

EXPERIMENTAL SYSTEMS

The ALS floor bustles with the activity of scientific discovery, as our beamlines and branchlines grow in number. Eighteen were operating as of mid-1997, each delivering synchrotron radiation with unique qualities tailored to the users' specific research goals. The needs of our users, present and potential, drive the efforts of the Experimental Systems Group. In conjunction with the Mechanical and Electrical Engineering Groups, we develop insertion devices and beamline instrumentation to keep ALS users on the leading edge of synchrotron radiation research.

MACROMOLECULAR CRYSTALLOGRAPHY FACILITIES

Among the beamlines completed during 1996/97 was Beamline 5.0, the first at the ALS dedicated to macromolecular crystallography. This technology, which requires a high flux of high-energy photons, is made possible by the beamline's source, a 38-pole wiggler, also new to the ALS. Built by the Mechanical Engineering Group and installed in April 1996, it is the fifth ALS insertion device and the first wiggler. We have been operating the wiggler at full power (1.9 GeV, 400 mA, 2.1 tesla). The beamline, which was built in conjunction with Berkeley Lab's Structural Biology Division, is currently being commissioned.

SPECTROMICROSCOPY OPTIONS

Also proliferating is our spectromicroscopy program, which boasts several new facilities. The scanning transmission x-ray microscope (STXM) on undulator Beamline 7.0.1 has now been complemented with an ultra-high-vacuum zone-plate microscope known as SPEM (scanning photoemission microscope). Like STXM, SPEM features micro zone-plate focusing, but uses an electron energy analyzer as a detector. Placing the zone-plate scanning stage in ultra-high vacuum and close to the analyzer was a real engineering challenge, but results indicate 0.3- μm resolution as expected, and initial studies on interface

formation have yielded spectacular data.

Another spectromicroscopy beamline, bend-magnet Beamline 7.3.1, is now complete and in use. This beamline has two branches, one wide-aperture branch (7.3.1.1) for a photoelectron emission microscope (PEEM) and a second branch (7.3.1.2) for microfocused x-ray photoelectron spectroscopy ($\mu\text{-XPS}$). The $\mu\text{-XPS}$ focusing system uses a pair of elliptically shaped grazing-incidence mirrors, produced by controlled bending within the ultra-high vacuum of the beamline. The microscope is designed to perform scanning XPS on semiconductor wafers with 1- μm -spatial-resolution. (To date, we have achieved 2- μm resolution in preliminary tests.) A co-development with Intel Corporation and Applied Materials, $\mu\text{-XPS}$ has many features especially designed for wafer samples, such as *in situ* optical fiducialization.

The photoelectron emission microscopy branchline (7.3.1.1) is essentially complete, and the PEEM system itself has a completion date of October 1997. The PEEM operates at high voltage (30 kV) and has two projector stages, a stigmator and several deflectors to keep the beam passing through the center of the lenses, and a directly coupled phosphor-fiber CCD detector. The branchline also offers *in situ* sample preparation and sophisticated sample-manipulation capabilities. A thorough theoretical study has shown that the system should be capable of 30-nm resolution. This instrument is being developed with IBM as part of a cooperative research and development agreement. We are also developing plans for another PEEM that will compensate for spherical and chromatic aberrations of the electron optics and should be capable of 3-nm resolution. This project is a joint effort with teams at IBM and Arizona State University. The system will ultimately use Beamline 4.0.3-4, which will receive light from an elliptical polarization undulator. Operation is scheduled for early 1999.

INFRARED MICROSCOPY SYSTEM

The infrared microscopy beamline (Beamline 1.4) has recently been completed, and commissioning is under way. The system consists of plane and ellipsoidal mirrors inside the storage ring shield wall to focus the radiation through a diamond isolation window, a system of collimating mirrors, and a commercial Nicolet interferometer and microscope. The light it delivers has a wavelength range of 2–35 μm , corresponding to the chemical fingerprinting range for organic species. This beamline will be used for a variety of chemical mapping studies, from identification of particles on silicon wafers to environmental studies of humic material.

DELIVERY SYSTEM FOR ELLIPTICALLY POLARIZED X RAYS

Beamline 4.0.1-2, the first of the beamlines designed to deliver x rays from the elliptical polarization undulator, is now taking shape, and we have contracted for construction of its high-power monochromator. Designed to deliver x rays with energies from 20 eV to 1600 eV, this beamline is expected to start operations during the summer of 1998. It will offer very high resolving power for studies on magnetic systems. We have now begun the optical design of a second monochromator, which will be optimized for high-spatial-resolution microscopy.

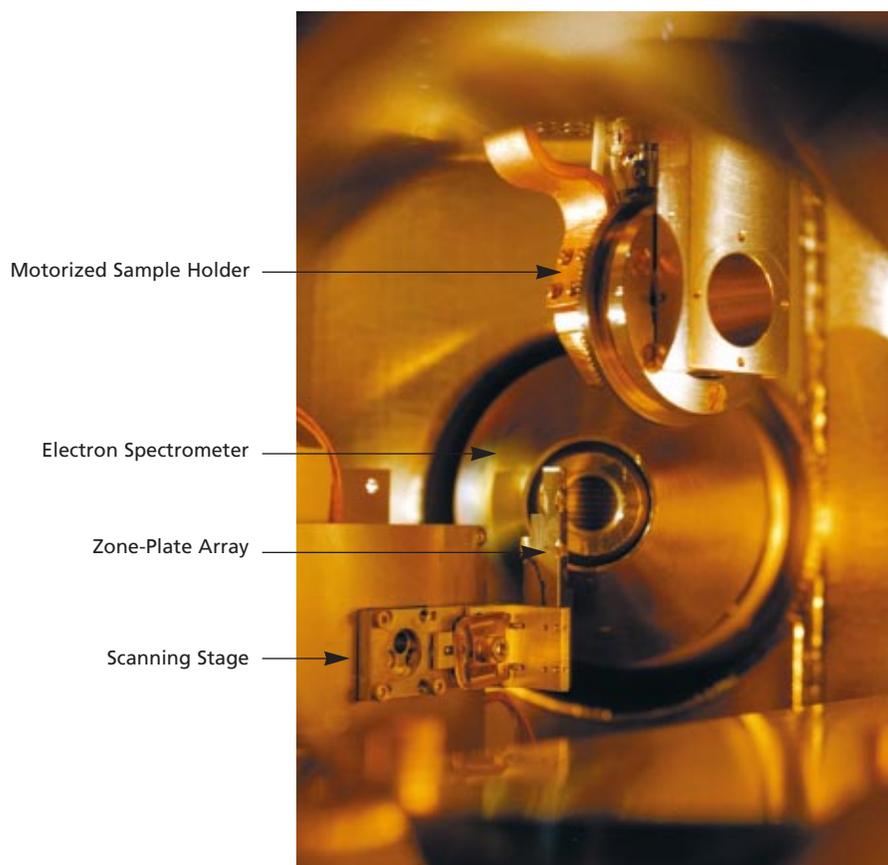
ONGOING ACTIVITIES IN BEAMLINE INSTRUMENTATION AND ENGINEERING

Development of advanced photoelectron emission microscopes (PEEMs) for high-resolution studies of materials such as polymers and thin carbon films (used in computer disk drives).
Commissioning of Beamline 1.4 for infrared microscopy. Potential uses range from chemical fingerprinting studies to investigations of surface adsorption and semiconductor defects.
Design and construction of the elliptical polarization undulators and the associated beamline (4.0) for microscopy and spectroscopy of magnetic materials.
Commissioning of Beamline 5.0 for macromolecular crystallography studies, such as determining the structures of proteins (see "Macromolecular Crystallography," p. 42).
Development of spectromicroscopy, including refinement of the new scanning photoemission microscope (SPEM) on Beamline 7.0.1 and μ -XPS on Beamline 7.3.1.2 (see "Scanning Photoemission Microscopy," p. 38, and "Micro X-Ray Photoelectron Spectroscopy," p. 40).
Design and construction of Beamline 7.3.3 for thin-film strain measurements and spatially resolved x-ray absorption near-edge spectroscopy (XANES).
Optics research for the development of optical metrology, holographic imaging, optical design, switching optics, bendable mirrors, and microfocusing systems.
Development of instrumentation for femtosecond x-ray diffraction studies.
Design and construction of a high-energy-resolution spectrometer (HERS) for photoemission studies of highly correlated systems.
Design and construction of a bend-magnet beamline for scanning transmission x-ray microscopy.
Commissioning and refinement of a system for Fourier transform interferometric spectroscopy at ultra-high resolution (Beamline 9.3.2).

SCANNING PHOTOEMISSION MICROSCOPY

A new high-resolution scanning photoemission microscope (SPEM) began operation at the ALS in March 1997. This instrument opens new avenues for spectromicroscopy research at the ALS. It combines the features of the scanning transmission x-ray microscope—which uses a Fresnel zone-plate lens to produce a microfocus for spectromicroscopy studies—with an electron energy analyzer in an ultra-high-vacuum environment. In designing and building such a system, our aim was to combine these attributes with 0.1- μm spatial resolution.

The new system has a unique method of scanning samples. Unlike other photoemission microscopes, in which the sample is moved in order to form a scanned image, SPEM forms images by moving the focused x-ray beam over a stationary sample surface. It is the first zone-plate photoemission microscope to operate in this way. Keeping the sample stationary makes it possible to use a large, conventional sample manipulator. Thus, researchers using the SPEM will be able to study samples that are cryogenically cooled or heated or to examine



Inside the scanning photoemission microscope, the sample sits on a motorized assembly that lowers it into position in front of the zone-plate array. This array contains three different zone plates, with their corresponding order-sorting apertures, arranged step-wise so that each is correctly positioned for its focal length. The zone-plate array attaches to a scanning stage, which moves the x-ray beam across the stationary sample.

small regions of large (25-mm) samples without having to cut the sample apart. SPEM features a 16-channel electron energy analyzer, which allows detailed spectroscopic analyses to be performed at points on the surface where the photoelectron image shows interesting features.

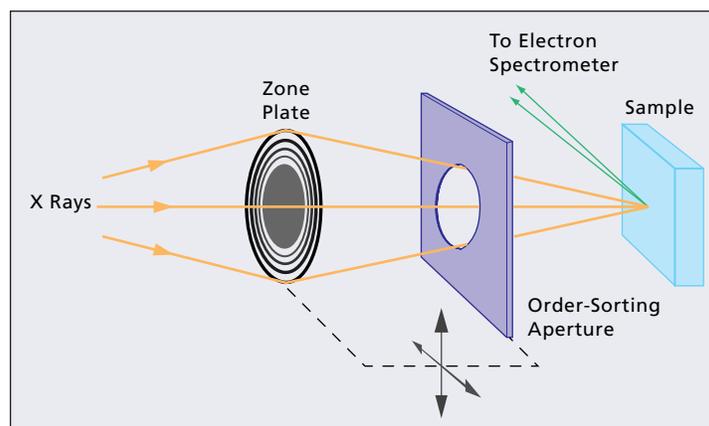
The new SPEM uses Fresnel zone-plate lenses a quarter of a millimeter in diameter to focus the x-ray beam. These lenses are made by electron-beam lithography and plated with gold to form a diffracting ring structure. The SPEM scans the lens in a raster pattern half a millimeter in front of the sample, moving the focused x-ray beam across the surface. Each lens is accompanied by an order-sorting aperture, which prevents zero-order light from reaching the sample. The aperture must move with the lens and is therefore built into the same assembly at a fixed focal length. Since the appropriate focal length varies with photon energy, we have designed the instrument to accept different assemblies for the various photon energy ranges of interest. The photon energy used in the SPEM can vary from about 200 eV to 900 eV, offering

researchers the flexibility of studying microscopic changes in valence band structure as well as performing more traditional core-level spectroscopy.

Early studies with SPEM have focused on problems in composite materials, imaging features a few microns across. In addition, metallurgical problems are particularly amenable to the scanning photoemission technique, as the samples are electrically conducting and can be polished. Electrically insulating samples from the semiconductor industry have been studied as well.

In its commissioning period, SPEM has demonstrated an x-ray spot size of about $0.3\ \mu\text{m}$. Spatial resolution of $0.2\ \mu\text{m}$ is expected from the zone plates in use now, with improvements of a factor of 2 or more as finer zone plates become available. The instrument's commissioning continues, and we expect it to be fully operational by the end of 1997.

Funding: Office of Basic Energy Sciences of the U.S. Department of Energy, National Science Foundation, University of Wisconsin at Milwaukee, and Lawrence Livermore National Laboratory.

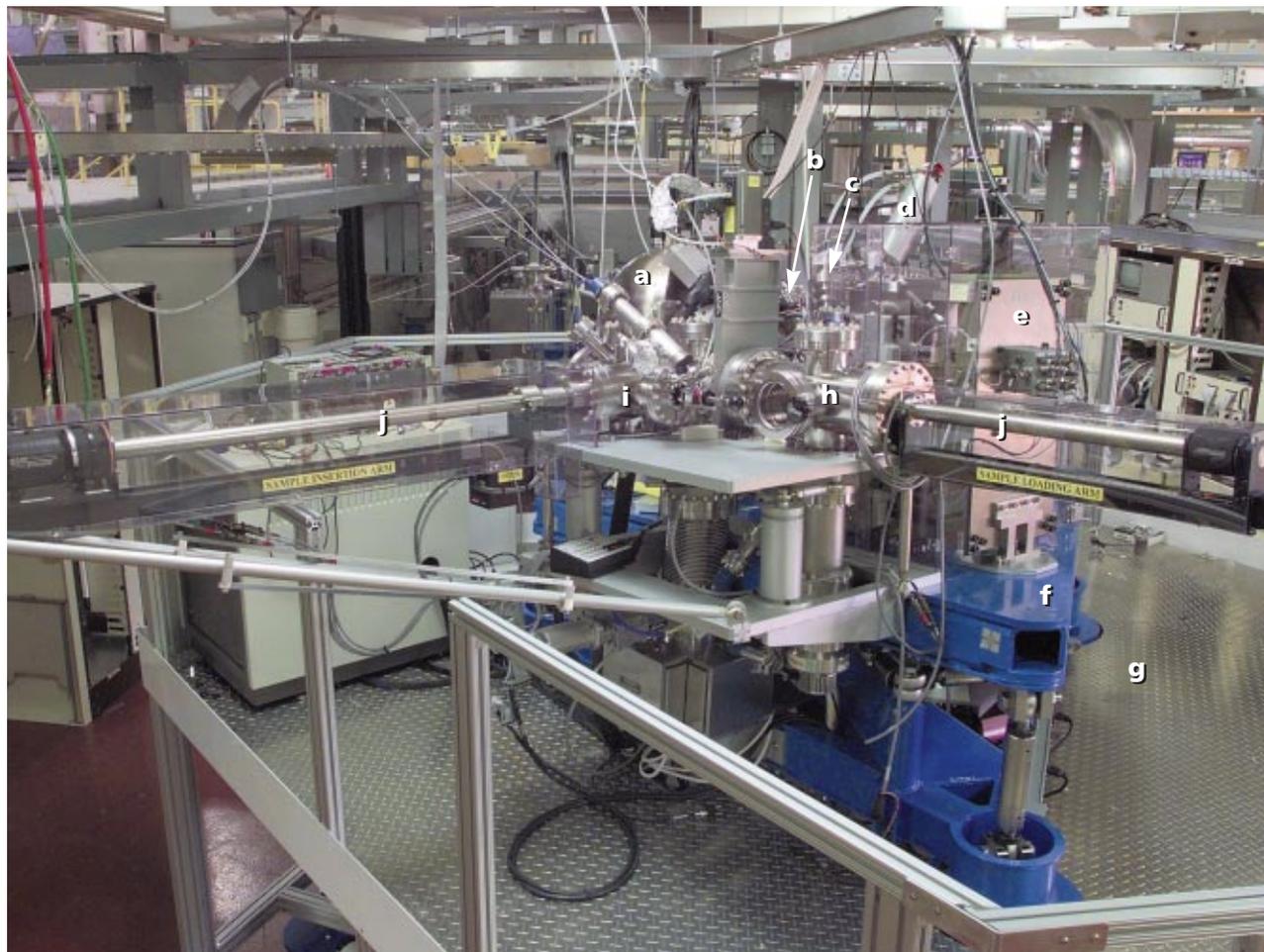


Schematic layout of the scanning photoemission microscope. The zone plate and the order-sorting aperture move in tandem to raster the x-ray beam over a stationary sample.

MICRO X-RAY PHOTOELECTRON SPECTROSCOPY

On January 23, 1997, the new micro x-ray photoelectron spectroscopy (μ -XPS) beamline reached a significant milestone: seeing its first synchrotron light. A joint development with Intel Corporation and Applied Materials, the μ -XPS project grew out of the need, identified in the Semiconductor Industry Association Roadmap, for a technique capable of surface chemical mapping at high spatial resolution. The project was conceived as an XPS system capable of 1- μ m spatial resolution on a bend-magnet source.

Achieving such high spatial resolution for surface chemical studies has not been possible with existing laboratory techniques. Infrared spectro-microscopy has limited sensitivity and spatial resolution but good selectivity for organic species. Scanning Auger microscopy (SAM) has excellent spatial resolution (0.1 μ m), but the chemical information that can be obtained is extremely limited. Sophisticated laboratory systems for scanning XPS exist, but though the best offer 10- μ m resolution, imaging times are very long because of the limited



The micro x-ray photoelectron spectroscopy endstation, showing (a) electron energy analyzer, (b) partial-electron-yield detector, (c) sputtering gun, (d) laboratory hard-x-ray source, (e) precision x-y sample manipulator, (f) six-strut chamber support, (g) independently supported "people platform," (h) sample introduction and parking area, (i) sample preparation and transfer area, and (j) magnetic sample transfer arms.

brightness of the source. Our goal, therefore, was to design a micron-resolution system with short image-acquisition times that would take full advantage of the tunability of synchrotron radiation. The selection of photon energy is important in two respects: first, it allows the cross section of the element of interest to be maximized; and second, it allows us to vary the kinetic energy of the photoemitted electrons and hence their escape depth. In this way, we can alter the surface sensitivity and minimize radiation damage.

The μ -XPS system uses a branchline off an existing monochromator in order to reduce the total system cost. This monochromator, together with a spherical, horizontally deflecting branching mirror, delivers a monochromatic focus to an adjustable pinhole, which is the object for the μ -XPS imaging system. The microfocusing is produced by orthogonal elliptical mirrors in a Kirkpatrick-Baez arrangement. These mirrors have to be nearly perfect to produce the desired micron-sized focus and are made by the controlled bending of flats. They must be bent to extreme curvature and therefore are made of high-strength steel. Because of the high demagnification, which requires a mirror-to-sample distance of 0.1 m, the whole optical system for the endstation is in ultra-high vacuum and very close to the sample stage. This unusual space constraint added another dimension to the engineering challenge.

Another key design issue is sample positioning. The patterned wafer samples to be studied are first examined elsewhere by conventional techniques, the areas of interest are identified with reference to fiducial points, and then the samples are trans-

ferred to the ALS. Here the samples pass through a fast-entry load lock, through a prep chamber, and into the analysis chamber, in which a high-resolution *in situ* optical microscope is used to find the fiducial points. From these, the system can locate the previously identified areas of interest. The sample holder can accept up to 2-inch \times 2-inch wafer sections, and designs are being assessed for a future system capable of handling full 12-inch wafers.

The μ -XPS project was completed and began its initial commissioning only ten months after construction started. Meeting this very tight manufacturing and commissioning goal was of key importance for our industrial partners. With minor adjustments to the system, we have achieved 2- μ m \times 2- μ m resolution at full aperture. With further adjustment, possibly using the new higher-quality mirror substrates we now have available, we are confident of reaching our 1- μ m target. Since the diffraction limit of these mirrors at a photon energy of 1 keV is less than 0.1 μ m, we can expect, with improved optics and a smaller pinhole, to achieve submicron resolution (given adequate photon flux). The ultimate goal is to build a system providing resolution that is a small fraction of next-generation microcircuit line widths (0.18 μ m). If the return on investment for the present system is adequate, we could meet this challenge with an undulator-based system capable of 0.1- μ m resolution.

Funding: Office of Basic Energy Sciences of the U.S. Department of Energy, Intel Corporation, and Applied Materials.

MACROMOLECULAR CRYSTALLOGRAPHY

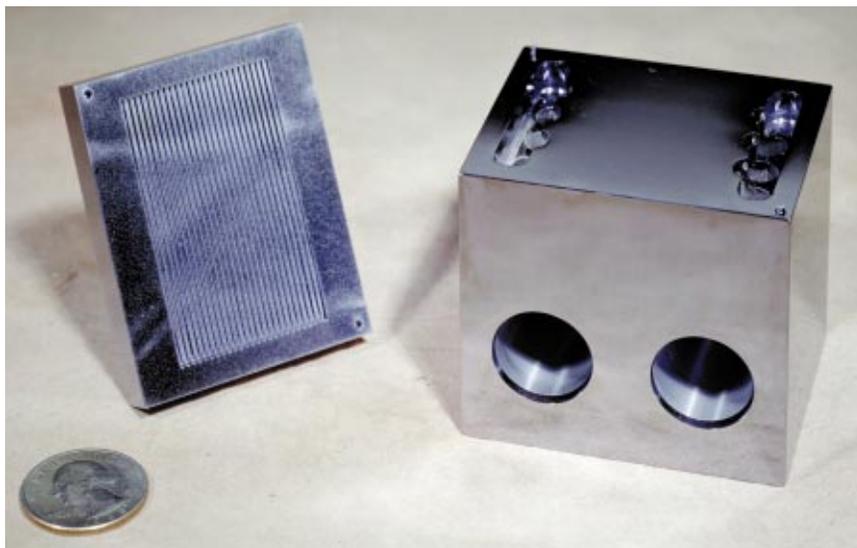
The end of 1996 marked an important achievement for the Experimental Systems Group, with the December delivery of first light to the macromolecular crystallography beamline. Developed in collaboration with Berkeley Lab's Structural Biology Division, the beamline forms the heart of the new Macromolecular Crystallography Facility at the ALS, which offers structural biologists from industry, government, and academia a choice of crystallographic techniques with semi-automated operation and rapid sample turnaround.

The light source for the beamline is a 38-pole, 2.1-tesla wiggler, emitting 8.4 kW at 1.9 GeV, 400 mA. Engineering a beamline to handle the heat load from such a powerful wiggler source has required several tactics. First, the soft x rays emitted by the wiggler are absorbed by a series of thin carbon filters. These reduce the power falling on the first optical elements substantially. Second, the optics themselves have been designed to handle a high flux of hard x rays.

The focusing arrangement for the completed central branchline is conventional in that a remotely adjustable mirror upstream of the monochromator

(inside the storage ring shield wall) provides vertical collimation, the monochromator is a double-crystal arrangement with a fixed exit height, and a toroidal mirror downstream focuses the light to a small spot in the hutch. Because of problems at the optics vendor contracted to make these mirrors, we decided to install temporary mirrors of reduced aperture while new full-size replacement mirrors are fabricated. The first temporary mirror is a silicon flat with simple internal cooling, and the second is a silicon toroid. This arrangement has allowed us to proceed with commissioning the beamline, but with a temporary decrease of about a factor of 2 in energy resolution and about a factor of 3 in flux.

Like the first mirror, the double-crystal monochromator requires cooling. In fact, its first crystal receives the highest power density of any optical component in the beamline, absorbing approximately 700 W/mrad² (up to 2.5 W/mm² at a Bragg angle of 30°). To preserve spectral resolution, any thermal distortion must be kept to a small fraction of the crystal rocking width (roughly 40 μrad for silicon at 10 keV). In order to achieve this under extreme power loading, the crystal is internally water-cooled



The water-cooled first crystal of the central branchline's monochromator is made up of two pieces of silicon. The thinner piece, one side of which serves as the diffracting surface, has 1-mm water channels machined into the reverse side (left). This piece is then frit-bonded, channel side down, to the second silicon block (right).

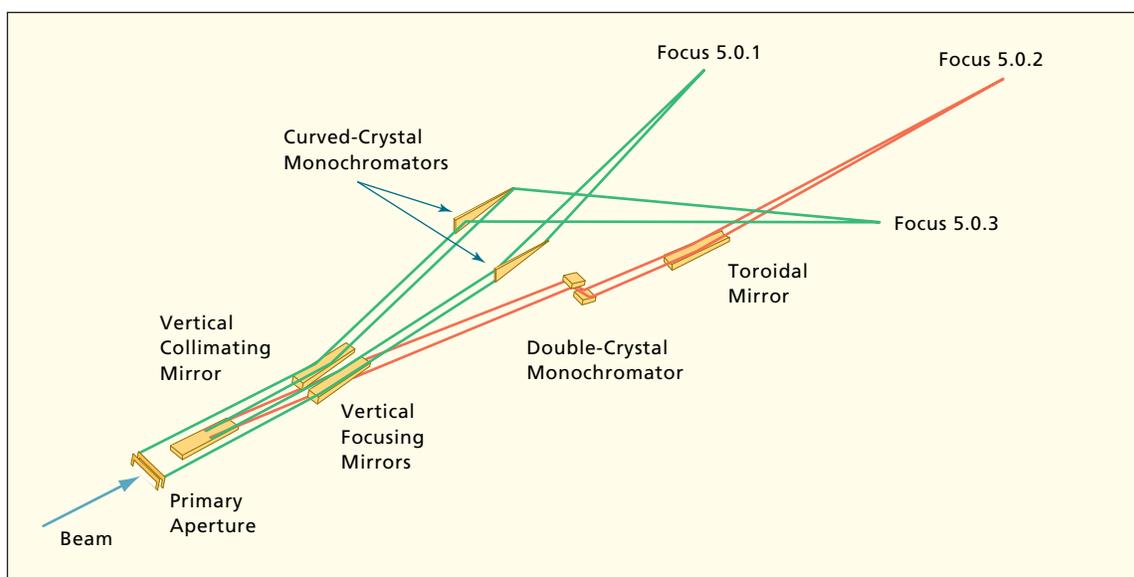
through 1-mm-wide channels machined into the crystal within 1 mm of the diffracting surface. In contrast to undulator beamlines at third-generation hard-x-ray sources, where liquid-nitrogen cooling is used, we chose to use water cooling because the monochromator receives a much lower power density even though it absorbs more total power.

The beam focused into the central branchline's hutch passes through an aperture, a flux monitor, an anti-scatter collimator, and a transport tube—all helium-filled to minimize x-ray absorption—before intercepting the crystal to be measured. Diffracted x rays are detected by a 2×2 -matrix CCD detector that is mounted on a slideway so that users can vary its distance from the sample. The slideway can also be rotated in order to collect the very high-resolution data sometimes available from exceptional crystal samples.

We are now developing up to two side stations that will have asymmetrically cut, curved-crystal

monochromators. The wiggler has been run at full power (14 mm gap, 2.1 tesla, 1.9 GeV, 400 mA), and the beamline has transported monochromatic light into the central branchline's hutch under these conditions. In the summer of 1997, we started the detailed characterization of the system, measuring resolution, flux, spot size, and stability. From our initial measurements, the existing beamline appears to be performing to specification. The combination of functioning beamline, state-of-the-art CCD detector, and accompanying crystallography support laboratories means that, overall, we now have one of the best facilities for biological crystallography in the world, and we can look forward to many years of successful operation.

Funding: Office of Biological and Environmental Research of the U.S. Department of Energy, Amgen, Roche Biosciences, University of California at Berkeley, and Berkeley Lab.



Optical layout of the macromolecular crystallography beamline (Beamline 5.0). Light in the central 1.5 mrad of the wiggler beam is reflected by a vertical collimating mirror toward the double-crystal monochromator and hence to the rest of Branchline 5.0.2. When the proposed side branchlines are completed, each one will take 3 mrad of light to one side of the beam's central area. This light will be reflected upward by a vertical focusing mirror to reach the corresponding branchline optics (Branchline 5.0.1 or 5.0.3).