer on a silicon wafer. UV light shines through a mask with absorbing and transmitting regions in the desired circuit pattern, travels through lens optics which demagnify the pattern, and then exposes the photoresist with the mask pattern in miniature. Further processing etches away the exposed photoresist and deposits other materials to produce the finished circuit. Today the minimum feature size for ICs is between 0.35 and 0.6 μm, limited by the wavelengths of UV light used to make the exposure. The IC industry is considering EUV lithography to produce features 0.1 μm across or smaller. The use of EUV light (with wavelengths around 130 Å) allows production of smaller features, but it also necessitates the use of reflective optics, since conventional refractive lenses will not transmit EUV light.

Multilayer mirrors for EUV have been developed which will, in theory, give the needed demagnification. In practice, however, a critical flaw in an EUV optical system can be tiny enough to escape detection by optical interferometry (the current state of the art in optical testing). To provide the needed measures of multilayer optical quality, researchers are developing an EUV interferometer for use in "at-wavelength" testing—testing performed using the wavelength at which the lithography system would operate—using an ALS beamline as their source for bright, spatially coherent EUV light.

Another area of lithography under development at the ALS is deep-etch x-ray lithography: the first step in a process called LIGA (a German acronym for lithography, electroforming, and molding), which can produce machine parts with lateral dimensions in the several-micron range, thicknesses of several hundred microns, and sub-micron precision. Such small, robust parts are called for in

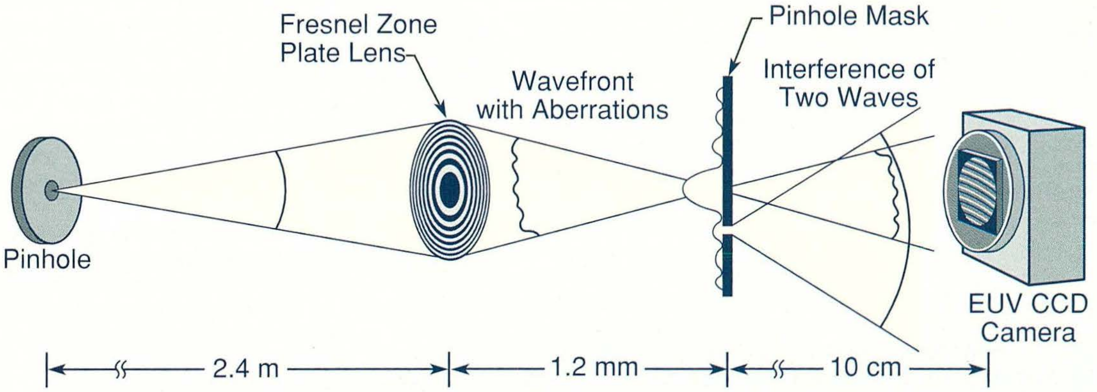
Research Techniques, 1994–1996

	Beamlines
• Deep-etch x-ray lithography (LIGA)	10.3.2
• EUV interferometry	9.0.1, 9.3.2*, 12.0*

*Techniques planned for use in 1995–1996

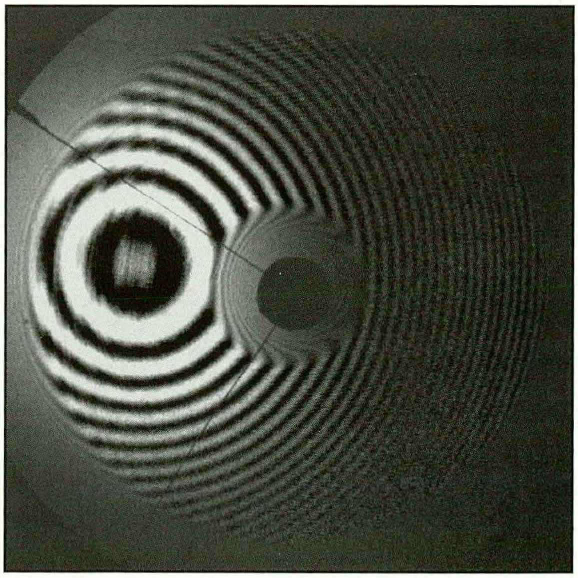
At-Wavelength Testing of EUV Optics

Schematic for point diffraction interferometer (PDI), a newly designed instrument for at-wavelength evaluation of extreme-ultraviolet (EUV) optical components and systems. The PDI was initially used to test a Fresnel zone plate lens (interferogram at lower right); future plans include the evaluation of multilayer mirror sets for EUV lithography, possibly using zone plate lenses to spread the thin undulator beam into a broad cone of light to illuminate these larger optical systems.



The PDI uses spatially coherent EUV light, filtered from an undulator, to illuminate the optical device being tested. The resulting wavefront strikes a thin absorbing membrane containing a tiny (<1500 Å) pinhole. Diffracted light from the pinhole forms a spherical reference wave-front which interferes with the wavefront transmitted through the membrane from the optical system under testing. The resulting interferogram recorded by a CCD camera, is analyzed for aberrations in the optical device. This approach is quite distinct from conventional interferometric optics testing, which compares a component under testing to a well-characterized optical reference surface by using a beam splitter to divide light between the two. The reference and aberrated wavefronts then recombine to form an interference pattern. EUV reference surfaces and beam splitters suitable for this use do not yet exist.

Experiments performed at Beamline 9.0.1, using the point diffraction interferometer, by scientists from LBNL's Center for X-Ray Optics and University of California Berkeley.



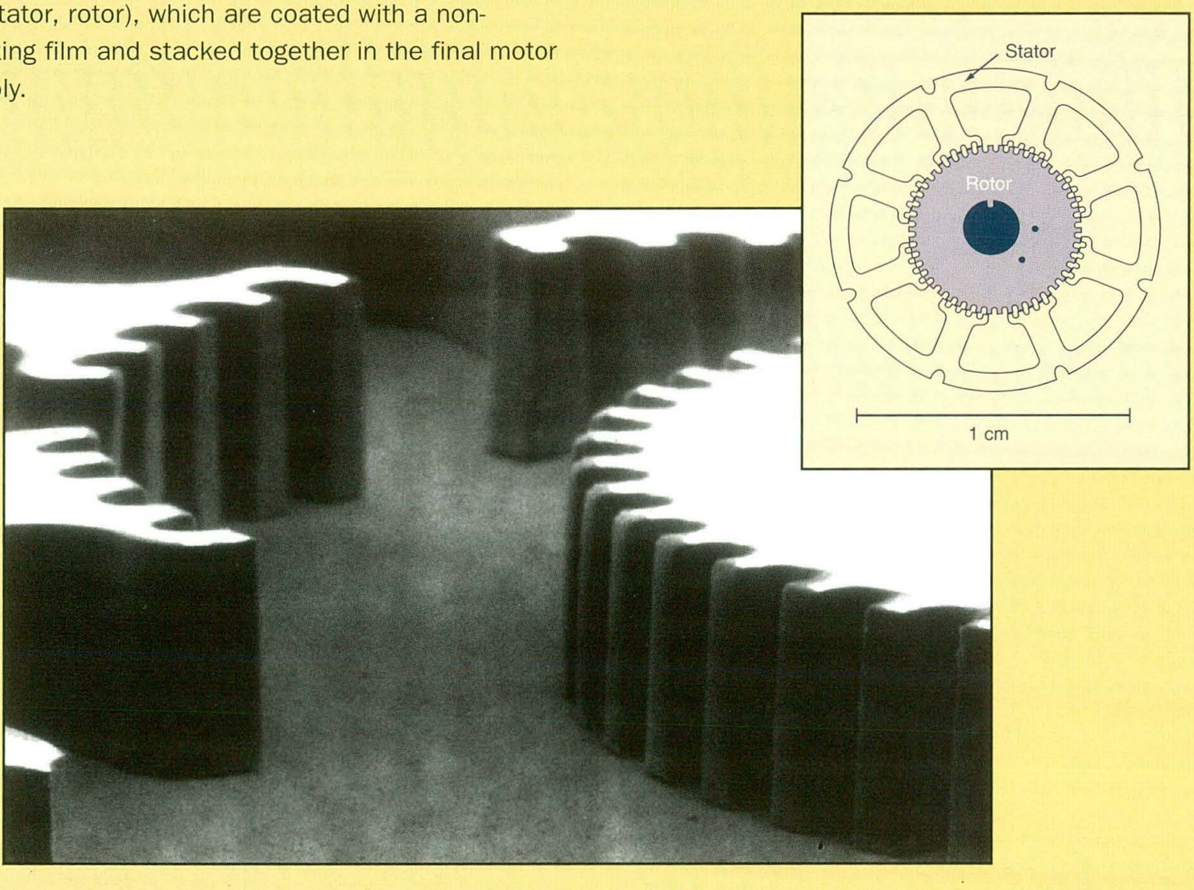
High-Performance Stepper Motor

Micrograph of Plexiglas™ stator and rotor templates for a high-performance stepper motor, fabricated using deep-etch x-ray lithography. The ALS is well-suited as a source for deep-etch x-ray lithography because it produces light with high flux, a high degree of collimation, and suitable wavelengths to penetrate deep resist layers (1 mm in this case). The parts shown represent steps in the development process for the motor, which involves refining the many stages involved in LIGA-based fabrication, e.g., mask making, resist development, x-ray exposure, etc. The final parts will be made of a nickel-iron alloy.

Stepper motors are electromechanical devices which rotate by a discrete step angle when energized electrically. Their key components are a series of metal plates known as stator and rotor laminations (see inset). A single motor requires 50 lamination pairs (stator, rotor), which are coated with a non-conducting film and stacked together in the final motor assembly.

The most common need for small-step-size motors is in instrumentation where the accuracy of the system is critical, such as controlling the angle of the communications antenna on a satellite. Conventional machining techniques are not effective below certain size limits because they lack the precision to produce minute parts with small tolerances; for instance, the width of the rotor teeth in the stepper motor under development is 200 microns, with 5 micron design tolerances. These specifications are well within the abilties of the LIGA process, however. The result is a motor four times smaller than is commercially available, with a step size of only 1.8 degrees.

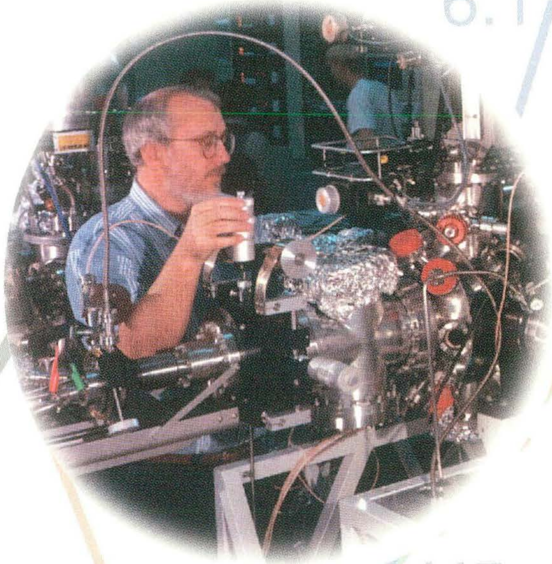
The deep-etch x-ray lithography was performed on the LIGA endstation at Beamline 10.3.2. The LIGA development work involves scientists from LBNL's Center for X-Ray Optics, Sandia National Laboratory (California), Jet Propulsion Laboratory, and Empire Magnetics.

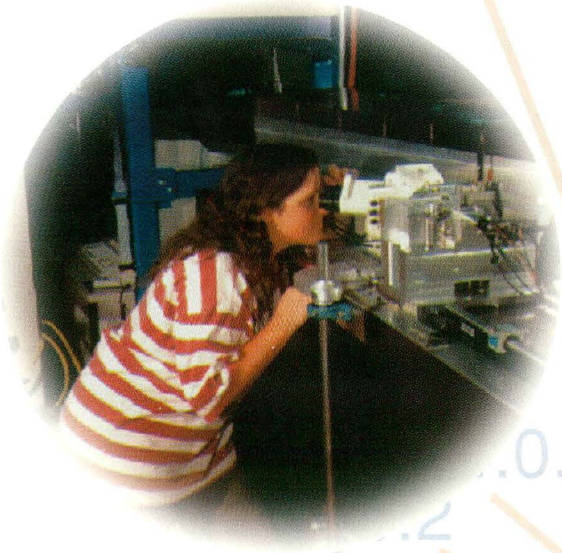


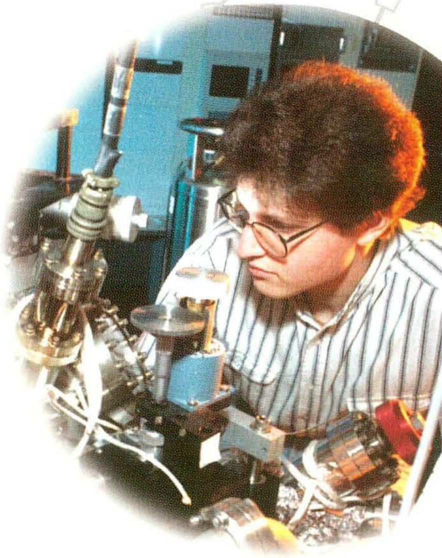
applications including satellites, computer hard disks, and medical and chemical research equipment. Obtaining this degree of precision with conventional machining methods poses immediate problems; for example, die stamping cannot produce rotors with very small teeth because dies sharp enough to stamp out such parts are impractical or impossible to produce.

The LIGA process varies somewhat according to the particular application, but a typical process begins with bonding a layer of Plexiglas™ or similar photoresist to a conductive substrate. This resist is exposed to hard x rays (3–9 keV) shining through a gold-patterned mask, and then it is developed in a chemical bath so that the exposed areas are removed, baring the conductive layer beneath. This conductive layer then becomes the electrode for an electroplating process that fills the gaps in the resist with metal. When the resist is removed, the remaining metal shape is the negative or complement of the resist shape. This metal form may be the final product itself, or it may be used as a mold for a final product of some other material. Developing resist to the required depth of almost a millimeter requires light with two characteristics found at the ALS: a large flux of hard x rays for exposing thick resist, and highly parallel beams to avoid distortion of the mask pattern at deeper levels of the resist.

Using the Advanced Light Source

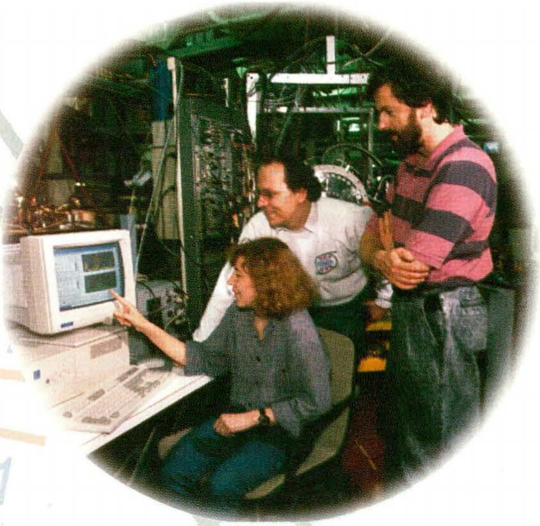


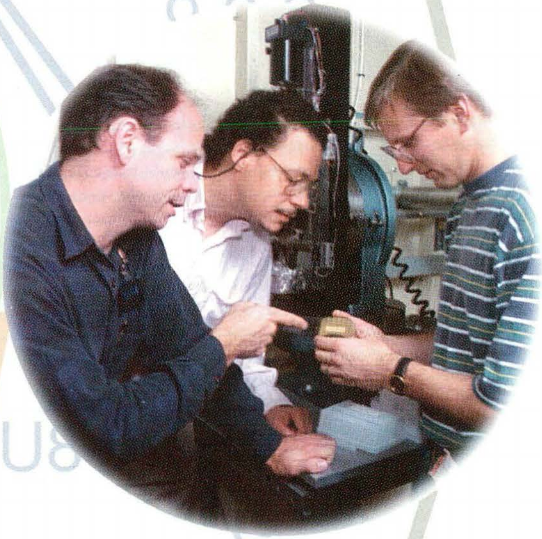




The ALS, a Department of Energy national user facility, welcomes researchers from universities, industry, and government laboratories. Qualified users have access either as members of participating research teams (PRTs) or as independent investigators. PRTs (groups of researchers with related interests from one or more institutions) construct and operate beamlines and have primary responsibility for experiment endstation equipment. They are entitled to a certain percentage of their beamline's operating time according to the resources contributed by the PRT. Through a proposal process, the remaining beamtime is granted to independent investigators, who may provide their own endstation or negotiate access to a PRT owned endstation.

The ALS does not charge users for beam access if their research is non-proprietary. Users performing proprietary research are charged a fee based on cost recovery for





ALS usage. All users are responsible for the day-to-day costs of research (e.g., supplies, phone calls, technical support).

The ALS storage ring is optimized to run at an energy of 1.5 GeV, although it can run from 1 to 1.9 GeV, allowing flexibility for user operations. At 1.5 GeV, the normal maximum operating current is 400 mA in multibunch operation. The spectral range of undulator and wiggler beamlines extends from photon energies of roughly 10 eV to 10 keV. Bend magnets produce radiation from the infrared to about 12 keV.

The ALS is capable of accommodating approximately 46 beamlines and more than 100 endstations. The first user beamlines began operation in October 1993, and there were nine operating beamlines with several more under construction by the end of 1994.

Beamlines 1994–1996

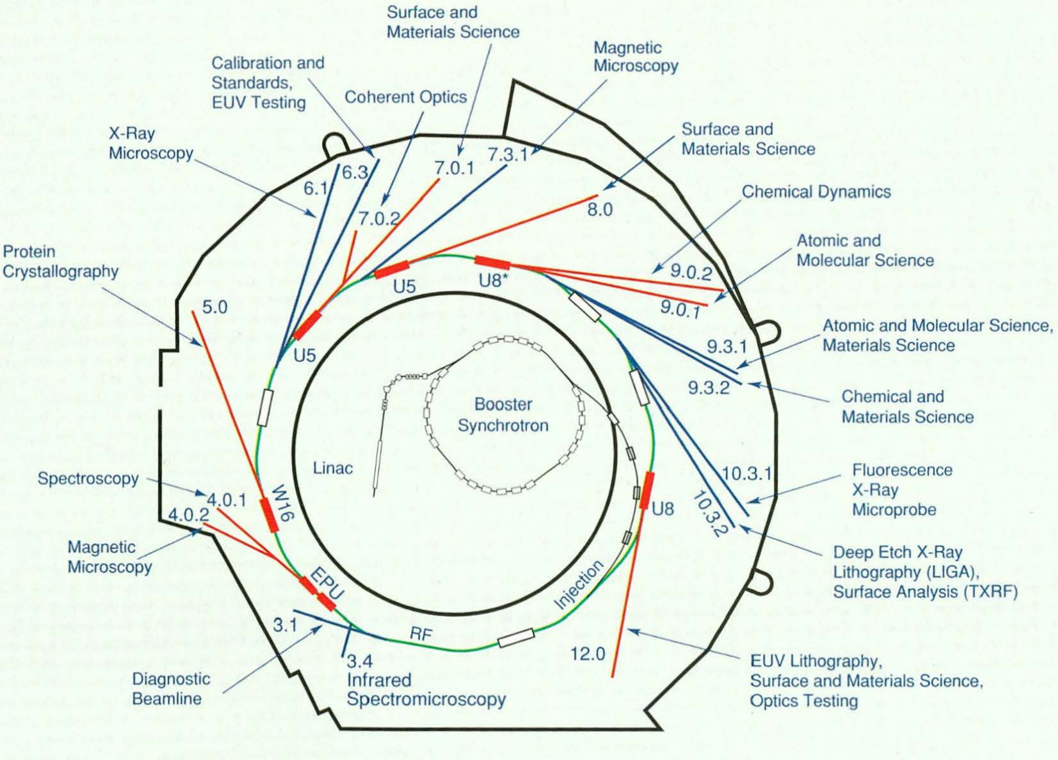
Beamline	Source	Areas of Research	Techniques*	Energy Range	Monochromator	Available
3.1	Bend magnet	Diagnostic beamline		200-280 eV	None	Now
3.4	Bend magnet	Infrared spectromicroscopy	8, 9	0.05-1 eV	FTIR	1996
4.0.1	EPU elliptically polarizing undulator(s)	Spectroscopy	7, 12, 19, 20, 21	20-1800 eV	V-SGM	1996/7
4.0.2		Magnetic microscopy	6, 10, 12, 17	100-1600 eV	SGM	1996/7
5.0	W16 wiggler	Protein crystallography	22	4-13 keV	Double Crystal	1996
6.1.2	Bend magnet	High-resolution zone-plate microscopy	23, 24, 28	250-600 eV	Zone Plate Linear	Now
6.3.1	Bend magnet	Calibration and standards, EUV optics testing	11, 15, 24	500 eV-4 keV	Double Crystal	1995
6.3.2	Bend magnet	Calibration and standards, EUV optics testing	11, 15, 24, 25	50-1000 eV	VLS-PGM	1995
7.0.1	U5 undulator	Surface and materials science, spectromicroscopy	6, 7, 11, 14, 17, 19, 20, 21, 24	60-1000 eV	SGM	Now
7.0.2	U5 undulator	Coherent optics experiments	1, 27	70-650 eV	None	1996
7.3.1	Bend magnet	Magnetic microscopy	10, 12, 14, 17, 24	260-1200 eV	SGM	1996
8.0	U5 undulator	Surface and materials science	7, 20, 21	70-1200 eV	SGM	Now
9.0.1	U8 undulator †	Atomic and molecular science, EUV lithography	2, 5, 7, 15, 16, 18, 25	20-310 eV	SGM	Now
9.0.2	U8 undulator †	Chemical dynamics	2, 3, 16, 18	5-30 eV	White Light Eagle	1995
9.3.1	Bend magnet	Atomic and molecular science, materials science	2, 7, 15, 16, 18, 20, 21, 25	700 eV-6 keV	Double Crystal	1995
9.3.2	Bend magnet	Chemical and materials science	5, 7, 12, 15, 19, 20, 21	30-1500 eV	SGM	Now
10.3.1	Bend magnet	Fluorescence x-ray microprobe	6, 13	3-12 keV	Multilayer	Now
10.3.2	Bend magnet	Deep-etch x-ray lithography (LIGA), surface analysis (TXRF)	4, 26	3-12 keV	White Light	Now
12.0	U8 undulator	EUV lithography, surface and materials science, optics development	5, 17, 24	60-320 eV	VLG-PGM	1996

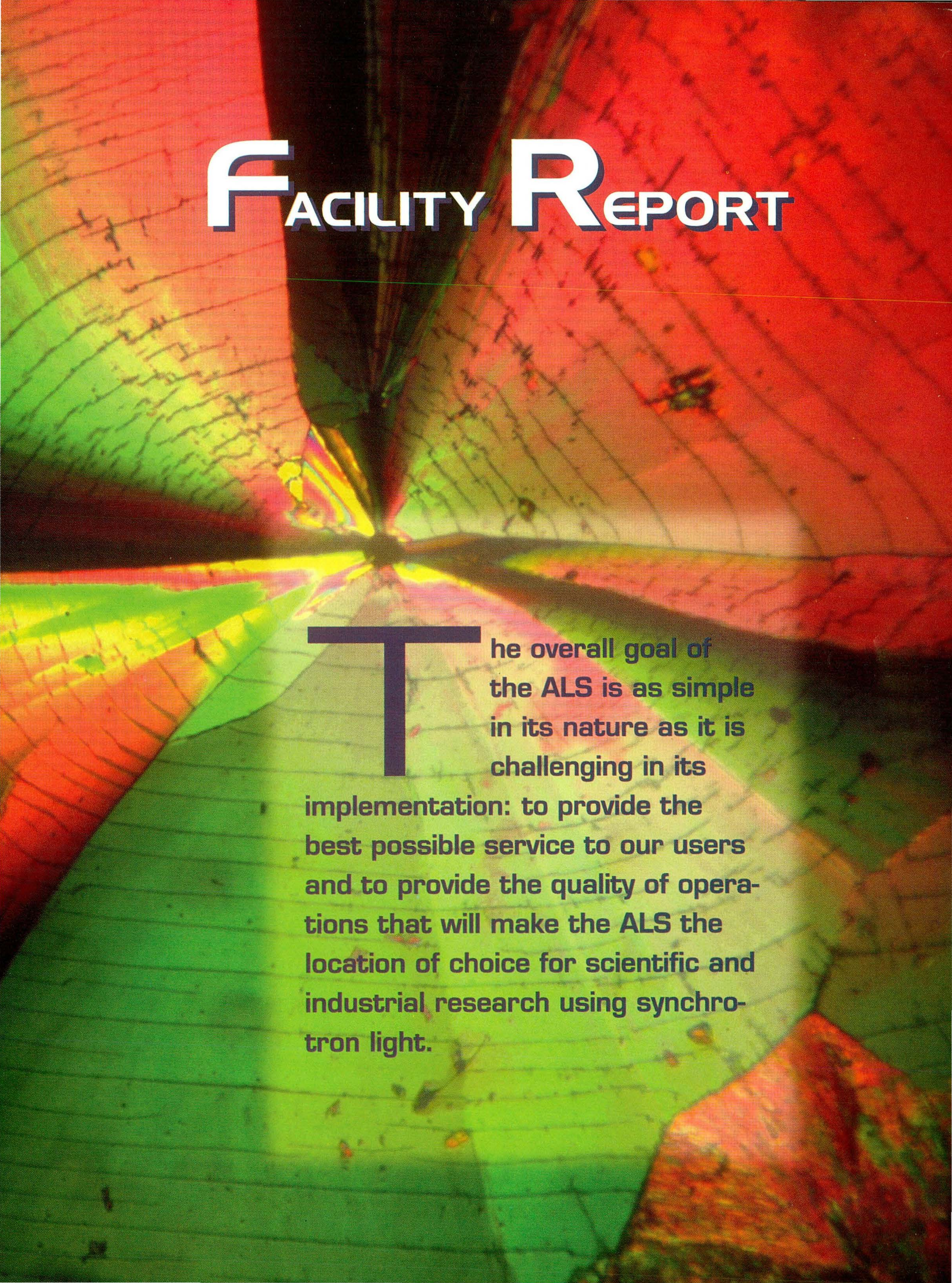
† Will change to U10 in September 1995

*See key at right

Key to Techniques

1	Coherent scattering
2	Coincidence detection
3	Crossed-molecular-beam dynamics studies
4	Deep-etch x-ray lithography (LIGA)
5	EUV interferometry
6	Fluorescence microscopy
7	Fluorescence spectroscopy
8	Infrared microscopy
9	Infrared spectroscopy
10	Magnetic microscopy
	Magnetic spectroscopy
11	Faraday rotation
12	Magnetic circular dichroism
13	Micro-diffraction
14	Photoabsorption microscopy
15	Photoabsorption spectroscopy (incl. EXAFS, NEXAFS, XANES)
16	Photodissociation
17	Photoelectron microscopy
18	Photoelectron spectroscopy (incl. ZEKE)
19	Angle-resolved valence spectroscopy
20	Core-level spectroscopy (ESCA, XPS)
21	Diffraction/holography
22	Protein crystallography
23	Soft x-ray interferometry
24	Spectromicroscopy
25	Time-of-flight spectroscopy
26	Total reflection x-ray fluorescence
27	X-ray holography
28	X-ray transmission microscopy





FACILITY REPORT

The overall goal of the ALS is as simple in its nature as it is challenging in its implementation: to provide the best possible service to our users and to provide the quality of operations that will make the ALS the location of choice for scientific and industrial research using synchrotron light.

The success and growth of the scientific program depends directly on the quality of operations the facility provides. In 1994, the ALS demonstrated its ability to deliver a high-quality beam to user experiments on a reliable schedule, and to design and construct beamline components and insertion devices to exacting standards. We made great strides toward our goal of push-button operation, as shown by the rapid return to full operation after a two-month planned shutdown in spring 1994, and ALS expertise in beamline and insertion device design and engineering have allowed beamlines to generate publishable data within days of their first commissioning efforts. The success of these efforts depends on the cooperation among ALS groups and between the ALS and users.

In addition to improving service to users and increasing the number of beamlines, we are committed to expanding the research resources available in order to capitalize on the investment already made in the construction and operation of the ALS. Two projects begun in 1994, the Structural Biology Support Facilities and the Macromolecular Crystallography Facility, will provide major enhancements to the scientific program.

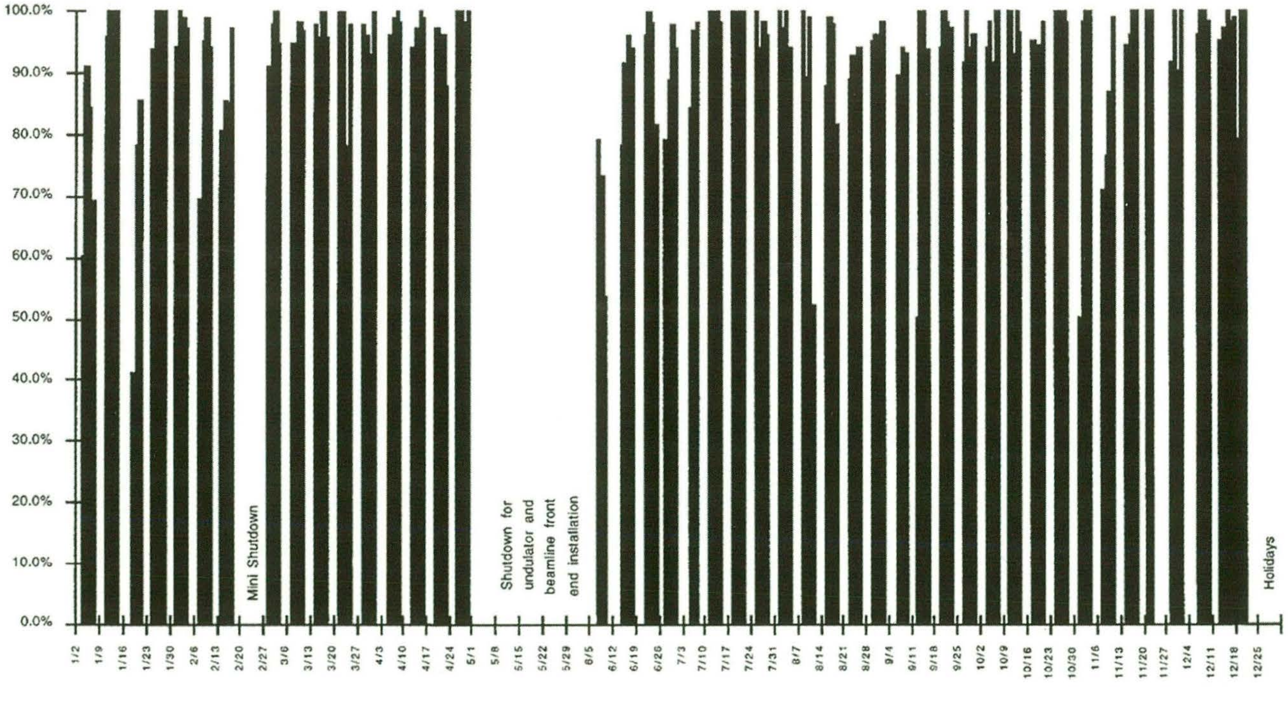
About photo (left):
The spectacular light show produced by an illuminated Vitamin C crystal is due to scattering of the incoming light (diffraction) by the crystal's neat mosaic of atoms sited on regular lattices. Every crystalline structure has its own unique diffraction pattern, and all crystals of the same substance produce identical patterns.

Operations Overview

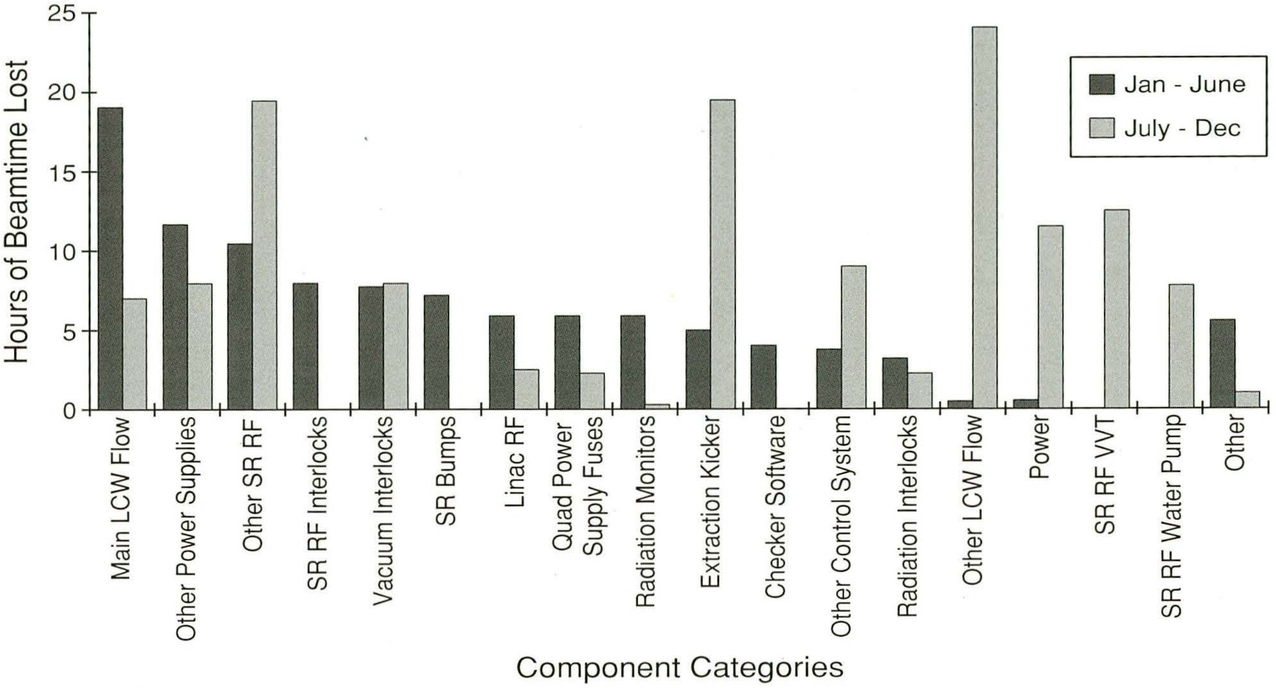
The ALS staff devoted considerable effort during the first full year of user operations to maintaining consistent, high quality machine performance and to establishing an efficient and safe work environment for users. The ALS averaged 94.5% for actual vs. scheduled beamtime for 1994, a truly remarkable achievement for a new machine and a testament to the quality of engineering and the hard work of the Accelerator, Operations, and Mechanical and Electrical Engineering groups.

Of course our ultimate goal is 100% delivery of scheduled beamtime to users, and this requires us to improve our quality of operations still further. To do this, the accelerator physics group continually works to meet the ever more demanding requirements of user experiments (see "Accelerator Physics," p. 40), and the operations team rigorously analyzes what causes our beamtime "outages" (i.e., the missing 5.5% in 1994). The team's diagnosis and correction of problems associated with the main low-conductivity water (LCW) flow reduced lost beamtime due to this item from 19 hours for January through June to 7 hours for the remainder of the year, and we expect to improve this figure still further in 1995.

We have worked closely with the user community to develop short- and long-term schedules that incorporate suggestions and requests solicited from the users for the modes of operations they prefer. The result was a new weekly and long-term schedule for operations which was put into effect after the February 1995 shutdown. The long-term schedule allocates specific weeks for the various modes of operation requested by users, including 1.0, 1.5, and 1.9 GeV operations, and two-bunch operation. Unfortunately, constraints caused by the ALS's low operating budget allowed us to operate for only fourteen 8-hour shifts per week, with nine allocated to user experiments and five set aside for installation, maintenance, and accelerator physics.



ALS performance for machine operations during 1994 was 94.5% availability of beam for user shifts (actual/scheduled): an excellent record. The gaps reflect the fact that there were no machine operations during weekends due to insufficient funding.



In 1994 the ALS initiated a process for analyzing the different component categories which contribute to lost beamtime in order to reduce future beamtime outages.

Another area of significant progress was in minimizing shutdown times for major equipment installation (a fact of life for a new and rapidly growing facility) so that they have a minimal impact on user operations. Our ability to reduce the length of our scheduled shutdowns is largely attributable to careful long-term planning and the use of the ALS-developed dry-tent technique (see p. 36) during equipment installation, eliminating the need for baking out the sections of accelerator and beamline vacuum chamber that have been brought up to atmospheric pressure.

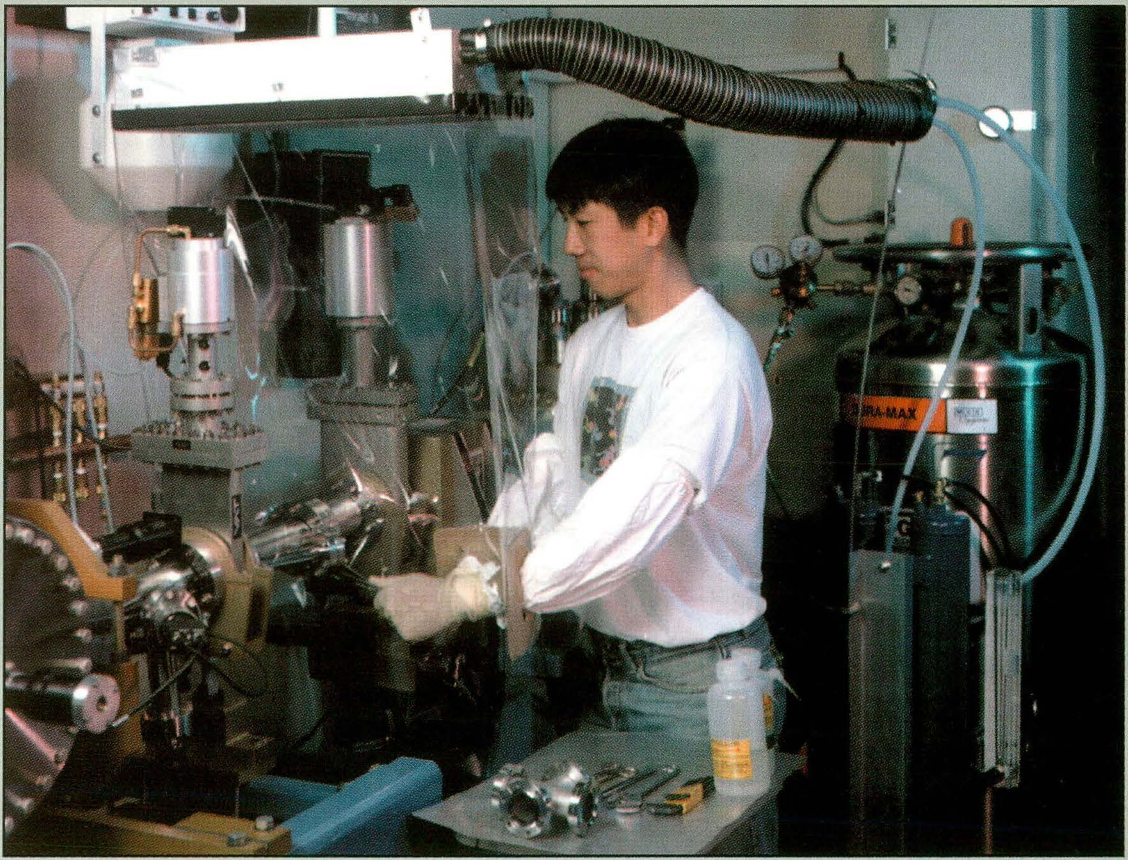
The application of these processes allowed us to complete the May-June shutdown for equipment installation on time and to resume full operations as scheduled. Major shutdown activities included the installation of two beamline front ends and an 8-cm-period undulator for Beamline 9.0. When machine operations resumed, it took only two days to achieve a storage-ring beam lifetime of 14 hours at 200-mA beam current, the same as it had been before the shutdown. The dry tent technique not only permitted rapid restoration of the storage-ring vacuum and beam lifetime but also reduced to five weeks what would have otherwise been an eight-week shutdown.

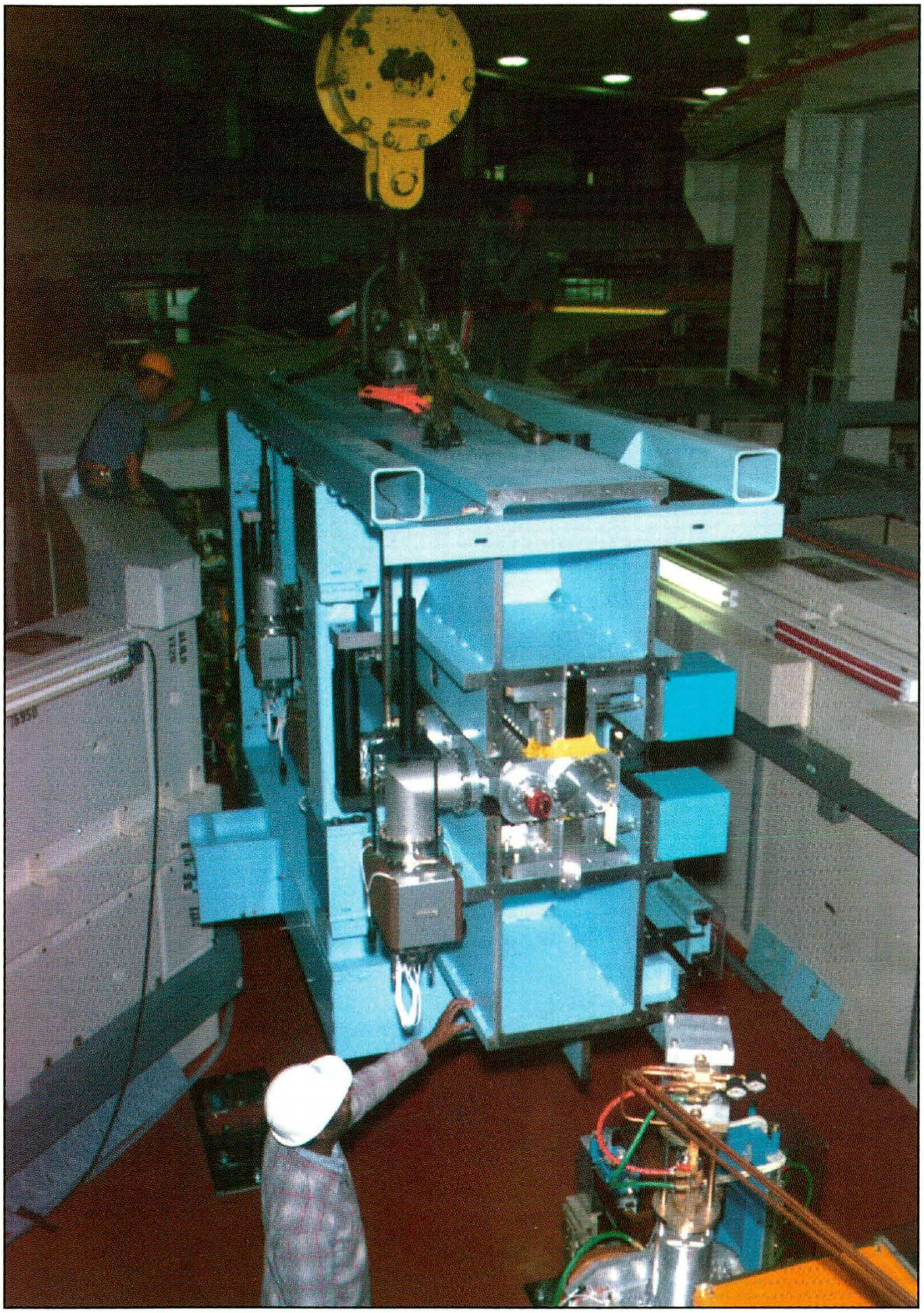
Scheduled shutdowns for 1995 include January–February to install a small-gap vacuum chamber for the 5-cm-period undulator (U5) for Beamline 7.0; and September–October to install a 10-cm-period undulator (U10) for Beamline 9.0, move the U8 presently in that location to sector 12, and survey the entire storage ring. The small-gap chamber allows the U5 undulator to be used with a smaller magnetic gap (and therefore a higher magnetic field) between the magnetic poles. At the narrowest gap, the maximum effective field strength will be increased to .85 T, extending the device's energy range to a lower limit of 50 eV. Some of the other activities planned for the winter 1995 shutdown include the installation of the front end for bend-magnet Beamline 7.3, and a mirror tank for Beamline 9.0.

Dry-Tent Technique Cuts Installation Times

A new vacuum technique developed by ALS mechanical technicians substantially reduces the length of shutdowns scheduled for equipment installation by obviating the need for baking out *in situ* sections of accelerator and beamline vacuum chamber that have been brought up to atmospheric pressure, a 1–2 week process formerly necessary during shutdown time to remove contaminants from ultra-high vacuum (UHV) components. The successful demonstration of the "dry tent" technique on a variety of vacuum joints means that the ALS can schedule future shutdowns to be shorter than was once necessary, thereby increasing beamtime available to users.

The dry tent process is named for the clear vinyl tent which is erected over the UHV connection point. Dry, filtered air is forced down through the tent from above, creating a laminar flow to keep moisture and contaminants out of the UHV components. Both the existing UHV chamber and the new UHV section are brought to atmospheric pressure using dry nitrogen, cleaned by a gas purifier. Then a technician, wearing double surgical gloves and working through penetration ports in the tent, removes the end caps from the UHV components (allowing the nitrogen to flow out) and completes the connection.





The third ALS undulator was installed in sector 9 of the storage ring on May 6. The 25-ton structure had to be lifted above the storage ring from the assembly area on the experiment floor, then lowered into place through the top of the removable shielding.

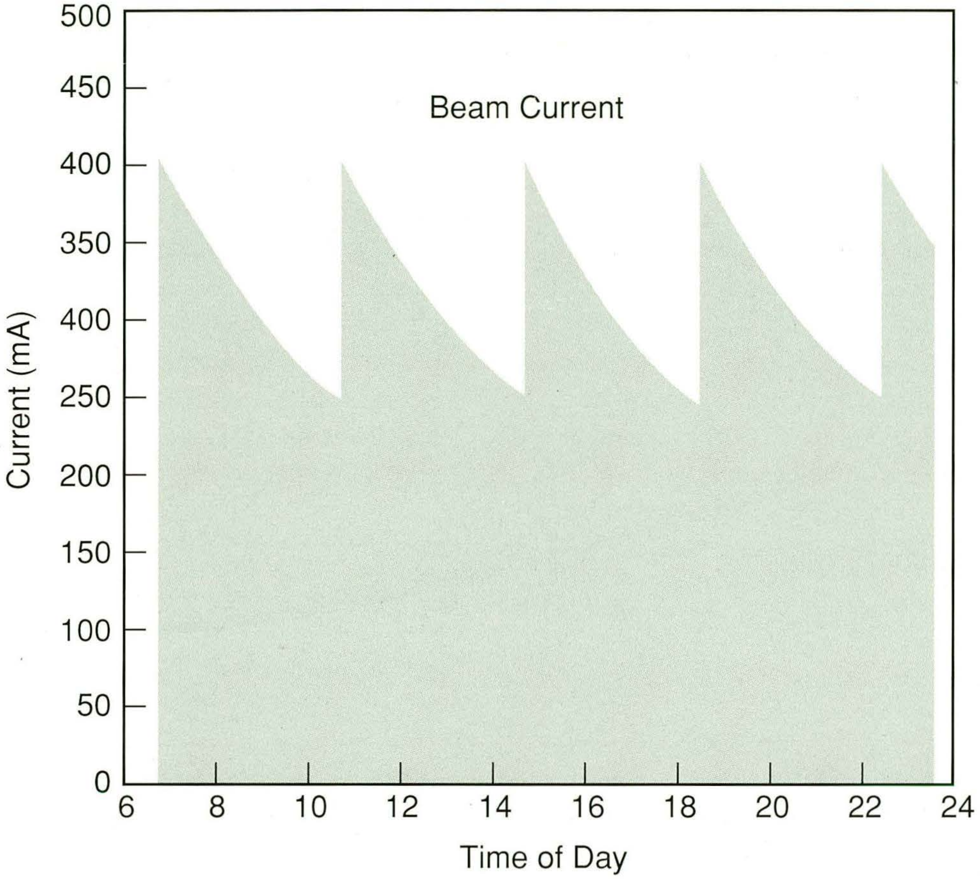
Another high priority this year has been to tailor our program and facilities to meet the needs of our users. Our objective in all cases is to facilitate research so users can work in a safe manner and complete their experiments with minimal interference. As such, we developed new procedures to guarantee that our users are adequately informed about safety issues at the ALS, and to streamline the experiment review process.

Monopods Simplify Surveyors' Tasks

The ALS has devised a new tool called a monopod that substantially decreases the time required for survey of ALS monuments, while increasing survey accuracy. The monuments (100 plus markers in the ALS floor) form a network of reference points which are used for maintaining proper storage-ring alignment (thus contributing to longer beam lifetimes) and for accurately aligning new beamline components. Careful initial alignments, coupled with high-quality design and engineering, have allowed beamlines at the ALS to obtain publishable data within days of their first commissioning efforts.

The advantage of using a monopod derives from the need to know the height of the surveying tool as exactly as its horizontal position. In the past, determining an instrument's height required making an additional sighting; since the monopod's height is a known constant, this step is now unnecessary.

The monopod's main component is a cylinder of carbon fiber, chosen for of its low coefficient of thermal expansion. At the monopod's bottom end is a beveled cup designed to rest on a spherical precision target placed in a monument; the top end has an adapter used to mount any of several surveying instruments or targets. The monopod shown extends through the storage ring shielding to a monument set in the floor under the storage ring; shorter ones are used for measurements made from the experiment floor.



An example of the high level of accelerator and storage ring performance achieved by the ALS. Routine operations in 1994 were at an energy of 1.5 GeV, and a starting current of 400 mA in 320 consecutively filled bunches out of a possible 328.

To provide site-specific training, for example, we produced a new safety video and *ALS Safety Handbook.*

To ensure we keep in touch with what our customers want, we developed a User Services Questionnaire to solicit feedback from our users on a regular basis. Their suggestions plus recommendations from members of the Users' Executive Committee have guided our efforts to develop new facilities and services for ALS users (described in "User Services," p. 56). We also initiated several new communication tools to keep our users and other members of the scientific community informed about ALS activities and scientific program developments. These include our electronic bulletin *ALSNews,* User Advisories to provide information on issues of concern to users, and the *ALS Users' Handbook.*

Accelerator Physics

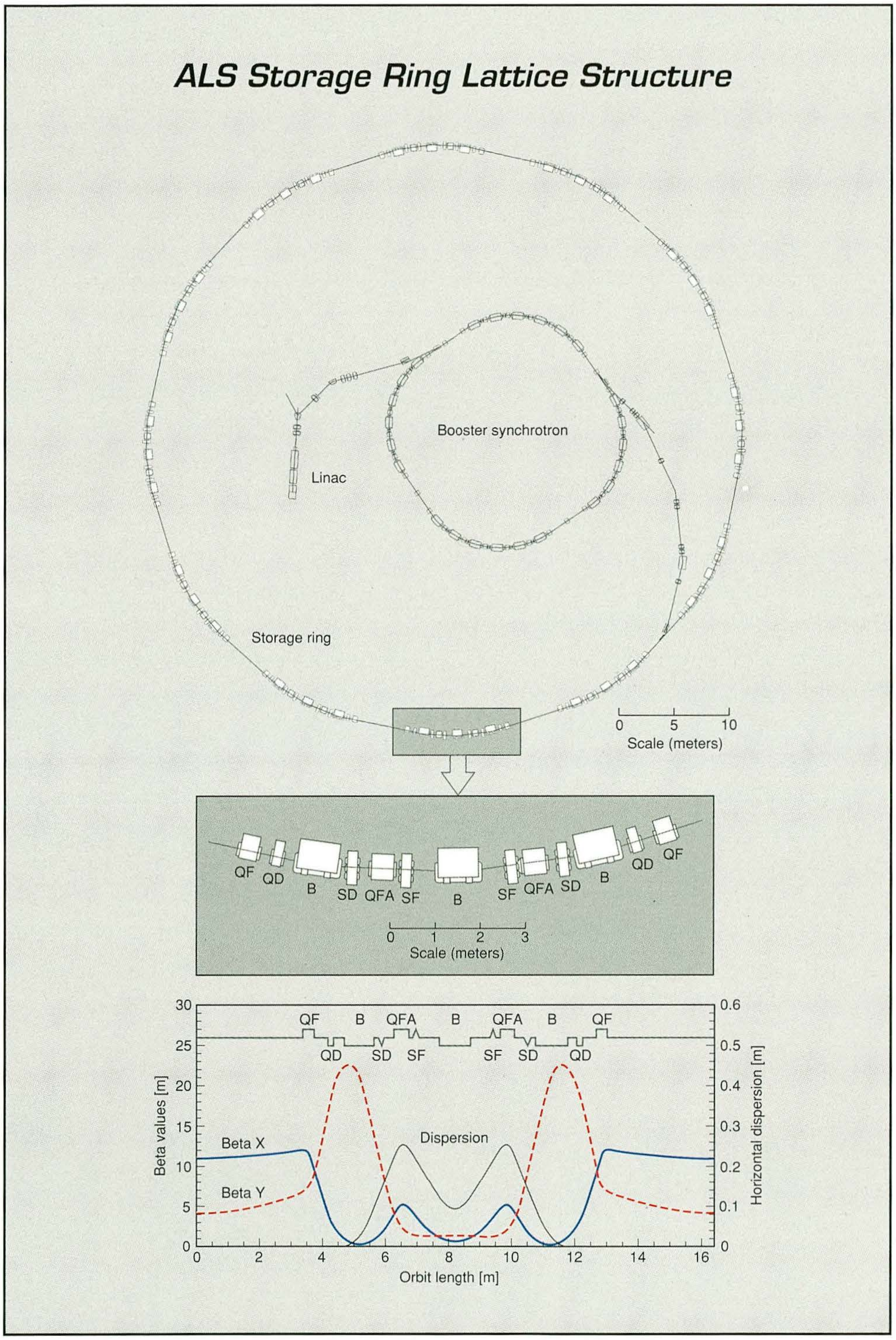
The ALS made great advances during 1994 in understanding the operational characteristics of the storage ring, and in establishing optimized parameters for routine user operations. Major accomplishments of direct benefit to user operations included improving beam stability, implementing user control of undulator gaps, and developing procedures to rapidly set the correct rf frequency (which determines the circumference of the electron orbit) and establish minimum closed-orbit distortions. The success of this work was due in large part to the efforts of the Accelerator Physics Group in collaboration with ALS operators, the Controls Section, instrumentation engineers, the Experimental Systems Group, ALS users, and the Beam Electrodynamics Group from LBNL's Center for Beam Physics (CBP).

Measurements of the electron beam made at low beam current, where collective effects (due to large charge density) and beam instabilities are negligible, show that the parameters of the beam match the design values. However at larger currents, such as the 400 mA routinely used as the starting current for user operations, the quality of the electron beam is degraded, mainly due to coupled-bunch motion that increases the energy spread of the beam. Development of a feedback system to correct this problem began in 1994, and tests with a prototype indicate that it will be able to restore high-quality beam up to the full operating current. This system to be is expected to begin operation in 1995.

ALS Storage Ring Parameters

Operating energy	
Nominal	1.5 GeV
Minimum	1.0 GeV
Maximum	1.9 GeV
Beam current	
Multibunch mode	400 mA
Single-bunch mode	20 mA
Natural emittance	3.6 nm·rad
RF frequency	500 MHz
Emittance ratio ($\varepsilon_y/\varepsilon_x$)	$\leq$ 3%
Horizontal beam size at symmetry	$\cong$ 0.20 mm
Vertical beam size	$\cong$ 0.03 mm
Filling pattern (multibunch mode)	320/328 bunches
Bunch spacing	2 ns
Beam lifetime* (multibunch mode)	14 hrs

* Beam lifetime defined as the time for the beam to decay from 400 mA to 147 mA.



(Top) The ALS storage ring has 12 arc-shaped and 12 straight sections. (Middle) One superperiod of the ALS triple-bend achromat lattice. A compact structure is achieved by including vertically focusing gradients in the bend magnets (B), and by using a single quadrupole family (QFA) to control the dispersion function, a quadrupole doublet family (QF and QD) to match the betatron tunes, and two families of sextupoles (SF and SD) to correct the chromaticity. (Lower) Horizontal and vertical β functions and dispersion for one superperiod of the storage ring lattice.

Beam stability was another area of considerable effort during 1994. We analyzed movements in the beam caused by the small residual dipole fields in the undulators and developed feed-forward algorithms to minimize the effects of these fields. Other investigations led to the correlation of periodic beam motion, with periods of around 10 minutes and 1 hour, to changes in the low-conductivity water (LCW) temperature and the local air temperature respectively. This work remains a high priority and will continue in 1995.

Improvements in Performance

Expanding the Range of Operating Conditions

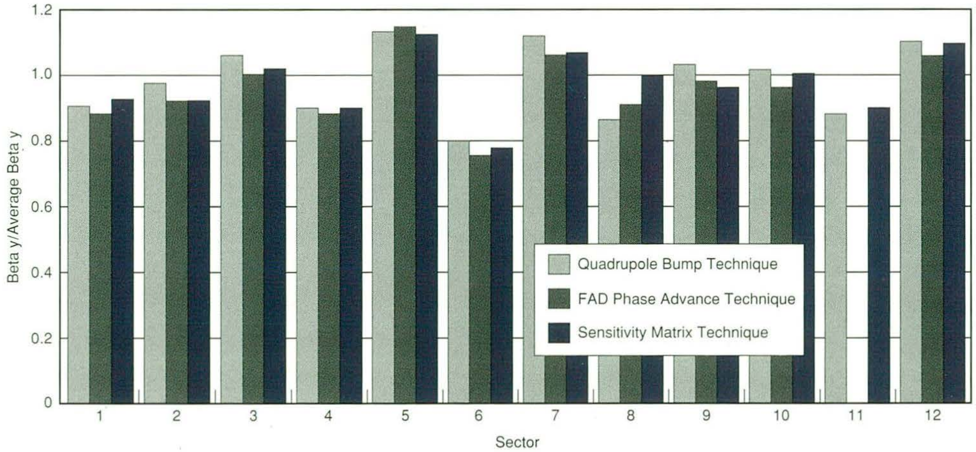
The ALS successfully tested and demonstrated its ability to run at 1.3 GeV, 1.9 GeV, and in two-bunch mode in response to user requests for these operating conditions. The 1.3 GeV operations test, motivated by the need of some users to run experiments using photon energies below 7 eV, proved successful on the first attempt. The accelerator physics group took less than an hour to bring the beam current up to 300 mA with an estimated lifetime of 11 hours, comparable to normal operations levels. In preparation for 1.9 GeV operation in 1995, the storage ring was ramped from its maximum injection energy of 1.5 GeV to 1.9 GeV. This required only minor corrections to magnet settings predicted from magnetic measurements made four years ago. After these corrections were applied, a 50 mA beam was ramped, without any loss of beam, in 20 minutes. Future work will concentrate on improving the rf system, so that higher currents can be accelerated (the immediate goal is 250 mA), and on improving the ramping speed.

Implementing User Control of Undulator Gaps

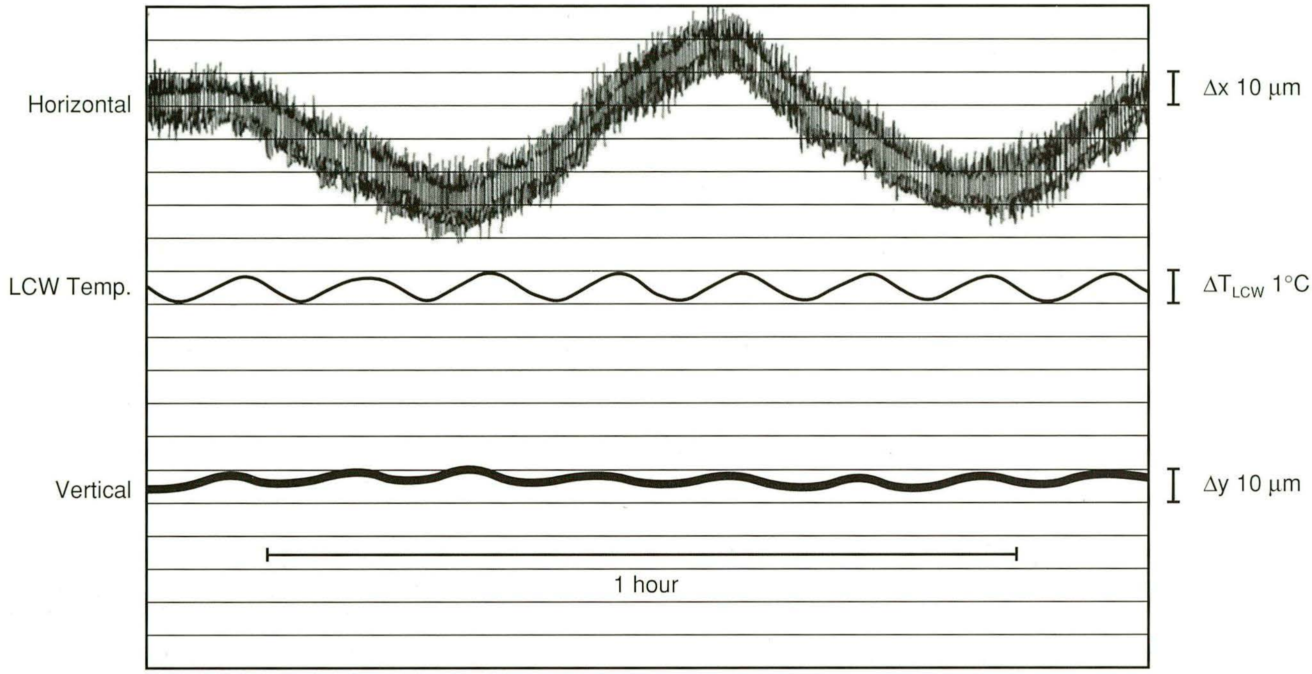
An important milestone in the ALS's commitment to being a user-oriented, user-friendly facility was creating the means to allow users to adjust the gap of their beamline's undulator independently, knowing the change would not disturb the beam delivered to other experiments. The new "feed-forward" algorithm making this possible was developed by measuring the effects on the electron and photon beams caused by changing the undulator gaps, and calculating the necessary changes to the local corrector magnets to compensate for those effects. Measurements show that the feed-forward mechanism holds beam position distortions under 30 μm for all adjustments.

Measurement-Based Model of Storage Ring

In pursuit of ever-higher accelerator performance, the accelerator group abandoned its original, simulation-based model of the storage ring and developed a better measurement-based model incorporating algorithms based on how the accelerator actually behaves. The model is derived by making changes to a single device, such as a corrector magnet, and measuring the effects of the changes on different parameters, including closed-orbit, chromaticity, betatron tune, and several others.



A compilation of three measurement methods giving the vertical β-function at twelve homologous positions around the circumference of the storage ring. The results confirm that the magnet-to-magnet quadrupole strength variations are within 10^{-3} rms, and closed-orbit displacements in sextupole magnets are within 200 μm rms.



Beam jitter of 20 μm peak-to-peak in the horizontal plane, and 4 μm in the vertical plane (well within the specified tolerance of 10% of the rms beam sizes) measured by the prototype electron-beam monitor. The jitter has a strong frequency component at 12 Hz which corresponds to a torsional oscillation mode of the girder on which the magnets are mounted. The cyclic vertical motion of 5 μm peak-to-peak with a period of about 10 minutes is shown to correlate with cyclic changes in the temperature of the low-conductivity water (LCW) cooling system.

These measurements combine to form response matrices, which can be used in control algorithms such as the "feed-forward" algorithm discussed above.

The accelerator measurements also show how well the storage ring conforms to the perfect twelve-fold symmetry with which it was designed. Beam measurements around the ring indicated that ALS magnet fields are within 10^{-3} of their design values, and magnet locations are accurate to 200 μm.

Beam Position Stability

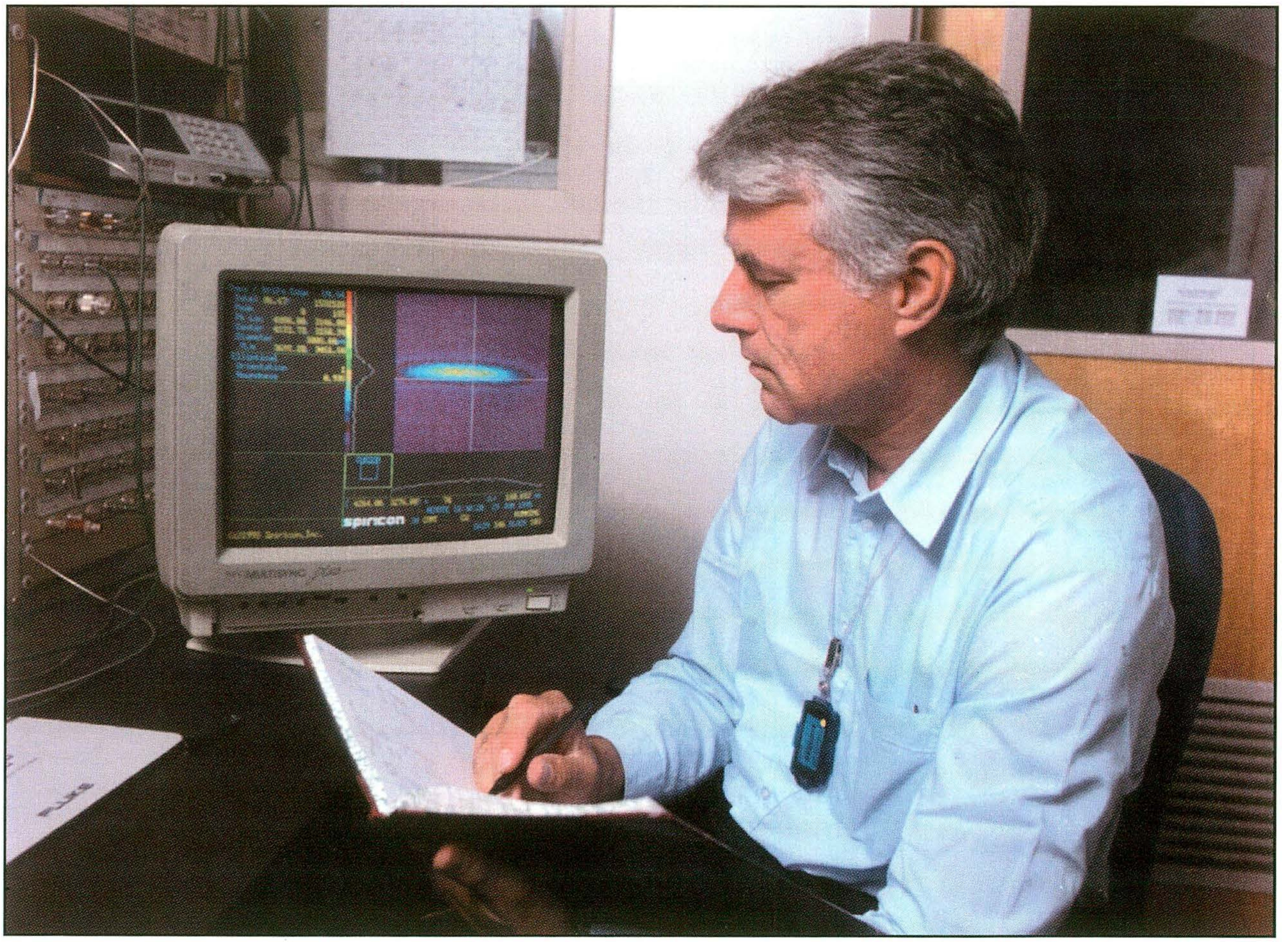
Photon beam drift and jitter was a major issue in 1994, and will continue to be into 1995. Part of the problem is that it can be very difficult to separate the photon beam motion caused by movement of the electron beam from that caused by movement of optical elements at individual beamlines. The present beam-position monitor system cannot detect electron beam motion at the levels necessary for establishing how electron beam motion is affecting photon beam motion. However, a prototype electron-beam monitor (being developed for a system to protect the vacuum chamber from errant photon beams in undulators) has proved to be sensitive to movements of less than 1 μm, and thus is perfect for observing electron beam jitter. Also, because the new electron-beam monitor is anchored firmly to the floor, it can be used to monitor longer-term drifts in beam position.

Measurements using this new diagnostic have revealed beam jitter (albeit well within the specified tolerances for beam position stability), in which one of the frequency components corresponds to a torsional oscillation mode of a girder supporting magnets in the storage ring. Other periodic beam motions have been correlated with changes of temperature in the low-conductivity water (LCW) cooling system and in the surrounding air. We will continue to address the beam stability issue using additional electron-beam monitors like those described above and the diagnostic beamline.

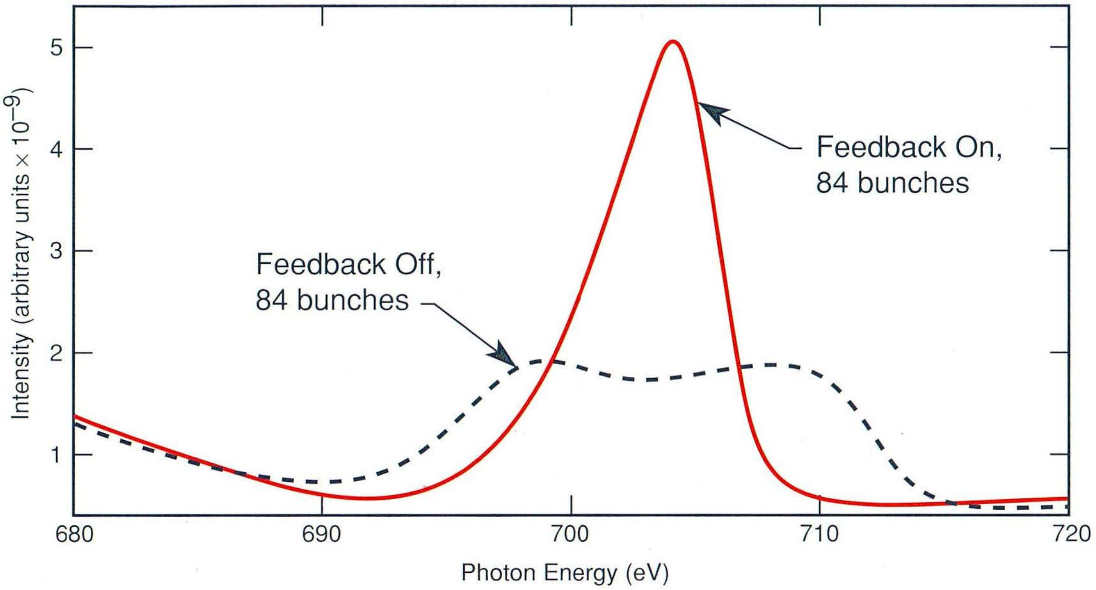
The diagnostic beamline, beyond its utility for observations of beam position, is an extremely versatile tool for diagnosis of instabilities and of drifts in accelerator mechanical and electrical systems. It began operations in its present (improved) configuration in December 1994, and allows accelerator physicists to make real-time observations of beam position, movement, and size, as well as electron bunch lengths, bunch purity, and time-dependent variations of all of these quantities.

Longitudinal and Transverse Instabilities

When electron bunches interact with each other through self-induced electromagnetic fields in the rf cavities and/or the vacuum chambers, coupled-bunch instabilities (CBI) result. In the ALS, the longitudinal CBI is responsible for a four-fold increase in the energy spread of the



The diagnostic beamline's ability to gather spatial and temporal information about the electron beam has proved to be a valuable tool for improving the quality and consistency of photon beams delivered to users. The real-time image of the beam is recorded by a CCD camera in the diagnostic beamline, and a digital representation of this image along with processed data on beam center location, ellipticity, etc., are displayed on a monitor in the ALS control room. By observing and recording these data, the ALS can correlate their changes with variations in other storage ring and injection parameters that are recorded over the same time period.



Measurement of the fifth harmonic of the undulator spectrum on Beamline 7.0 with and without longitudinal feedback. The feedback system uses state-of-the-art digital processing to reduce the energy spread of the electrons in each bunch, thereby improving the linewidth and intensity of the undulator harmonics.

electron beam. This energy spread dominates the linewidths of third, fifth and higher undulator harmonics. A novel feedback system using state-of-the-art digital processing techniques is being developed jointly with groups from Stanford Linear Accelerator Center and LBNL's Center for Beam Physics (CBP). Feedback system tests with 84 electron bunches in the storage ring showed the expected improvement in spectral performance; the production version will be installed in 1995.

When the longitudinal instability was brought under control in feedback system tests, a transverse instability (driven by the interaction of the electron bunches with the walls of the vacuum vessel) appeared and increased the vertical emittance of the beam. The feedback system for this instability was developed and successfully commissioned by the CBP Beam Electrodynamics Group.

Plans for 1995

Accelerator physics efforts in 1995 will concentrate on the following activities, with the order depending to a large extent on user requests.

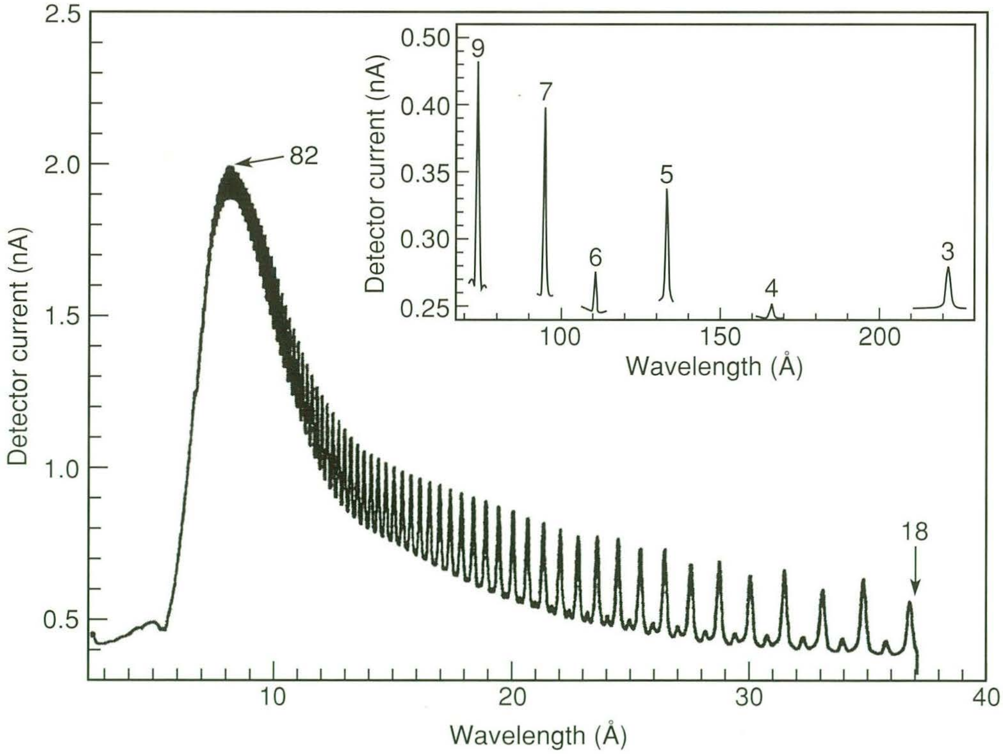
- Beam stabilization: Decrease drift and jitter.
- Magnet "feed-forward:" Provide undulator steering and tune compensation, but will also facilitate faster energy ramping.
- Multi-bunch feedback: Produce higher quality undulator spectra.
- Storage ring injection efficiency: Improve filling times, facilitate trials of top-off operation.
- Characterize the effects (particularly on transverse instabilities) of the narrow gap vacuum chamber to be installed in sector 7, and the effects caused by the higher magnetic fields when the 5-cm-undulator in sector 7 is used with smaller magnetic gaps.

Experimental Systems

From insertion devices to beamlines and endstations, the work of the Experimental Systems Group includes the components needed to get synchrotron light from the storage ring to user experiments. Through R&D, commissioning, and scientific support for users, the group strives not only to provide state-of-the-art instrumentation that will allow experimenters at the ALS to conduct research at the forefront of their fields, but also to develop new projects that will enhance the scientific capabilities of the facility both in the near term and in the more distant future. Getting our most complex systems to work the first time with little or no adjustment is truly a team effort, and we acknowledge the superb support from the ALS Mechanical and Electrical Engineering Groups.

Undulator Performance

We extended the work we began last year on measurements of the angular and spectral characteristics of the 5-cm-period undulator (U5) for Beamline 7.0 to include a full and detailed measurement and analysis of the undulator's performance. The measurements were taken using an ALS-designed transmission grating spectrometer (TGS). The conclusion from this work was that the U5 undulators (a second U5 illuminates Beamline 8.0) are essentially perfect—even in the higher harmonics—so that the quality of light they deliver is limited primarily by such properties of the electron beam as emittance and energy spread. Among other results, we measured values for the emittance of 4×10^{-9} m·rad and $< 2 \times 10^{-10}$ m·rad in the horizontal and vertical directions, respectively.



Even small errors in the position and alignment of the poles of an undulator cause the higher harmonics to broaden into a continuum so that they are no longer distinguishable. Observation of higher harmonics is therefore an important test of undulator quality. Here, the spectrum of the 8-cm-period undulator (U8) taken using the transmission grating spectrometer shows harmonics from the third to the eighty-second (and beyond) at a value of K = 5.24 (25 mm gap), thereby helping to confirm the near-perfect performance of the device.

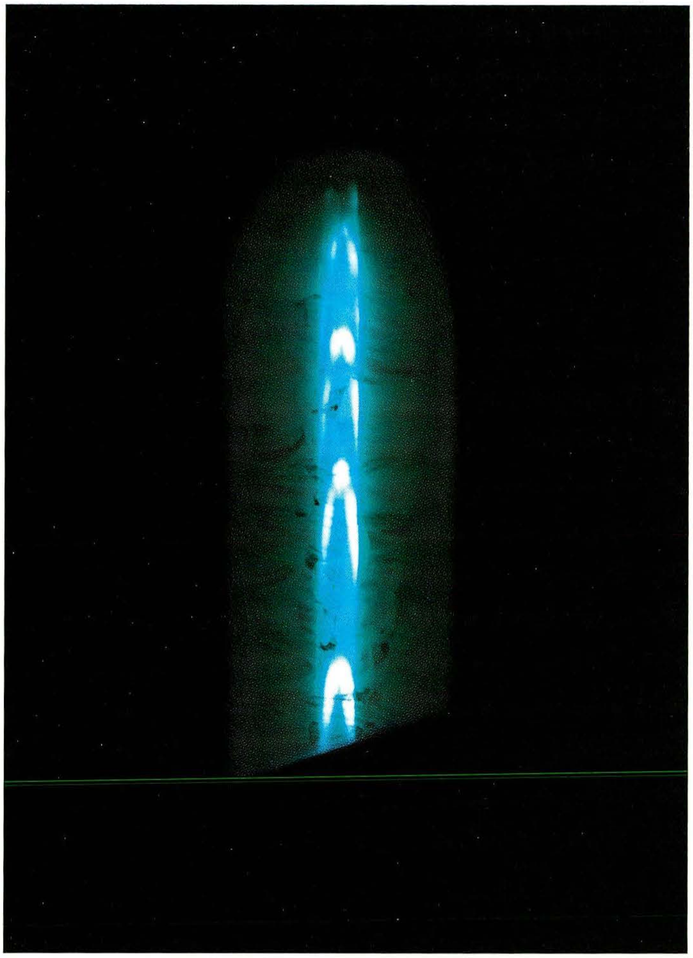
These numbers are considerably better that the "book" values previously published for the ALS, although it should be noted that our measurements were made at currents of < 0.5 mA because of restrictions imposed by the maximum power load that the TGS can handle.

In August, we re-enlisted the TGS in order to characterize the light from the newly installed 8-cm-period (U8) undulator for Beamline 9.0. At K = 5.2, we were able to measure about 100 harmonics before they merged into a continuous broad band, which confirmed the near-perfect performance of the device. An overview of ALS insertion devices and their performance parameters is given on page 53.

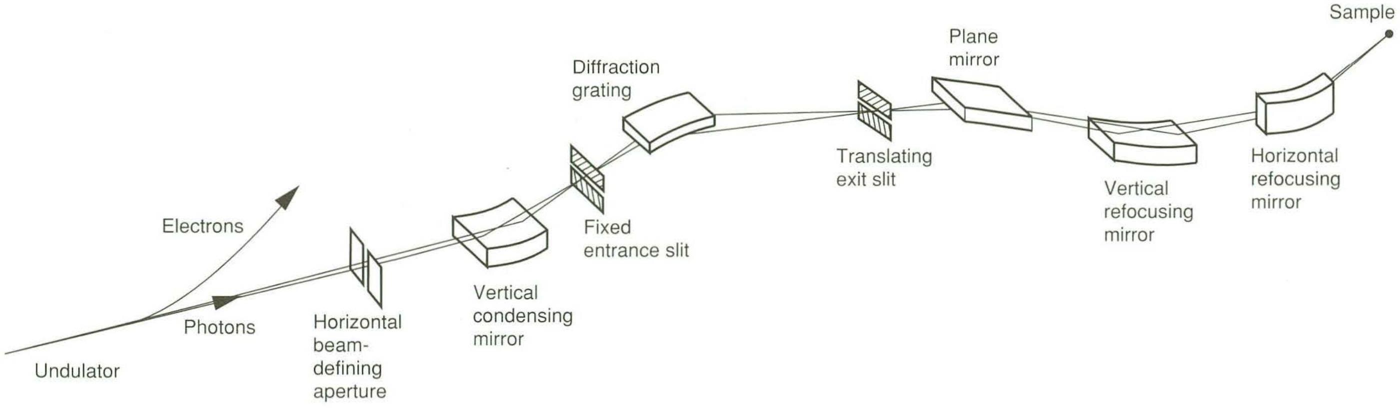
Beamline Commissioning

The initial group of user beamlines grew from three to eight in 1994, with ten more scheduled to begin operation by the end of 1996, for a total of 19 beamlines (including the accelerator diagnostic beamline). The Experimental Systems Group directed or contributed to the commissioning of several of these beamlines, particularly undulator Beamlines 7.0 and 9.0, whose performance characteristics were extensively measured.

Beamline 7.0. This beamline is a spherical grating monochromator (SGM) system, covering the photon energy range from 50 to 1200 eV. It consists of a spherical premirror to focus light vertically onto the entrance slit of the monochromator, three interchangeable spherical gratings, a translating exit slit, and post-exit-slit refocusing optics. The refocusing optics include a plane mirror, a vertically reflecting mirror that directs light from the exit slit to the sample, and a horizontally reflecting mirror that focuses the light from the source onto the sample. Beamline 7.0 was commissioned in record time, and its design parameters have been met or exceeded. In constant use since commissioning, it has proved to be



Undulator radiation incident on a zero-order light baffle, which is phosphor-coated and located directly downstream from the monochromator on Beamline 8.0, produces a characteristic pattern of dispersed light from the various undulator harmonics. A similar pattern was observed on Beamline 7.0. The light is distributed in parabolic arcs, with structure within each arc dependent on the harmonic number. The general parabolic shape results from the decrease in photon energy as the observation angle increases and from the increased diffracting power of the grating as the photon energy decreases.



Schematic diagram of the path of the synchrotron light through the optical components of Beamline 7.0. All of the upstream components from the horizontal beam-defining aperture through the monochromator gratings are water-cooled for protection against distortion due to the high heat load from undulator radiation.

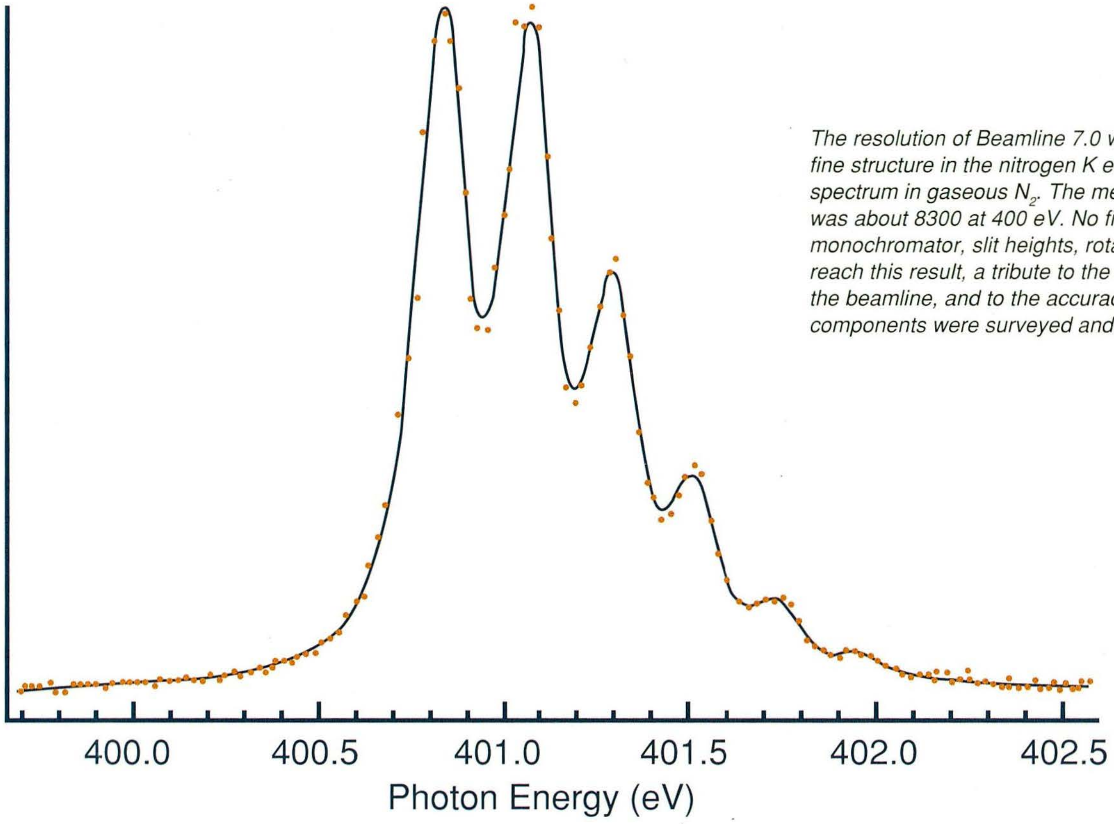
extremely reliable and robust, enabling a tremendous range of science to be performed in a relatively short time. The commissioning of the beamline proceeded in the following three steps:

Beam size at the monochromator entrance slit. We measured the size of the beam focused vertically on the entrance slit by rotating the premirror and monitoring the signal immediately after the slit and after the monochromator. Measuring the signal after the monochromator yielded a full-width-at-half-maximum (FWHM) beam size of 8 μm. This value agreed with expectations.

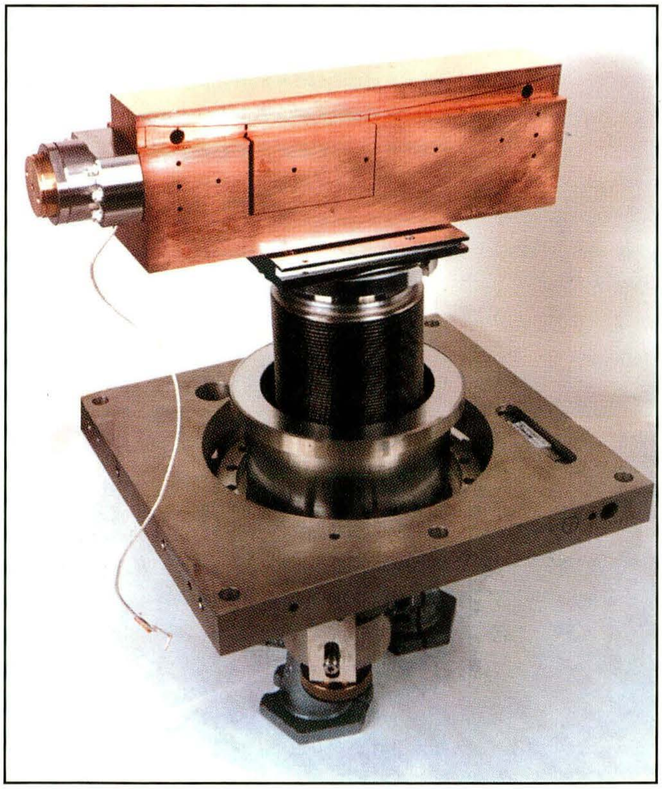
Resolution. To test the resolution of Beamline 7.0, we used the standard method of measuring the $1s \rightarrow \pi^*$ absorption spectrum in N_2 gas at the nitrogen K edge. We observed the usual series of vibrationally split peaks, and, after a few hours to calibrate the exit-slit motion, we obtained the anticipated resolving power of about 10,000. Our ability to achieve this very high resolving power so rapidly is a tribute to the mechanical engineering of the beamline and to the accuracy with which all the components were surveyed and aligned.

Beam size at the sample. One of the problems with a standard SGM beamline is that, as the photon energy changes, the exit-slit position must change in order to keep the beam in focus. Since Beamline 7.0 is designed to supply light to soft x-ray microscopes, we made the beamline's vertically refocusing mirror bendable, so that it could image the variable position object (the exit slit) onto the fixed sample position required by the microscope. We measured the focused beam size at the sample using a knife-edge technique, obtaining sizes of 45 μm (horizontal) by 20 μm (vertical).

Beamline 9.0.1. This beamline covers the energy range from 20 to 310 eV and is similar to Beamline 7.0 except for the addition of a horizontally deflecting mirror as the first element in the beamline. A range of techniques was used to measure the performance of the beamline, in



The resolution of Beamline 7.0 was tested by measuring the fine structure in the nitrogen K edge 1s→π absorption spectrum in gaseous N_2. The measured resolving power was about 8300 at 400 eV. No fine alignment of the monochromator, slit heights, rotations, etc., was required to reach this result, a tribute to the mechanical engineering of the beamline, and to the accuracy with which all the components were surveyed and aligned.*

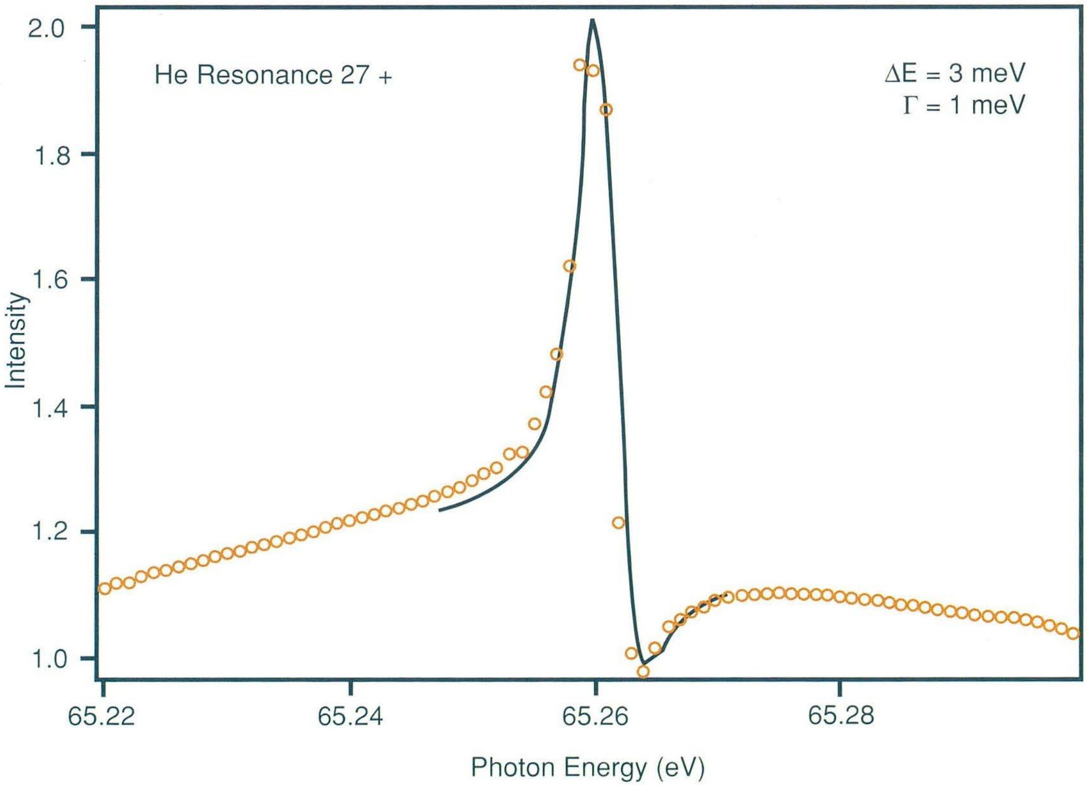


The Beamline 7.0 integral-bendable x-ray mirror was designed so that it could image the moving exit slit to the fixed sample position in scanning and full-field imaging microscopes. The thickness of the mirror varies as the cube root of distance along the mirror. This structure is machined into a single Glidcop™ block using the technique of wire electric-discharge machining (wire EDM). By applying a voltage to a piezoelectric drive system, one can obtain the required radius of bend.

particular the key resolution and flux parameters. Beamline 9.0.1 was commissioned extremely rapidly and was in routine operation during the last quarter of the year.

The standard resolution test at the lower photon energies serviced by Beamline 9.0 is the double-ionization series in the absorption spectrum of helium. The lifetime broadening of the higher term members of the series is extremely small and gives us an excellent resolution test. Measurements taken at approximately 65 eV indicate that the resolving power is close to 20,000, giving a line width of about 3 meV. This performance indicates that the system is extremely well aligned (owing to the accurate initial survey, as with Beamline 7.0) and that the grating used in this measurement has a low slope error.

Beamline 9.3.2. This bend-magnet beamline uses a standard SGM to cover the photon energy range from 30 to 1500 eV. This particular SGM was developed as the prototype ALS monochromator and was initially installed and tested on Beamline 6 at SSRL. The beamline underwent many modifications to make it compatible with the high brightness of the ALS, including a new cooled horizontally deflecting premirror and a new vertically focusing mirror. Another significant upgrade was the design and construction of a rotating platform, which allows light to be switched between the two experiment stations mounted on the platform without breaking vacuum. The beamline received its first light at the ALS in June 1994, at which time standard resolution tests, using a simple photoabsorption cell, indicated a resolving power of around 10,000. Now in routine use, the beamline is equipped with a variety of detection systems, including a high-resolution Scienta photoelectron spectrometer.

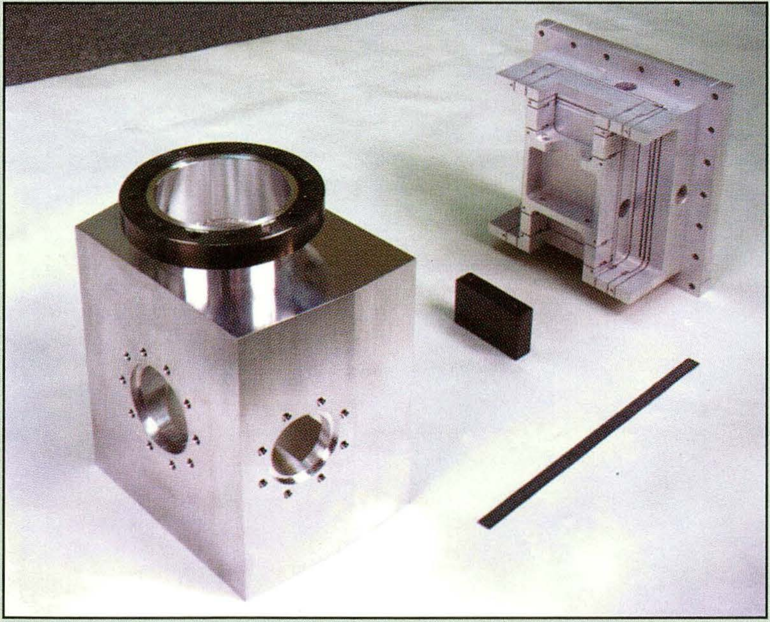


The absorption spectrum of the helium double-ionization series was used as a resolution test for Beamline 9.0.1. Since the lifetime broadening of the higher term members of the series is extremely small, this series is an excellent resolution test. The structure shown at approximately 65.25 eV indicates that the resolving power is close to 20,000, with a line width of about 3 meV.

ALS Innovative Engineering

Accurately steering the small, bright photon beam produced by the ALS through complex beamline optical systems is one of the many challenges being met by the ALS Mechanical Engineering Group. This challenge is compounded by the narrow entrance slits of high-resolution monochromators and the need for high stability in beam position. A stable beam is particularly important at Beamline 9.0.2, a high-resolution and high-flux beamline for chemical dynamics studies, in order to minimize noise in the experimental data. To achieve the steering sensitivity required, ALS engineers designed a precision actuator which is used to position the three mirrors (M4, M5, and M6) that steer the photon beam through the monochromator entrance slit, which has a typical width of 10 microns, and focus it onto the sample. (See p. 10 for a schematic of Beamline 9.0.2.)

The actuator has a novel design and uses advanced manufacturing techniques. It adjusts the mirror's position using a series of flexural pivots configured to convert linear motion to rotational motion. Initial tests of the actuator have shown that it is capable of achieving an angular resolution of .0003 degree. Aluminum was selected for the substrate material to take advantage of its benefits in ultra-high vacuum (UHV) environments. Aluminum has a low permeation rate (the rate at which gases move through materials), and the outgassing rate (the rate at which materials release adsorbed gases under vacuum) is 1–2 orders of magnitude lower than that of stainless steel, depending on material preparation. These factors allow the use of smaller, lighter vacuum pumps and reduce the pumping time required to achieve high vacuum. Another advantage of aluminum is its high level of machinability. Manufacturing the actuator from a single piece of material and using integral flexural pivots eliminates the need for contact bearing motion to adjust the mirror—a highly desirable feature for UHV environments. The actuator's ability to provide precise adjustment of an optical component could have application in other areas such as laser and visible light optics.



To complete the mirror tank assembly, the M5 silicon carbide mirror is mounted in the center of the actuator, which is then placed into the vacuum chamber and sealed using aluminum wires. Precise adjustment of the mirror is provided by a series of flexural pivots which convert linear motion to rotational motion.

Future Beamlines

Beamline 9.0.2. The installation of the chemical dynamics Beamline 9.0.2, which time-shares light from the U8 undulator with Beamline 9.0.1 by means of an insertable mirror, made steady process. This beamline is being constructed for use by the chemical dynamics PRT and has been a collaborative design effort. The beamline will have two branches, which themselves time-share their allotment of the light. The "white light" branch (9.0.2.1) is scheduled to be operational in late February 1995. Branch 9.0.2.2 will be equipped with a 6.65-m off-plane Eagle monochromator, designed in collaboration with LBNL's Center for X-Ray Optics, for extremely high resolution in the scanning mode. The complex geometry and stringent performance requirements of this beamline present many challenges in the area of beamline engineering (see "Innovative Engineering," p. 51).

Infrared Beamline. There has been considerable interest in the construction of an infrared spectromicroscopy facility at the ALS using light from a bend-magnet port, and we began the initial design for an infrared beamline. A key element is the system of mirrors to deflect the infrared to an experiment endstation on the experiment floor outside the storage-ring shielding. Owing to its close proximity to the radiation source, the power density incident on the first mirror is very high, and considerable effort is being made to determine the optimum design and location. Initial operation is scheduled for spring 1996.

Other Experimental Systems Group projects described elsewhere in this report include the protein crystallography beamline (p. 14), an application-specific beamline for magnetic spectroscopy and microscopy (p. 23), and a soft x-ray interferometer (p. 8).

Insertion Devices

The ALS has specified the major parameters for six of the ten insertion devices that the storage ring can accommodate. The latest in the series, an 8-cm-period undulator (U8), was installed in sector 9 of the storage ring during the May–June 1994 shutdown, bringing the in-the-ring total to three. Measurements of the undulator light indicate that the U8 is essentially perfect in its conformity to design specifications (see "Experimental Systems," p. 46). By late August, the undulator was delivering high-quality beams of extreme brightness to user experiments.

Work progressed on three additional insertion devices planned for installation in 1995 and 1996: a 10-cm-period undulator (U10), a 16-cm-period wiggler (W16), and a device to produce elliptically polarized radiation. In addition, the design and fabrication for the narrow gap vacuum chamber for the Beamline 7.0 U5 undulator was completed. After this chamber is installed in early 1995, it will be possible to close the undulator's gap to a minimum of 1.4 cm. At this gap setting, the maximum effective field strength will be increased to .85 T, extending the device's energy range to a lower limit of 50 eV. Assembly of the magnetic structure for the U10 was well underway by the end of 1994, the support structure and drive system were completed, and all aspects were on schedule for installation in September 1995.

Another key area of activity was the magnetic design and fabrication work for the W16 wiggler, the source for the protein crystallography beamline (see "Crystallography Beamline," p. 14), scheduled for operation in early 1996. Macromolecular crystallography requires a short-wavelength x-ray source that is intense and has a size and divergence matched to the size of the protein crystal and to the angular resolution defined by the size of the crystal

Insertion Devices for 1994–1995

Device	Beamline	Status	Energy Range (at 1.5 GeV)	Energy Range (at 1.9 GeV)	Period Length	Number of Periods	Operating Gap Range	Peak Effective Field Range
U5 Undulator	8.0	Operational	130–1900 eV	210–3000 eV	5.0 cm	89	2.3–4.5 cm	.46–.10 T
U5[1] Undulator	7.0	Operational	50–1900 eV	80–3000 eV	5.0 cm	89	1.4–4.5 cm	.85–.10 T
U8[2] Undulator	9.0	Operational	18–1200 eV	30–1900 eV	8.0 cm	55	2.5–8.3 cm	.80–.07 T
U10 Undulator	9.0	Design and construction in progress	5–950 eV	8–1500 eV	10.0 cm	43	2.4–11.6 cm	.98–.05 T
EPU[3] Elliptically Polarizing Undulator	4.0	Design in progress	60–1200 keV	100–1500 keV	5.0 cm	38	1.6–5.5 cm	.75–.10 T
W16 Wiggler	5.0	Design and construction in progress	5–13 eV	5–21 KeV	16.0 cm	19	18.0–1.4 cm	.03–2.0 T

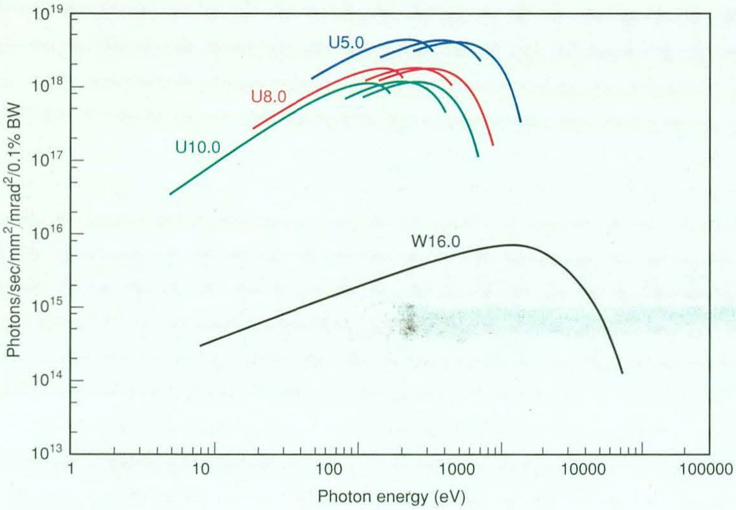
[1] Values given for performance with the narrow gap vacuum chamber installed in January 1995.

[2] To be moved to Beamline 12.0 in September 1995.

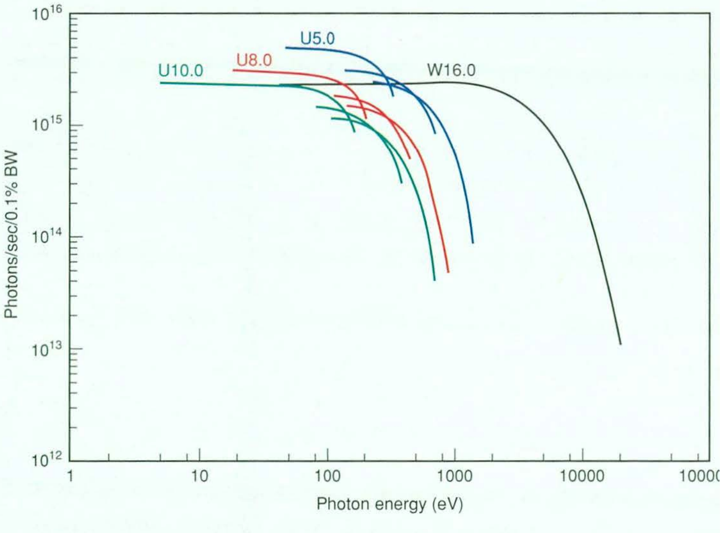
[3] In March 1995, it was decided to build an elliptically polarizing undulator (EPU) for sector 4 rather than an elliptical wiggler for sector 11.

unit cell and the wavelength. The spectrum and flux of radiation produced by a wiggler is dependent on the peak field strength of the magnets used in the device, and the 38-pole wiggler will have a peak field strength of 2 T (by comparison, the ALS bend magnets operate with a field of 1 T). In addition, operating the ALS storage ring at its maximum energy enhances both the flux and brightness delivered by the wiggler. For operation at 1.9 GeV, for example, there is a brightness advantage of a factor of three at 1 Å (the wavelength conventionally used for macromolecular crystallography) as compared to operation at 1.5 GeV. The magnetic structure of the W16 wiggler will be of a similar hybrid configuration as the U5 and U8 undulators now operating in the storage ring.

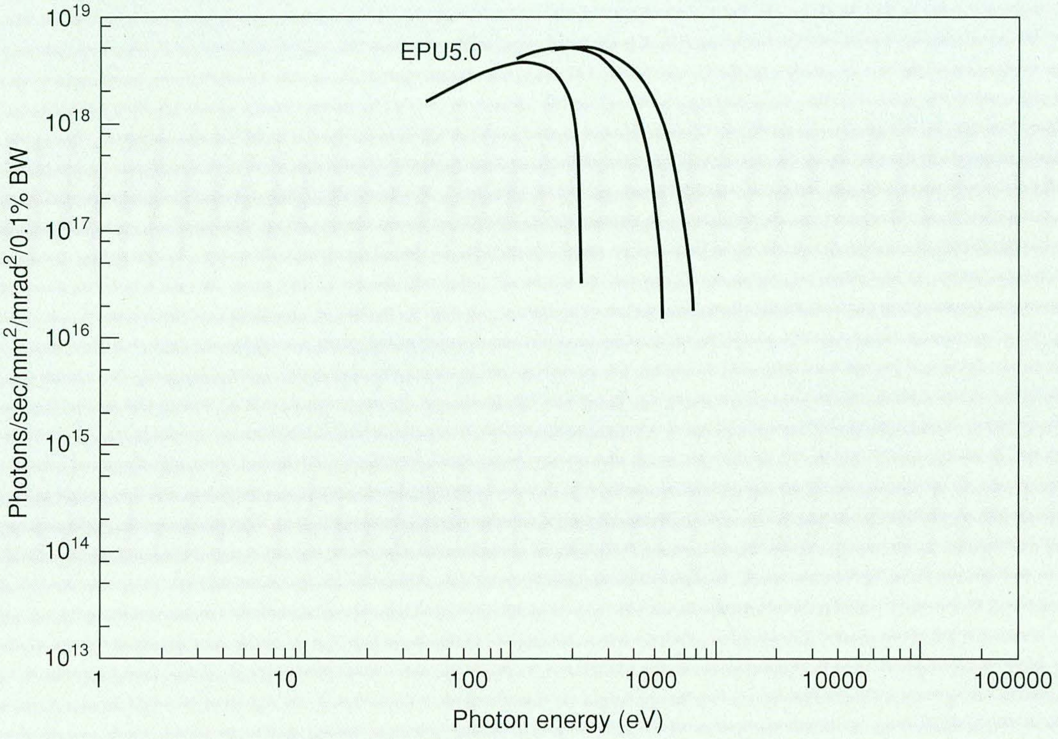
Some detailed design work on the magnetic structure and the support structure and drive system for an elliptical wiggler to produce circularly polarized light was started, but it was decided to redirect this effort toward a newly designed elliptically polarizing undulator (EPU), of a type pioneered by Shigemi Sasaki. The 5-cm-period EPU will produce a continuously adjustable magnetic field which can cause storage ring electrons to follow paths of arbitrary elliptical helicity, producing light ranging from pure right or left circular polarization to pure horizontal or vertical linear polarization, with changes of polarization taking a few seconds. Completion is scheduled for late 1996.



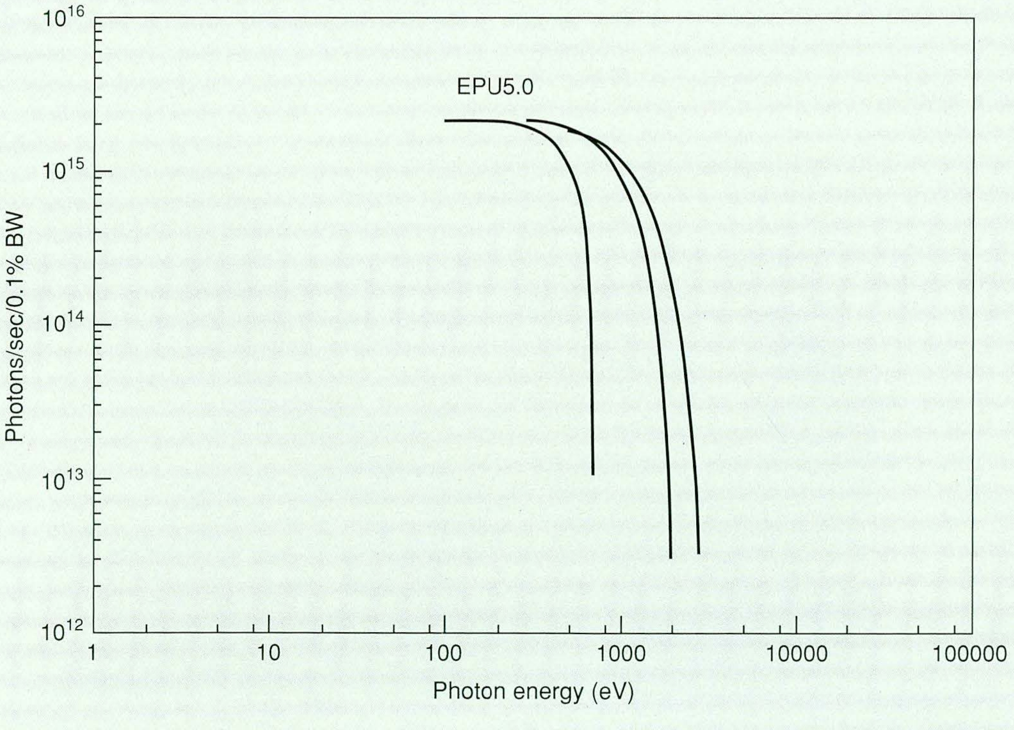
Brightness as a function of photon energy for the ALS undulators and wiggler. The brightness values plotted here reflect a 1.5-GeV, 400-mA electron beam. The undulator plots show the curves for the first, third, and fifth harmonics.



Photon flux as a function of photon energy for the ALS undulators and wiggler. The flux values plotted here are for a 1.5-GeV, 400-mA electron beam with a horizontal aperture of ± 2.5 mrad. The undulator plots show the curves for the first, third, and fifth harmonics.



Circular polarization merit function brightness for the elliptically polarizing undulator. The plots show the curves for the first, third, and fifth harmonics with a 1.5-GeV, 400-mA electron beam. The merit function brightness is defined as $M_B = P_c^2 B$, where P_c is the degree of circular polarization and B is regular brightness.



Circular polarization merit function flux for the elliptically polarizing undulator. The plots show the curves for the first, third, and fifth harmonics with a 1.5-GeV, 400-mA electron beam. The merit function brightness is defined as $M_F = P_c^2 F$, where P_c is the degree of circular polarization and F is regular flux.

User Services

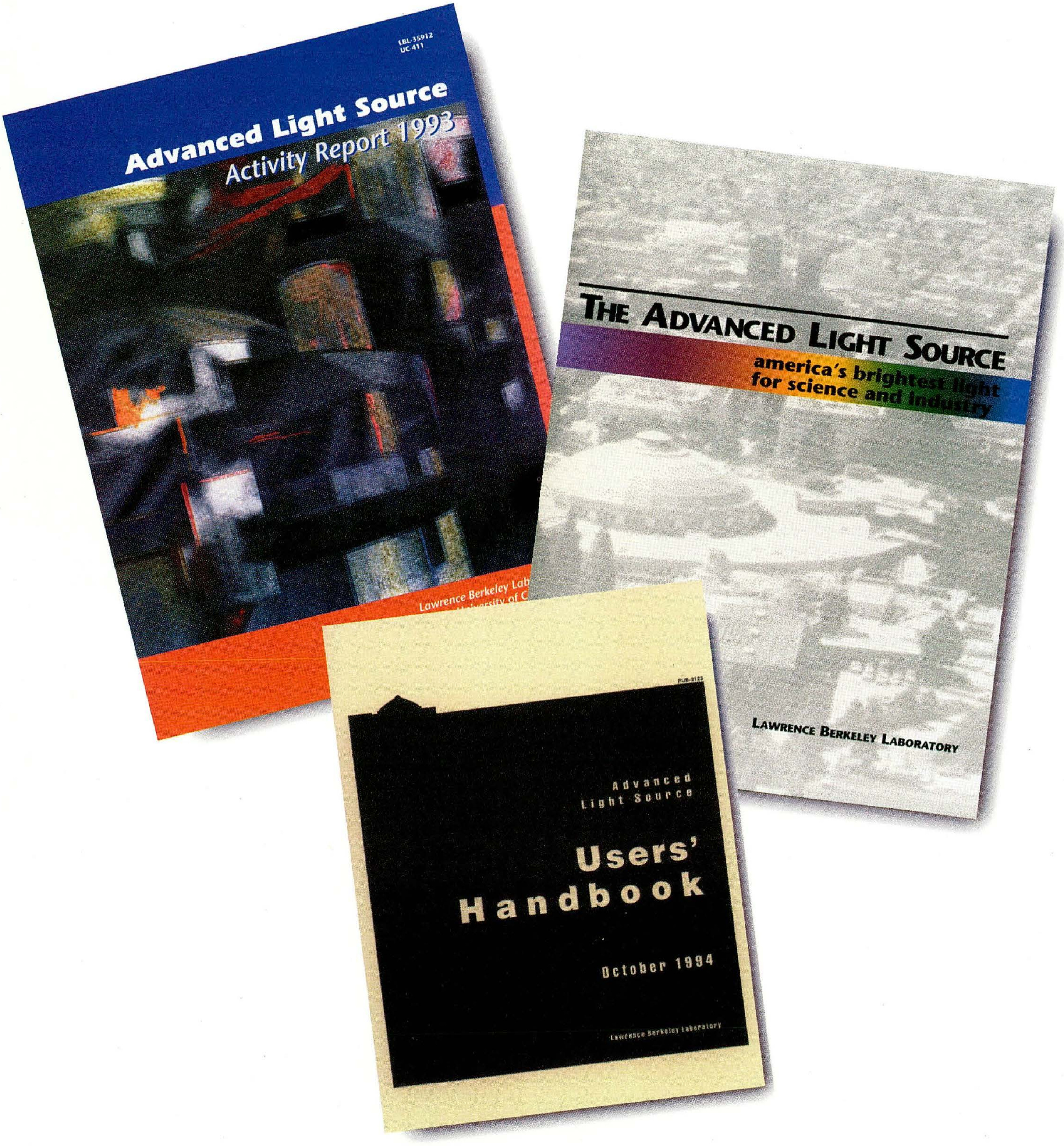
The ALS views high-quality service to users and scientific outreach as major responsibilities for a national user facility. This translates into developing a comprehensive customer service program designed to streamline users' access to the facility and provide optimal support for the installation and operation of experiment equipment, and communicating to the community at large about the research capabilities of the facility, results from the scientific program, and R&D activities.

Working closely with members of the Users' Executive Committee, the ALS continues to allocate its resources based on the priorities of the users. User services areas now available include a clean assembly area, a user machine shop, a chemistry laboratory, and a gas storage room. These facilities are exclusively for ALS users and are conveniently located next to the ALS experiment floor. In the works are a shipping and receiving area to help expedite delivery of user equipment, a set of loaner equipment for temporary use, and a supplemental stockroom of items that users often need on an emergency basis.

As one of the first third-generation synchrotron sources in operation, the ALS believes in communicating to others the possibilities such facilities offer, and to share the excitement of new results from the user scientific program. This information is relevant and interesting not only to the existing synchrotron community, but also to researchers unfamiliar with synchrotron science who might benefit from the ALS's research capabilities. New communication tools launched in 1994 for this purpose included the *Activity Report for 1993* (the predecessor of this document), *The ALS: America's Brightest Light for Science and Industry,* which highlights a few of the applied research and industrial applications of the ALS, and *ALSNews,* our bi-weekly electronic news bulletin. An instant success, *ALSNews* attracted over 500 subscribers in over 25 countries in its first few months, and has received an overwhelmingly positive customer satisfaction rating for its content and style.

Other outreach activities in 1994 included four workshops designed to explore research opportunities and applications using synchrotron radiation. The well-attended annual ALS Users' Association Meeting in October also provided an opportunity to learn about the scientific program and future plans of the ALS. See "Special Events" for a summary of the workshops and annual meeting.

ALS Mechanical Technician Wayne Oglesby (left) demonstrates some of the user machine shop equipment to researcher Reid Brennen.

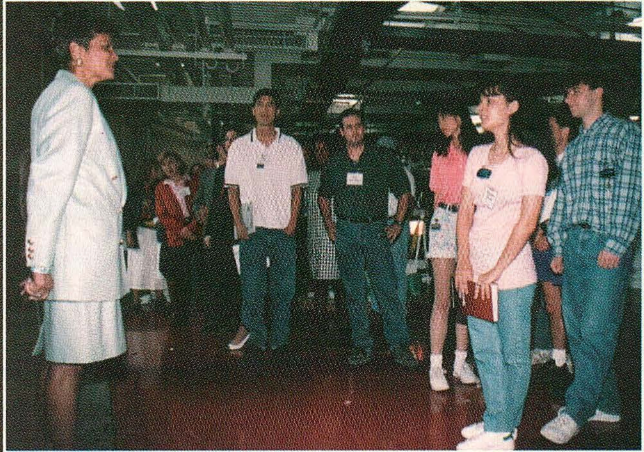


A few of the ALS publications inaugurated in 1994. The America's Brightest Light brochure received an award of "Excellence" for informational brochures in the Society for Technical Communication's international technical publications competition, after winning "Best of Category" in the Northern California regional competition.

SPECIAL EVENTS & WORKSHOPS

The ALS was honored by a visit from U.S. Department of Energy Secretary Hazel O'Leary during a meeting with her Scientific Advisory Board held at LBNL on June 30. After receiving an introduction to the ALS given by Director Brian Kincaid, the Secretary enjoyed the opportunity to talk with several of the students involved in research projects at the ALS, sharing ideas on how their work experience benefits their academic studies and their pursuit of science as a career.

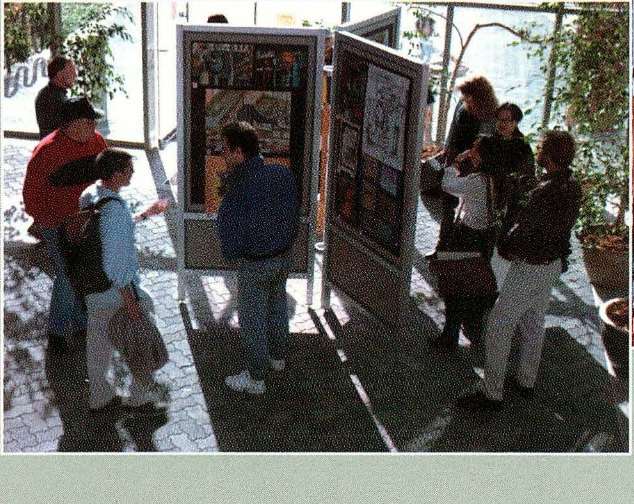
Secretary O'Leary Visits ALS



Art Exhibit Features ALS

A special exhibit of student artwork, sponsored by David Attwood (LBNL's Center for X-Ray Optics), was held at the ALS in April. The paintings and drawings originated from an evening art session held at the ALS as part of a visual arts course given by Joseph Slusky (University of California, Berkeley Architecture Department). A highlight of the exhibit's opening reception was a talk by Iran Thomas (DOE Office of Basic Energy Sciences) entitled "Creativity in Art and Science."

Popular subjects featured in the artworks were the ALS building and beamline equipment, such as the drawing of the Beamline 9.3.2 monochromator by Co Chau which appeared on the cover of *Synchrotron Radiation News*.

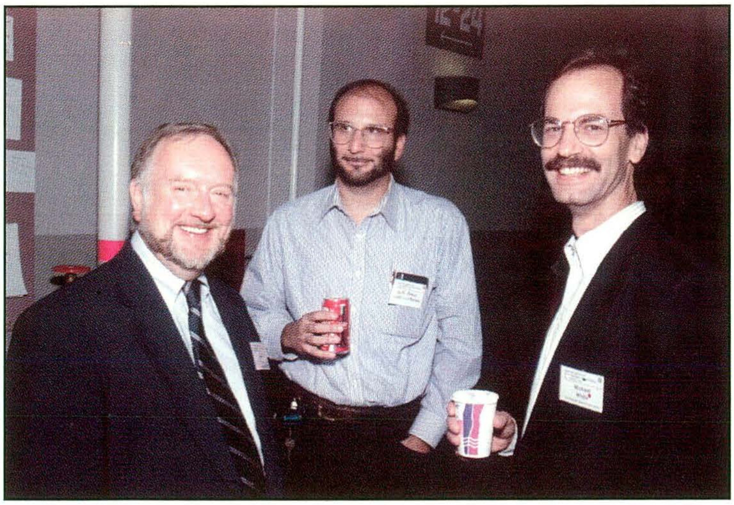


Users' Association Meeting

News of the exceptional science emerging from the first year of the ALS user program drew a record crowd of over 250 to the Annual Meeting of the ALS Users' Association on October 20 and 21. Five new user beamlines had joined those available a year before, and it was clear that promises of "unique research opportunities" and "experiments not possible anywhere else" made at the inception of the ALS are indeed coming true.

Organized by Users' Executive Committee Chair Michael White (Brookhaven National Laboratory), the program began with remarks from Bill Oosterhuis (DOE Office of Basic Energy Sciences), who congratulated the ALS for having exceeded user expectations and described efforts to increase ALS funding in FY96. ALS Director Brian Kincaid expanded on the budget theme by detailing how the ALS would use the proposed additional funds; for instance, a 10% increase above President Clinton's FY95 budget would allow the ALS to reach a 7-day operating schedule, increasing user shifts from 9 to 16 per week (a 78% increase) while improving user support services.

The rest of the morning featured an overview of ALS operations, beamline construction and commissioning activities, and scientific program development given by ALS management. Ben Feinberg, Head of Operations, described recent work at the ALS designed to improve operations by tracking down and eliminating causes of down time. These efforts produced a 93% efficiency rating (actual/scheduled user beamtime) for April–September 1994.



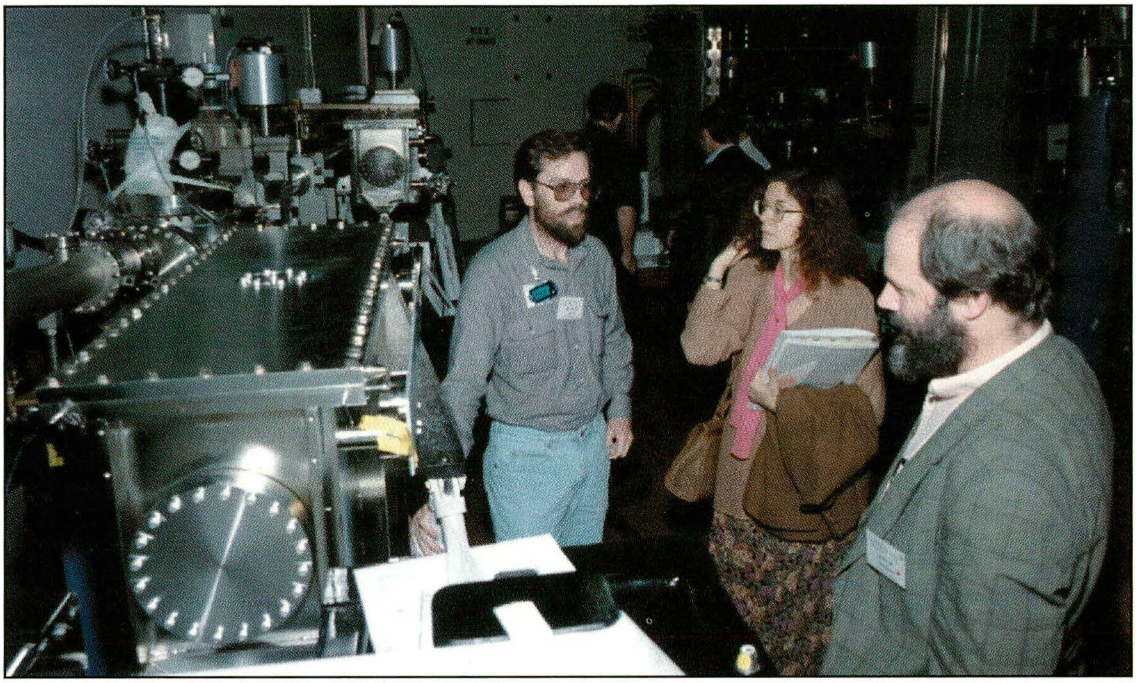
Michael White (right), chair of the Users' Executive Committee (UEC) for 1994, chats with Neville Smith (left) and Jeffrey Bokor, vice-chair of the UEC for 1995.

Accelerator Group Leader Alan Jackson described how the ALS has responded to user needs by operating the storage ring over its full energy range of 1.0–1.9 GeV, improving single-bunch purity, and using feedback systems to correct multibunch instabilities. Howard Padmore, Experimental Systems Group Leader, highlighted the successful commissioning of undulator Beamlines 7.0 and 9.0.1 and bend-magnet Beamline 9.3.2. ALS Scientific Program Head Neville Smith concluded the session by giving research highlights from some of the first operating beamlines and describing industry involvement at the ALS. Lunchtime brought an opportunity for participants to explore the ALS experiment floor and vendor exhibits.

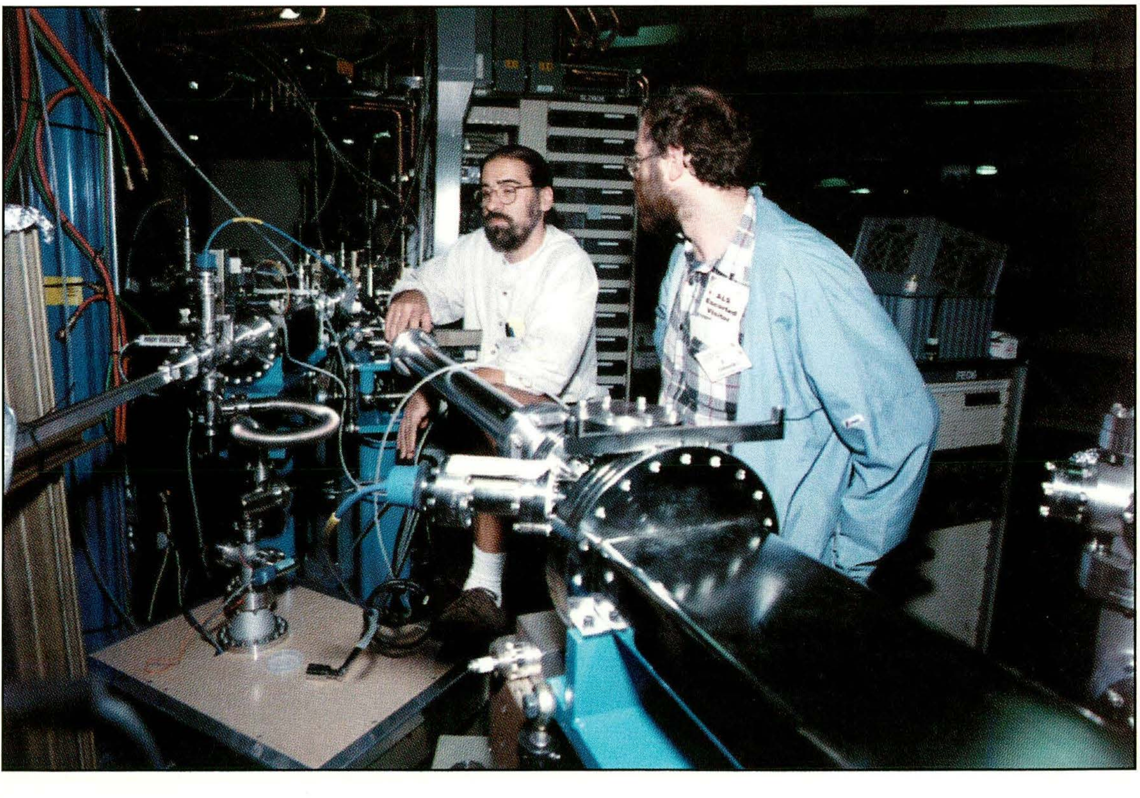
Spotlight on New Results

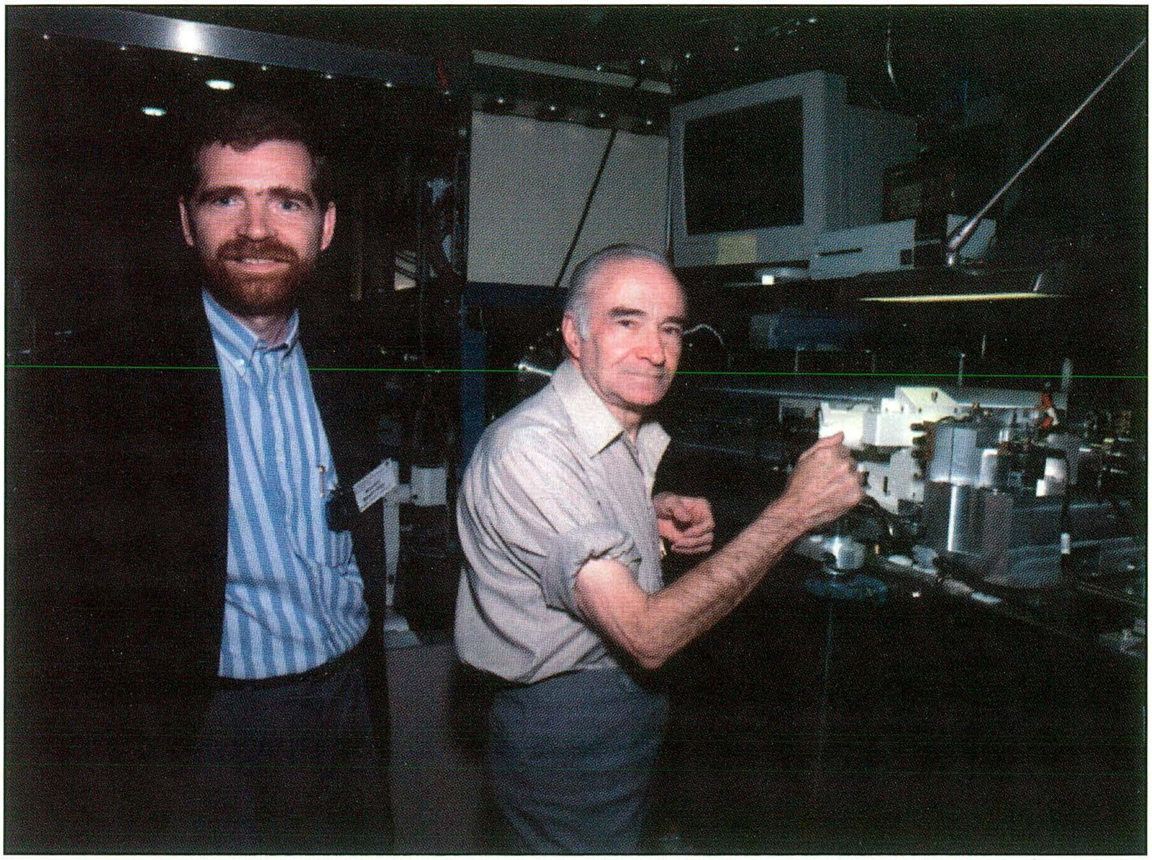
The afternoon session was devoted to new results from ALS users. Brian Tonner (University of Wisconsin, Milwaukee) began by summarizing developments at Beamline 7.0's SpectroMicroscopy Facility, where photoemission, photoelectron diffraction, soft x-ray fluorescence, and soft x-ray microscopy are being used to study detailed electronic structures of a variety of materials. Joseph Nordgren (Uppsala University) discussed his research group's high-resolution soft x-ray fluorescence studies with buckyballs, benzenes, and substituted benzenes, with a particular focus on distinguishing the symmetries of molecular orbitals in these molecules.

Anders Nilsson (Uppsala University) described his research team's collaboration with IBM to commission a new endstation at Beamline 8.0 and to study resonant photoemission processes in metal systems, with the goal



Meeting participants enjoyed the opportunity for a closeup view of the beamlines on the ALS experiment floor. (Above) Dennis Lindle, PRT Spokesperson for Beamline 9.3.1, explains its features to Novella Piancastelli (University of Rome) and John Hepburn (University of Waterloo). (Below) John Bozek of the ALS discusses Beamline 9.0.1, used primarily for atomic and molecular physics research, with Denis Cubaynes (University of Paris).





One highlight of the meeting was the presentation of the second annual Halbach Prize for outstanding instrumentation in the field of synchrotron radiation at the ALS. This year's winners were Werner Meyer-Ilse and Héctor Medecki (both of LBNL's Center for X-Ray Optics) for their x-ray microscope, the XM-1. Its design allows users to alternate visible-light and x-ray microscopy for superior resolution with minimal damage to samples.

of gaining a better understanding of multilayer interfaces. Adding to the good news from this beamline, Tom Callcott (University of Tennessee) reported on studies which have taken advantage of the ALS's brightness to map band structures, characterize chemical bonding in buried monolayers, and generally expand the previous limits of fluorescence studies.

Denise Caldwell (University of Central Florida) reported on her group's results from high-resolution photoemission studies of simple atoms and molecules using Beamline 9.0.1. Their photoelectron spectra from gas-phase experiments, with resolutions rivaling those of absorption spectra from other facilities, promise new opportunities in atomic physics. Keith Jackson (LBNL's Center for X-Ray Optics) outlined progress at LBNL in micromachining using deep-etch x-ray lithography. ALS Beamline 10.3.2 had just been commissioned for this work, after demonstration experiments at neighboring 10.3.1.

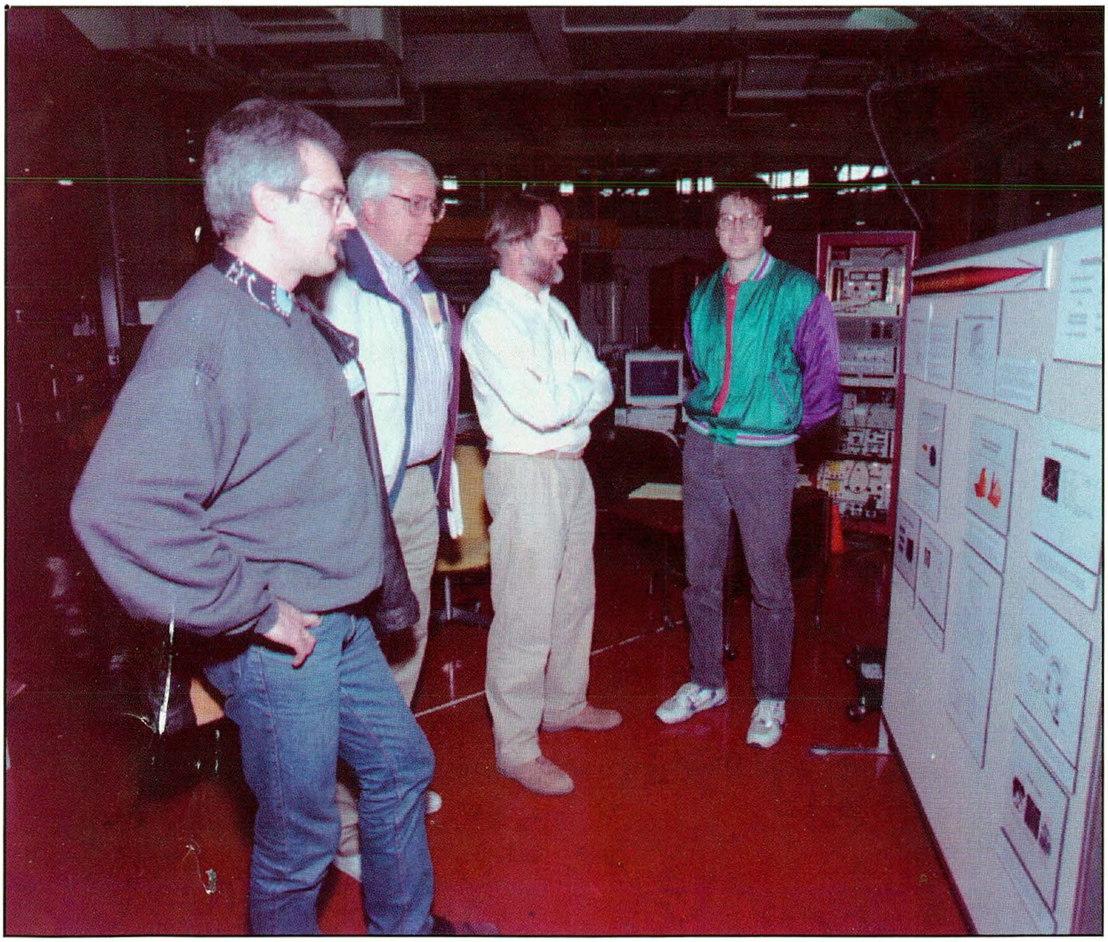
The first day's activities culminated in a banquet, where speaker Jay Davis (Lawrence Livermore National Laboratory) provided a first-hand account of his experiences as a member of the United Nations inspection team in Iraq in his talk, "Nuclear Non-Proliferation and the UN Nuclear Inspections of Iraq."

A Look Toward the Future

The theme on the second day was future scientific opportunities at the ALS. François Wuilleumier (University of Paris) began by speaking about experiments in atomic physics that would make use of the high brightness, flux, and polarization of ALS undulator beams. A talk by Robert Stroud (University of California, San Francisco) described applications of synchrotron-based protein crystallography to rational drug design. He pointed out several advantages of the ALS as a source for x-ray diffraction studies, including the increase in speed of data collection compared to conventional laboratory x-ray sources, and the advantage of a tunable source in the technique of multiple-wavelength anomalous diffraction (MAD) to determine phases directly from diffraction amplitudes.

Specific industrial applications for ALS light were the focus of talks by Richard Brundle (an independent consultant, formerly of IBM/Almaden) and Harald Ade (North Carolina State University). Brundle enumerated several advanced materials applications for synchrotron radiation, including those in the semiconductor and magnetic recording industries, and described what changes will be necessary for the national synchrotron facilities to become effective partners with industry in these fields. Ade then spoke on the potential of x-ray spectromicroscopy to solve industrial problems involving polymers.

Gwyn Williams (National Synchrotron Light Source) focused on infrared microscopy, describing an ALS bend-magnet infrared beamline scheduled for availability in early 1996. Infrared techniques are known to be valuable research tools in materials science, and can be even more effective using a synchrotron source. The final speaker, ALS User Liaison Group Manager Fred Schlachter, offered details on the proposal process and new services for users, and invited everyone to attend the poster session after lunch. Attendees, crowding around poster displays and asking questions, showed their intense interest in the work already being done at the ALS, and many looked forward to proposing their own research to take advantage of its bright beams. (A summary of the meeting presentations is available from the ALS upon request.)



The poster session allowed attendees to learn about recent scientific results from research groups working at the ALS.

Workshops

International Workshop on Photoionization

The ALS hosted over 100 photoionization experts from 16 countries at the third International Workshop on Photoionization in San Francisco on October 24–27. Organized by François Wuilleumier (a member of the ALS Program Advisory Committee) and Fred Schlachter (ALS), the workshop provided a forum to discuss the status of atomic and molecular photoionization research, including prospects for advances in the field using third-generation synchrotron light sources such as the ALS.

Both synchrotron light sources and lasers have fueled rapid advances in photoionization, contributing to exciting results in atomic and molecular physics. Workshop participants expressed enthusiasm about the active interplay between experiment and theory, and about the important advances photoionization is stimulating in the fundamental understanding of atomic and molecular processes. One important area of progress is electron correlation, which involves looking at atoms as whole entities rather than in a simplified single-particle model.

The results presented at IWP94, including talks by John Bozek (ALS) on the ALS gas-phase undulator beamline (9.0.1) and by several recent ALS users, gave participants a sampling of the exciting new science in atomic and molecular photoionization which is expected to come from all the third-generation synchrotrons. These include soft x-ray sources such as the ALS and ELETTRA, which cover the photon energy range from the visible to a few keV, and new high-energy sources such as ESRF, APS (available in 1996 for research), and SPring-8 (available in 1998).

Infrared Microspectroscopy Workshop

The successful implementation and utilization of the U2B infrared microspectroscopy beamline on the VUV ring at the National Synchrotron Light Source (NSLS) for the study of a wide range of problems in materials science has raised the possibility of installing a similar facility at the ALS. To explore this issue, an informal workshop chaired by Gwyn Williams (NSLS) was held on October 21 to review the performance of the NSLS beamline and discuss the possibility of an infrared (IR) beamline at the ALS.

Infrared spectroscopy has been a mainline analytical tool for decades, both in industry and in the laboratory, owing to its ability to identify molecular constituents of complex materials from their vibrational spectra (molecular fingerprints). In the last decade, advanced optics and detectors have made spatially resolved infrared spectroscopy (microspectroscopy) a popular technique for the analysis of inhomogeneous samples and small particles. IR spectromicroscopes have been limited in resolution because of the low brightness of a black-body source.

Synchrotron radiation, in contrast, is a very bright source of IR radiation; for example, the NSLS VUV ring, the source of the two NSLS infrared beamlines in operation, is at least 300 times brighter than a black-body source over the wavelength range from 1 to 1000 μm.

Workshop talks, primarily given by industrial users of the NSLS infrared beamline, included a comparison of results obtained using IR microspectroscopes and the NSLS VUV ring as the source, and an overview of some of the industrial research applications. The strong desire expressed by the scientific community for additional infrared beamlines led to the decision to build one at the ALS, with operation scheduled for early 1996.

Monochromator Design Workshop

Continuing a series of workshops that began at BESSY in 1991, approximately 60 specialists in the design, construction, and use of grating-based monochromators convened at the ALS on October 24–26 to survey the current status of vacuum-ultraviolet (VUV) and soft-x-ray monochromators.

From the beginning, this workshop series was designed to bring together equipment manufacturers and their customers in the synchrotron radiation community. This year several companies accepted the invitation to display their work, and were included in the oral presentations.

In several talks, speakers described existing facilities and reviewed the major types of grating-based monochromators—spherical grating (SGM), plane grating (PGM), and varied-line-spacing (VLS). For example, Jim Underwood (LBNL's Center for X-Ray Optics) presented recent results with a VLS monochromator in which the spectral resolution at the N_2 1s edge in the gas phase approached the state of the art. The monochromator, which was designed by a BESSY/LBNL collaboration, has since been shipped to Berlin after completion of testing at LBNL, and a similar monochromator optimized for the ALS is now in operation at ALS Beamline 6.3.2.

Other hot topics at the workshop included multilayer coatings to enhance the efficiency of optical elements and/or to modify the state of polarization, bendable mirrors with variable focusing, x-ray interferometry, and micro-focusing and imaging.

At the conclusion of the workshop, meeting participants concluded that no single monochromator design clearly leads the others in performance, and that a variety of designs would continue to be constructed. Conference organizers were Wayne McKinney, Howard Padmore, and Malcolm Howells at the ALS, and Fred Senf, Bill Peatman, and Wolfgang Gudat from BESSY.

Analytical Applications Workshop

At a workshop co-sponsored by the Stanford Synchrotron Radiation Laboratory (SSRL) and the ALS on October 19, scientists gathered to discuss the new SSRL beamline for molecular-level analysis of chemical contaminants in diverse environments, and plans to develop facilities for measurement of microcontamination of silicon wafers (eventually as a service to the semiconductor industry).

The morning session featured talks on the potential of the comparatively new field of molecular environmental science to play an important role in radioactive and other waste cleanup, using elemental and chemical sensitivity. A review of recent experiments indicated that x-ray absorption spectroscopy, particularly EXAFS (extended x-ray absorption fine-structure spectroscopy) and XANES (x-ray absorption near-edge structure), appears likely to be one of the main techniques employed in environmental science studies.

The second major focus of discussion at the workshop was to promote discussion on the issues related to the detection of surface contaminants on integrated circuits. Sematech, the collaborative research organization of the U.S. semiconductor industry, is studying the applicability of synchrotron-based total reflection x-ray fluorescence (TXRF) for wafer screening prior to production runs. Piero Pianetta (SSRL) reviewed plans underway at both SSRL and the ALS for establishing dedicated TXRF facilities with advanced detectors and wafer handling capability.

Analysis of some kinds of wafer microcontamination can benefit from spatially resolved spectroscopy with chemical state sensitivity. Tony Warwick (ALS) described both scanning and full-field imaging spectromicroscopy endstations present and planned at ALS undulator Beamline 7.0, as well as a dedicated bend-magnet Beamline 7.3 that will be optimized for full-field imaging with high spatial resolution and a circular polarization capability.

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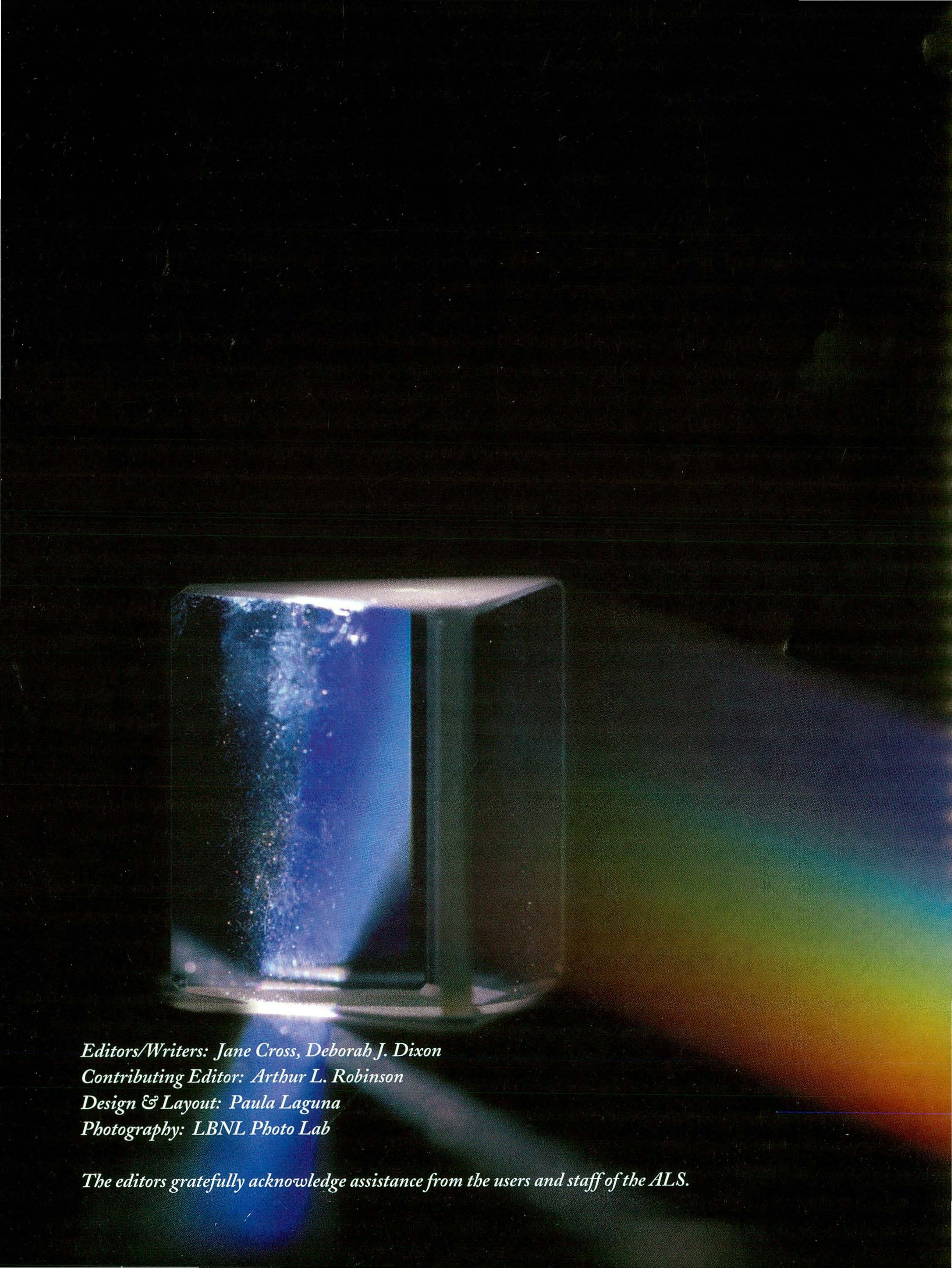

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