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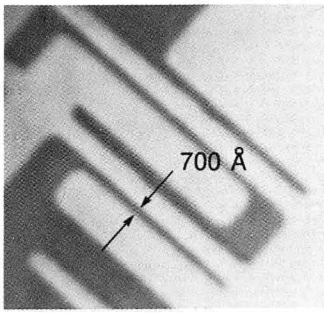

CENTER FOR X-RAY OPTICS

. . . to make those capabilities widely and readily available . . .

IN 1988, THE CENTER FOR X-RAY OPTICS continued its two complementary roles: demonstrating the capabilities and usefulness of the x-ray and ultraviolet regions of the electromagnetic spectrum, and developing equipment and techniques to make those capabilities widely and readily available. We continued our program of collaborations with scientists outside the Laboratory through participation in research projects at several facilities. Our University Research Initiative contract enabled us to maintain a student-training effort, with both graduate and undergraduate student assistants contributing to our research projects. And we continued to participate in the organization of an Applied Science and Technology Program on the University of California-Berkeley campus.

The fabrication of Fresnel zone-plate lenses for focusing soft x-rays and the use of zone plates in x-ray microscopy systems were areas of significant achievement during the past year. In collaboration with IBM, we improved the spatial resolution of zone-plates lenses made by electron-beam lithography to the 500-Å level; then we tested their ability to image structures similar to those on future generations of advanced integrated circuits with an imaging x-ray microscope at the BESSY synchrotron radiation facility in Berlin. We also prepared for a second round of biological imaging experiments with a scanning x-ray microscope on a new beamline at the National Synchrotron Light Source (NSLS) by developing a sample-holder that will allow us to change the chemical environment of our zymogen granule samples. And while preparing for the next round of experiments, also to be conducted at the NSLS, we improved our ability to reconstruct images from holograms of zymogen granules numerically.

Continuation of our long-term effort to develop high-reflectivity multilayer coatings for XUV optical elements, such as mirrors and gratings, was a second major focus of activity. We experimented to clarify the

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structure and stability of tungsten-carbon multilayers by means of high-resolution transmission electron microscopy. By performing a theoretical study of the benefits of pre-mirrors in the next-generation synchrotron sources equipped with wigglers, we followed up experiments conducted last year at NSLS in which a multilayer-coated pre-mirror was used to reduce the power load on optical elements in synchrotron radiation beamlines. And we widened the range of sample types that could be investigated with our high-sensitivity x-ray microprobe with multilayer-coated mirrors to include spatially resolved elemental analysis of ancient documents and artwork.

Generation of the fundamental data needed to plan experiments and design x-ray optical elements has always been part of our charter in x-ray spectroscopy. During the past year we completed a major extension of our existing, widely-used tables of the real and imaginary parts of the x-ray scattering factors of the elements from Z = 1 to Z = 92, carrying them to a wider range of photon energies. A measure of our success in translating new ideas into practical spectroscopy systems was an R&D 100 award for the invention of the high-resolution, erect-field spectrometer that uses a varied-line-space grating. And we completed an investigation of the properties of candidate materials for high-sensitivity photocathode x-ray detectors.

Our synchrotron radiation projects group pioneered the development of water-cooled, metal optical elements for beamlines at advanced synchrotron light sources that generate intense beams of XUV radiation by means of wigglers and undulators. A water-cooled grating now installed in a wiggler beamline at the Stanford Synchrotron Radiation Laboratory (SSRL) is the first of its kind and complements our previous development and testing of water-cooled mirrors there and at the NSLS.

Soft-X-Ray Imaging

The extension of high-resolution imaging techniques to the soft-x-ray spectral region offers several special advantages. With wavelengths ranging from several angstroms to perhaps one hundred angstroms, this relatively short-wavelength radiation permits one to both "see" and "write" smaller patterns than are now possible. Furthermore, the associated photon energies, from approximately one hundred to several thousand electron volts (eV), span the primary resonances of many elements. Resonances constitute a sensitive mechanism for element identification, for elemental mapping, and, in some cases, for the determination of chemical bonding. During the past year, we advanced the technology of soft-x-ray imaging and demonstrated some potential applications in the physical and life sciences. The spatial resolution that we have achieved in x-ray microscopy and holographic imaging is better than 1000 Å and in some cases approaches 500 Å.

Microscopy with Fresnel Zone Plates

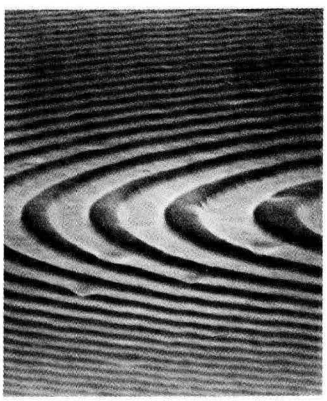
A form of x-ray microscopy that has shown particular promise is based on the use of Fresnel zone-plate lenses, which are diffractive structures of alternately transmissive and opaque circular zones whose width decreases with radial distance. Major attributes of a perfect zone-plate lens include the ability to focus distant radiation to a Rayleigh-limit spot size of 1.2 Δr (where Δr is the width of the smallest, outermost zone) and the ability to form images in the usual sense of a lens with focal length $f \approx 4N (\Delta r)^2/\lambda$, where n is the total number of zones and λ is the wavelength. (Note that such a lens is highly chromatic and that the diffractive efficiency to first order is just under 10%, even for a perfect lens.) In materials and surface science, soft x-rays make it possible to incorporate spatial resolution in photoelectron and other spectroscopic techniques. The mesoscopic dimensions resolvable with soft x-rays lie between the few Å of atomic-resolution techniques and the µm or so of visible microscopy. For those in the electronics industry and elsewhere interested in extending microfabrication techniques to ever smaller dimensions, soft-x-ray techniques offer the potential for new capabilities in both mask inspection and pattern reduction to dimensions much smaller than 1 µm. Soft x-rays also make it possible to image biological material in its natural, unaltered environment at a spatial resolution beyond that of the visible-light microscope.

As in the past, our Fresnel zone-plate lenses were fabricated at IBM's Thomas J. Watson Research Center as part of a collaborative effort in which a CXRO scientist is in residence at IBM. The effort involves a special electron-beam lithography system designed by IBM specifically for writing nanometer patterns. To accurately calibrate the field size for orthogonality and squareness, calibration marks produced by electron lithography were monitored by laser interferometry. Figure 1 shows details of the latest Fresnel zone-plate lens.

This zone plate has been used as an objective lens to image nanostructure patterns in the microscope beamline developed by the University of Göttingen's X-Ray Microscopy Group at the BESSY synchrotron radiation facility in Berlin. The x-ray optical arrangement is similar to that of a visible light microscope in the transmission mode. Broadband synchrotron radiation from a bending magnet is filtered by a zone plate monochromator before illuminating the test object. The x-rays transmitted through the test object are diffracted by the zone-plate objective lens, thereby forming an enlarged image (300× or 500×) on Kodak 2462 x-ray-sensitive film. Special test objects consisting of gold patterns on silicon nitride substrates were prepared in a manner similar to that used for fabrication of the zone plates. Patterns were selected to demonstrate the capability of imaging nanostructures relevant to future directions of the electronics industry, to test spatial resolution, and to characterize the degree of astigmatism.

The x-ray micrograph in Figure 2 illustrates the ability of zone-plate lenses to image nanostructure patterns with deep-sub-µm geometries typical of those expected in future electronic chips. The image, which was obtained with 45-Å x-rays generated by the 0.8-GeV storage ring at BESSY, shows a typical pattern detail for two levels of an experimental test circuit with 0.1-µm metal-oxide-semiconductor field-effect transistors. The pattern was specially replicated for these studies in gold on a thin x-ray transmissive silicon nitride membrane. The

Figure 1. As shown in this oblique view from a scanning electron microscope, our latest zone-plate lens consists of opaque gold zones on a transmissive silicon nitride membrane. The zone plate has 500 zones, a nominal outer zone width of 400 Å (40 nm), a bar-to-space ratio of 2:1 in the outer region, and a gold thickness of approximately 700 Å. The outer diameter of 80 µm and the focal length of 0.71 mm at a wavelength of 45 Å give it an effective aperture of F/9. The diagram at right shows how such a lens can be used for diffractive focusing of x-rays.



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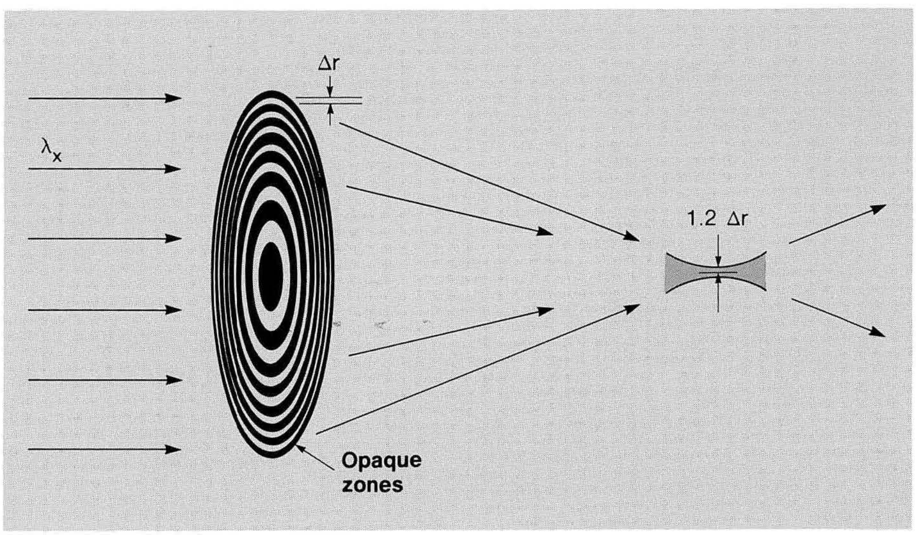

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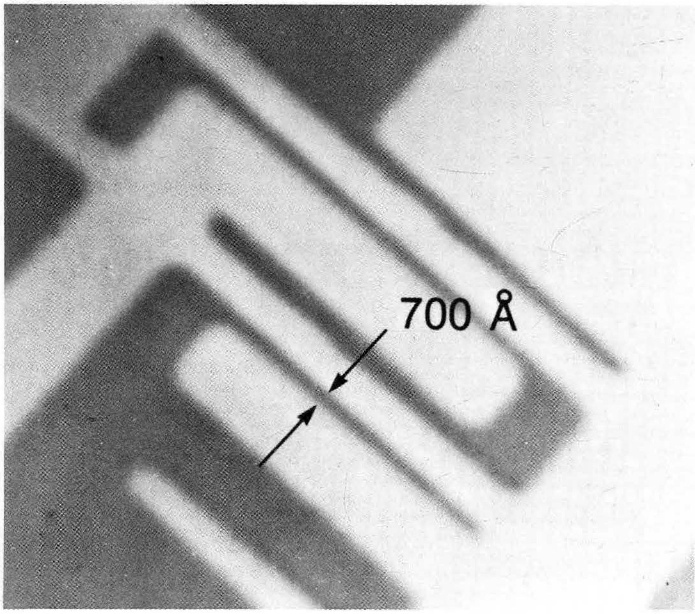

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Figure 2. This x-ray-microscope image shows fine details of a nanostructure pattern associated with a future microelectronic device of 0.1-μm feature size. (Note the clearly resolved 700-Å gap.) The experiment demonstrated the utility of x-ray optical systems for applications involving electronic structures with features much smaller than 1 μm. We are also involved in several related efforts utilizing demagnification techniques.

micrograph shows a clearly resolved 700-Å gap between gold features. Additional test objects were prepared to examine the questions of spatial resolution and astigmatism (the quality of the image in orthogonal directions).

Figure 3 shows a micrograph of a circular pattern with variable ring widths as small as 430 Å, along with a radial display of recorded film density versus position. The modulation in film density between opaque and transmissive rings is an indicator of resolution. The film density is observed to be relatively constant for x-rays passing through the large circular gaps, but it decreases for the smaller gaps located near the pattern center. Only the smallest circular gap of approximately 430-Å width shows a significant drop in modulation amplitude. The next circular gap (moving radially outward), with a width of approximately 800 Å, is close to full amplitude. Digitization and analysis of the film density show only a slightly decreased performance in the orthogonal direction. Based on analysis of these and other digitized film records, we believe that the spatial resolution of this lens of nominal 400-Å outer zone width approaches the theoretical limit (1.2 Δr) of approximately 500 Å.

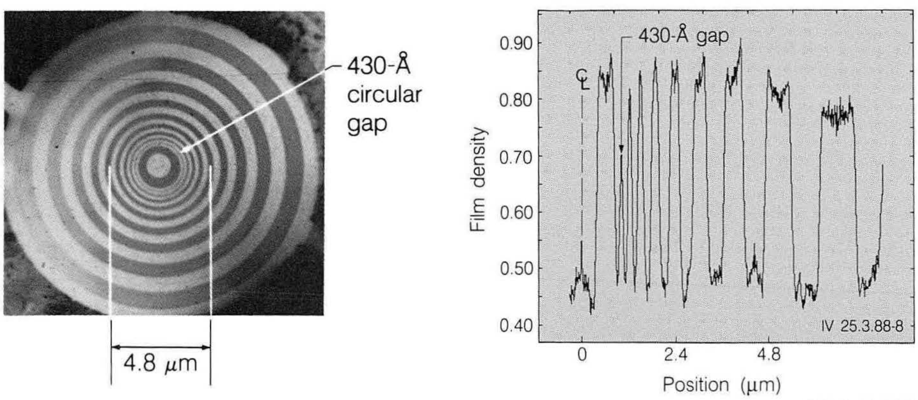

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Figure 3. When we imaged a circular pattern of varying ring width (photo), modulation amplitude was off significantly for only the smallest gap, approximately 430 Å wide, near the center, as indicated by the lack of definition of that ring in the photo and also by the amplitude drop in the graph. Performance decreases only slightly in the orthogonal direction. The decrease in film density as a function of the distance between opaque and transmissive rings provides a measure of spatial resolution.

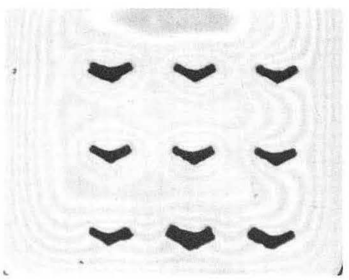
Biological X-Ray Microscopy

Last year we reported on scanning x-ray microscopy studies on the X-17T beamline at NSLS in which we and our IBM colleagues collaborated with scientists from the University of California at San Francisco and the State University of New York at Stony Brook to make images of zymogen granules. These are secretion granules isolated from the pancreas of the rat that contain 20 or so digestive enzymes. The purposes of these studies are to develop high-resolution x-ray microscopic imaging methods for samples in their native state in an aqueous environment and to be able to follow microscopic processes dynamically.

In the first experiments, we used the elemental specificity of x-ray imaging to distinguish between protein-rich and water-rich areas of the zymogen granule at the spatial resolution of the microscope. After these initial experiments, which generated images with spatial resolution better than 1000 Å, we have been developing a unique microscopic sample chamber in preparation for the next experiments on the NSLS X-1 beamline (which will be a substantially brighter x-ray source than X-17T). This new wet cell, shown in Figure 4, has 200-by-200-μm silicon nitride windows spaced 2 μm apart. The 1-μm-diameter zymogen granules are held by very small shelves or chevrons deposited on the windows. This arrangement satisfies the need for a vertical chamber to match the horizontal x-ray beam. Input and output ports are etched into one of the windows to accommodate small needles that carry fluid into and out of the sealed chamber; thus the contents of the chamber can be changed during viewing. We have been testing chamber designs, developing techniques for chamber assembly, and establishing the flow characteristics of the chamber.

X-Ray Holographic Microscopy

In earlier years we used undulator radiation from the X-17T beamline at the NSLS to record high-resolution holograms made with 25-Å x-rays in PMMA (polymethyl methacrylate) resist. By virtue of the superior properties of this combination of source and detector, we were able to record hologram fringe detail down to at least a 500-Å period with zymogen granules as the specimens. These were the highest-



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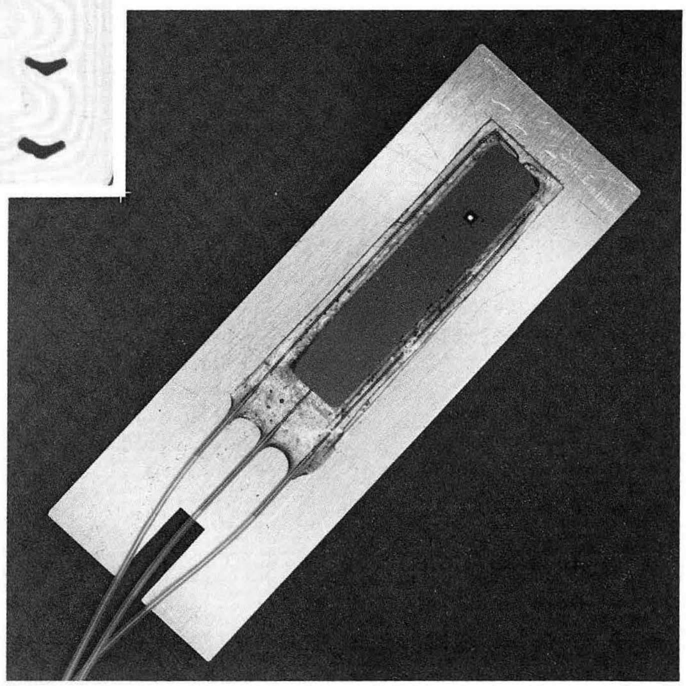

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Figure 4. In this newly developed sample chamber, zymogen granules can be held in place on very small shelves or chevrons (inset) deposited on the window near the right-hand end. This arrangement effectively provides a vertical chamber, which is needed because the x-ray beam is horizontal. Input and output ports are also etched into one of the windows to accommodate small needles, permitting changes of fluid content, pH, and other aspects of the biochemical environment during testing. The entire device shown in this photograph is slightly more than 40 mm long.

resolution holograms ever recorded; they present the challenge of reconstructing the final image with equally high resolution. In preparation for the availability of the new, brighter X-1 beamline at the NSLS, our activities during the last year have focused mainly on achieving an optimum procedure for reconstructing the already-recorded holograms and analyzing the meaning of the various structures that appear in the reconstructions.

Reconstruction begins with the making of a transmission electron micrograph of the recorded hologram, which is prepared by developing the exposed PMMA resist and shadowing it at a glancing angle with metal for enhanced contrast. The resulting photographic negative is digitized by means of a microdensitometer. The reconstruction process is carried out by computer simulation of the classical procedure of illumination of the digitized hologram with the original reference wave. A final image obtained in this way is shown in Figure 5 alongside the original hologram record. The main issues that have to be addressed in this method of reconstruction are focusing, twin-imaging, and linearity.

Focusing is carried out essentially by trial, using a sharpness criterion to choose the best-focused image. The ability to do this through computer calculation (and thus without any radiation exposure to the sample) is one of the advantages of this technique. The twin-image problem has been addressed by some simple approaches, but the amount of twin-image corruption of the image is still unsatisfactory. We are presently studying the best way to address this shortcoming; several demonstrated methods have appeared in the literature, and we are working toward applying them to our holograms.

Our studies of the meaning of the various features that appear inside the reconstructed image of the granules have concentrated mainly on comparative microscopy in which we try to observe the same types of structure by other methods, such as scanning electron microscopy and high-energy (1.5-MeV) electron microscopy. Thus far we have seen some correlation between our image features and those observed through other techniques. Nevertheless, we must conclude for the time being that the features are quite similar to the noise present in the open spaces around the granules, so caution is required in making any biological interpretation. We believe that with improved ways to suppress the twin-image noise and other noise, we will be able to make a more definitive judgment.

We have also been making a more careful estimation of the resolution of the entire holographic imaging process than that obtainable from the fringe spacings of the holograms. Both the intrinsic resolution of the system and the smallest resolved feature in the image of any particular sample are significant. One way to measure the resolution is to image something which approximates an idealized

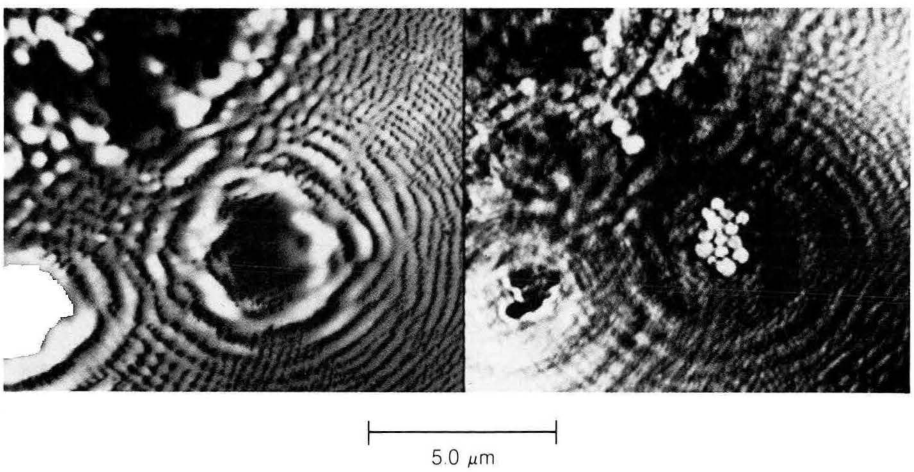

Figure 5. An x-ray hologram record (left) and the image of a cluster of dried zymogen granules numerically reconstructed from it (right). The hologram was obtained in Gabor geometry with 25-Å x-rays. The left photograph is a transmission electron micrograph of the resist, taken after development and shadowing of the fringe pattern. The information in the record was digitized with microdensitometer techniques; the resulting numerical data was used to reconstruct a hologram through computer emulation of the x-ray illumination process that would ordinarily have been used.

object, such as a step. We have done this using a bar on an electron microscope grid; the result is a step image whose width (25–75% of height) is about 660 Å. Since the grid bar is somewhat wedge-shaped and is not a perfect step, it is necessary to estimate the contribution of the bar shape to the observed image width. At present we believe that the system resolution of the overall process is 600–700 Å or better.

Reflective Optics for Hard X-Rays

Multilayer coatings, which consist of multiple ultrathin layers of alternating high- and low-atomic number (Z) materials, are effective reflectors of x-rays over a broad wavelength range. In effect, multilayer coatings are artificially structured crystals, so the wavelengths and angles of incidence for which they are highly reflective are determined by the Bragg equation with the d spacing equal to the period of the multilayer; that is, the sum of the thicknesses of one high-Z and one low-Z layer. Our effort encompasses fabrication of multilayers, fundamental materials-science studies of these structures, and design and implementation of these optics in a wide variety of x-ray applications.

Multilayers fabricated in our laboratory have been incorporated into a wide variety of x-ray optical systems at other laboratories across the country. X-ray holography experiments with soft-x-ray lasers at Lawrence Livermore National Laboratory used mirrors coated with molybdenum-silicon multilayers fabricated here. Imaging of laser-produced plasmas with multilayer-coated mirrors was carried out. We continued to exploit multilayer-coated focusing optics in a hard-x-ray microprobe for spatially resolved elemental analysis at NSLS and SSRL. In addition, we have begun a collaboration with the University of Wisconsin on the development of a soft-x-ray microscope based on a multilayer-coated Schwarzchild objective (see Figure 6) for photoelectron spectromicroscopy of condensed-matter subjects.

Microstructure of Multilayer Coatings

In addition to fabricating high-quality tungsten-carbon and molybdenum-silicon multilayers, we continually investigate untested materials combinations that may lead to multilayer structures with improved reflectivity of short-wavelength x-rays at near-normal incidence. Such investigations have shown, for instance, that ultrathin amorphous silicon-carbide films can be incorporated into single-layer and multilayer structures of high optical quality.

Because the achievable reflectivity and other optical properties of multilayer coatings are determined by the details of their microstructure, we are pursuing an investigation of the relationship between structures and x-ray optical properties. Our arsenal of characterization methods has expanded to include high-resolution

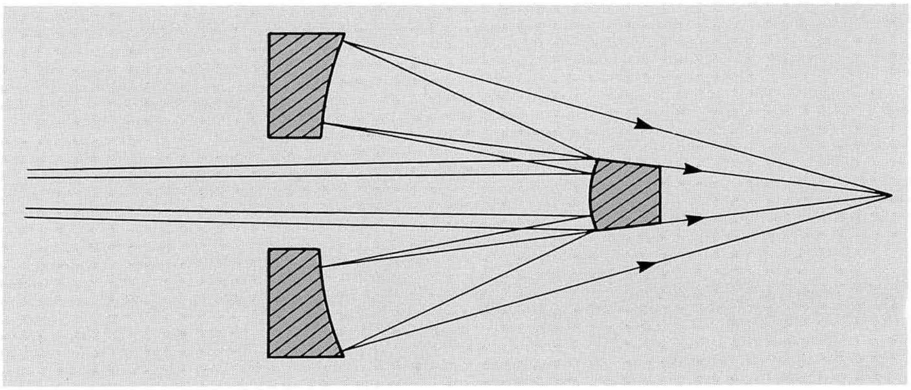

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Figure 6. In collaboration with colleagues at the University of Wisconsin Synchrotron Radiation Center, LBL scientists are developing normal-incidence optics with multilayer coatings to focus synchrotron radiation into a submicron spot, as diagrammed in this Schwarzchild-objective microscope. Related demagnification techniques are being considered for use in microlithography.

transmission electron microscopy (in collaboration with the Laboratory's National Center for Electron Microscopy) and new x-ray scattering techniques.

In 1988, we performed fundamental studies of how interatomic structure relates to the stability and x-ray optical performance of multilayers. This understanding is especially important for multilayer applications that require a long lifetime or those in which multilayers will be subject to thermal annealing or harsh radiation environments, as in synchrotron radiation beamlines or in x-ray laser systems. Such understanding may also help us to develop multilayers from untested constituent materials with superior performance. In particular, we continued our investigation of the tungsten-carbon multilayer system, which was one of the first multilayers of demonstrated utility in x-ray optical systems. We have begun systematic studies of the structural changes of tungsten-carbon multilayers with annealing, using transmission electron microscopy techniques and x-ray diffraction.

The high-resolution electron micrographs in Figure 7 show a tungsten-carbon multilayer whose period is roughly 70 Å, first in the as-prepared state and then after being annealed at 500°C for four hours in high vacuum. The micrographs show that the initially well-defined layers changed significantly upon annealing under these conditions, but that they maintained a layered structure. Changes occurred primarily in the tungsten-rich layers, and, in this specimen, the tungsten-rich layer crystallized extensively into the compound tungsten carbide (WC).

Figure 8 is a micrograph of an annealed specimen with a period of roughly 120 Å. The tungsten-rich layer also crystallized into WC in this annealed specimen, while the carbon-rich layers remained amorphous after annealing. It is remarkable that single WC crystallites extend over such large distances within the layers while maintaining high-quality interfaces with the carbon-rich layers. Studies to date have revealed systematic trends in the nature of atomic rearrangements that occur on annealing and confirm that the multilayer period expands during annealing. These transmission electron microscopy studies complement previous x-ray-scattering experiments on similar multilayer systems. These types of studies will be extended to other multilayer systems of interest in x-ray optical applications; multilayers being studied include amorphous silicon carbide and, for longer-wavelength applications, molybdenum-silicon.

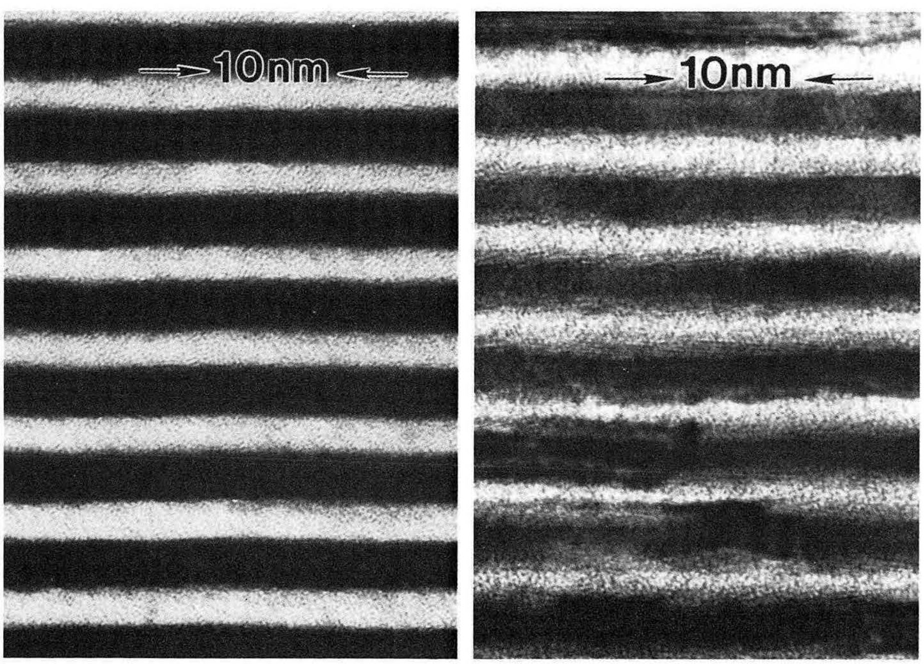

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Figure 7. Before-and-after images show that, although annealing significantly altered this tungsten-carbon multilayer, the basic layered structure remained intact. Changes occurred primarily in the dark tungsten-rich layers, which crystallized extensively into tungsten carbide while maintaining high-quality interfaces with the carbon-rich layers. The period *d* of this multilayer (the combined thicknesses of one high-Z and one low-Z layer) is about 70 Å.

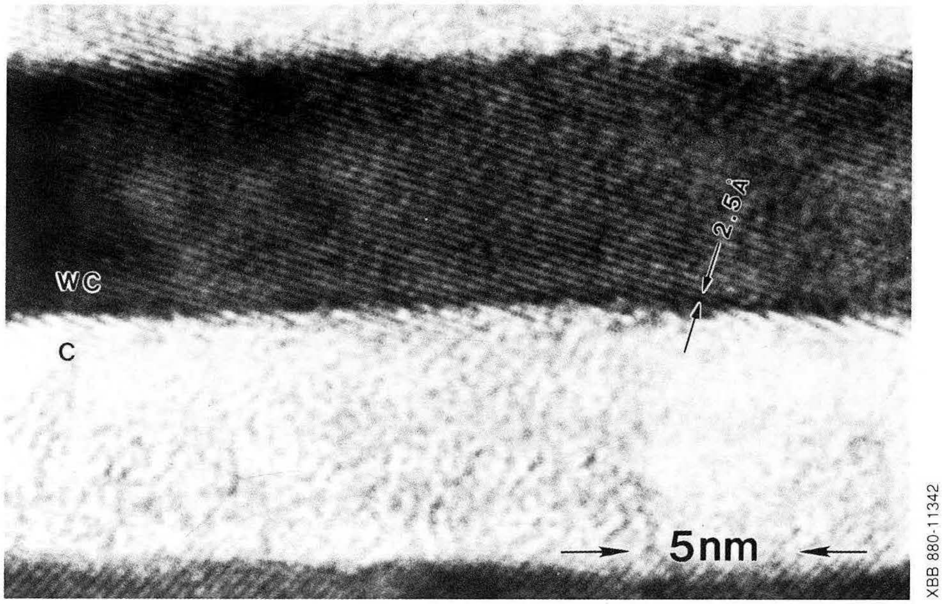


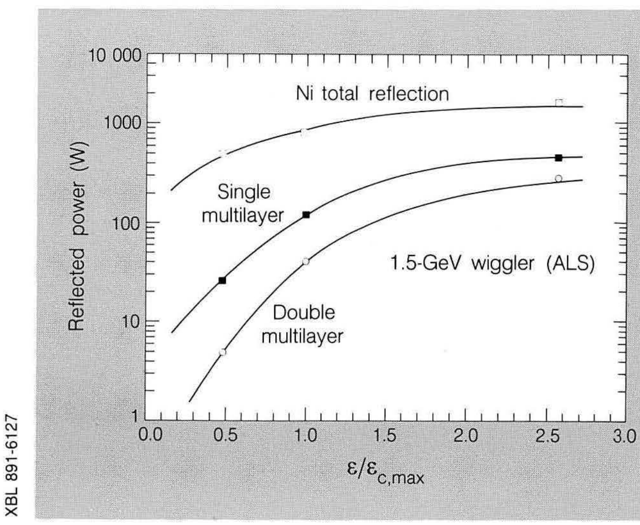
Figure 8. Detail of an annealed WC multilayer whose period is approximately 120 Å. The dark tungsten carbide crystallites extend quite far into the amorphous carbon-rich layer while maintaining sharply defined interfaces with it.

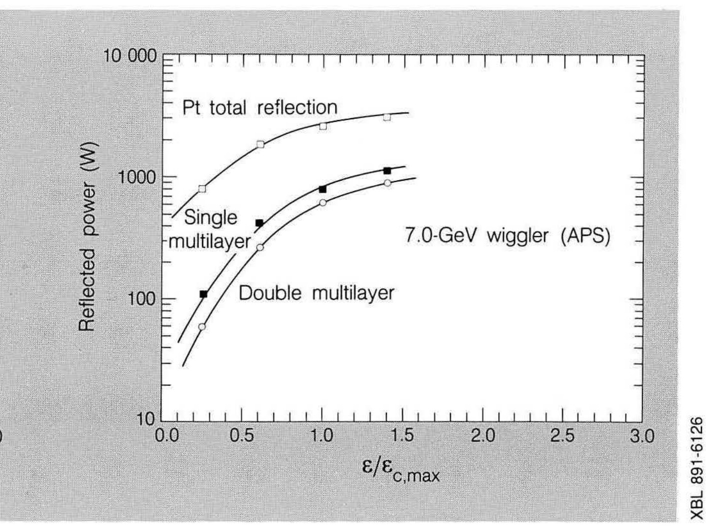
Multilayer Power Filter

Because of the wavelength selectivity of multilayer-coated mirrors, they can be used in synchrotron radiation beamlines to reduce the power at unwanted photon energies that is incident upon downstream optical elements. With this application in mind, calculations were performed to model this power-filtering performance in beamlines proposed for construction on the upcoming third-generation, high-brightness synchrotron radiation sources. In these facilities, x-ray optical elements will encounter unprecedented power loads. Cases considered include wigglers on 1.5- and 7.0-GeV synchrotrons at a set of photon energies spanning the maximum critical energy of each wiggler.

These calculations (see Figure 9) show that a multilayer-coated mirror, if used instead of a total-reflection mirror as the first mirror in a beamline, can reduce the unwanted power reflected down the beamline by roughly an order of magnitude. This advantage is most pronounced at photon energies below the critical energy of the wiggler.

Figure 9. Calculations indicate that a pair of multilayer-coated mirrors always reflects less unwanted energy into the beamline than does a total-reflection mirror. However, its advantage—initially an order of magnitude—drops off as photon energy approaches the critical energy of the wiggler radiation source.





The cost of the reduction in reflected power impinging on downstream optics is that the power absorbed in the multilayer-coated mirrors is greater than in total-reflection mirrors. This power absorption can cause adverse thermal-loading effects which must be dealt with. Furthermore, some of the absorption occurs at the desired wavelengths. And, because of the limited passband of multilayers, broad tunability is a more-complicated matter. Nonetheless, these results suggest that multilayer-coated mirrors should be seriously considered as first optics in the upcoming generation of synchrotron radiation sources, even though they are not preferable to total-reflection mirrors in all applications.

Spatially Resolved Elemental Analysis

We continued to demonstrate the capabilities of the hard-x-ray (8–12 keV) microprobe that we had constructed earlier and have been testing. The microprobe uses as focusing elements two concave, spherical mirrors, coated with WC multilayers, in a Kirkpatrick-Baez geometry; this configuration can direct a synchrotron radiation beam into a spot approximately 6–7 μm on a side. (While zone plates provide better resolution, they are nearly transparent at these photon energies. Multilayer-coated reflective optics appear to provide the highest resolution for hard x-rays.)

Figure 10 shows a multilayer-coated mirror in its holding block in the microprobe. Because of the wavelength selectivity of the mirror , the instrument passes a 1-keV-wide slice of x-radiation near 10 keV. Quantitative nondestructive detection of elements is accomplished by measuring the characteristic fluorescent x-rays emitted by the irradiated sample. The microprobe has an elemental sensitivity of femtograms for elements from potassium to zinc in the periodic table, and the samples require no special preparation and no special environment. Additional goals of our ongoing effort include improving the spatial resolution still further (to about 1 μm) and increasing the elemental sensitivity.

In experiments at SSRL, we examined a variety of samples with the microprobe. One group of specimens consisted of pieces of parchment or paper taken from antique documents and from artwork. The goal was to determine

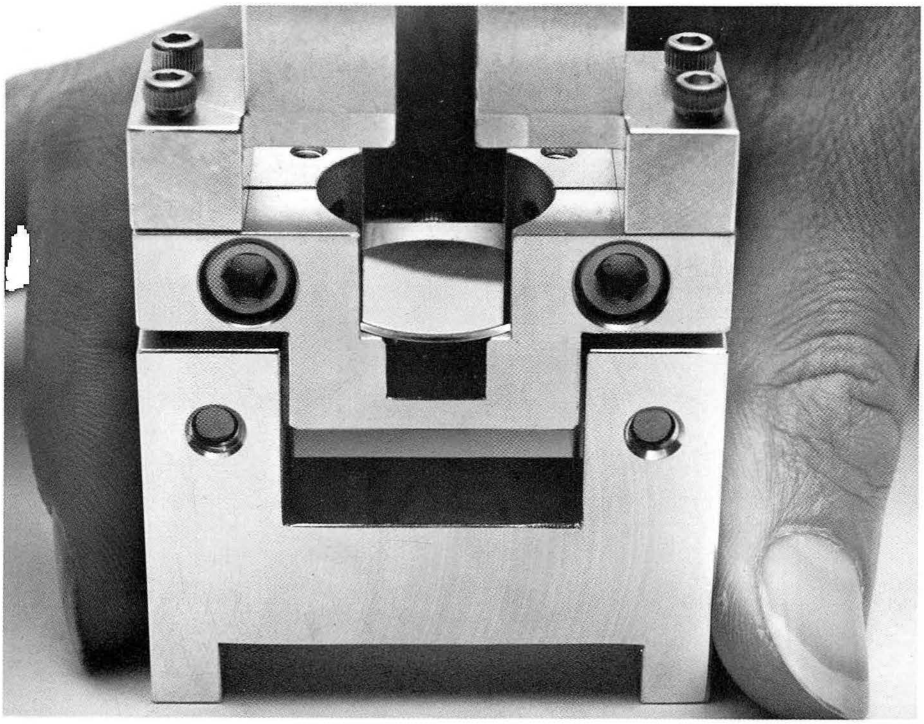


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Figure 10. A multilayer-coated mirror in its holding block in the hard-x-ray (8- to 12-keV) microprobe.

whether the microprobe is a useful tool for answering questions about the manufacture of the paper and the composition of the inks, and therefore whether it could provide a method for document authentication. One result of this investigation was a clue to the origin of "foxing," which is the appearance of brown or gray spots on the surface of the paper in old books and documents. The fox marks, which may be several millimeters in diameter or larger, disfigure the documents. We found that in many cases a particle of iron or iron oxide is embedded in the center of the mark.

Figure 11 is a linear scan across a fox mark that shows the very localized (less than 50 μm in diameter) and quite concentrated (130 $\mu g/cm^2$) elemental iron distribution. The iron probably came from a small iron particle that was imbedded in the paper when it was manufactured. Diffusion of the iron as it oxidized in the presence of atmospheric moisture over many years evidently caused the discoloration, a hypothesis which suggests that little can be done to prevent this type of foxing. We also determined the composition of a variety of ancient inks.

A second group of specimens, for which preliminary measurements were made, consisted of desmids, which are large, single-cell water plants. Desmids can extract and concentrate minerals, such as strontium and barium, from the water they live in. Biologists are interested in the mechanism by which desmids can accomplish this even when the water contains much larger concentrations of chemically similar calcium.

Laser-Plasma Imaging

Unlike uncoated x-ray optics, multilayer-coated mirrors can exhibit high reflectivities at near-normal angles of incidence for sufficiently long wavelengths. In an experiment carried out in collaboration with the Naval Research Laboratory and the Laboratory for Laser Energetics at the University of Rochester, we obtained x-ray images of imploding laser-fusion targets with the use of a multilayer-coated spherical mirror. The mirror was coated to reflect the 2*p*–3*d* spectral lines of silicon +11 ions (silicon with 11 electrons removed, also referred to as Si XII) at 44.1 Å. These lines are produced in the hot coronal plasma regions surrounding the glass Microballoon and its supporting stalk. The Microballoon had a diameter of 274 μm and a wall thickness of 2.8 μm, and was filled with deuterium gas at a pressure of 50 atmospheres. It was irradiated by the 24-beam ultraviolet (351-nm) laser at Rochester, which deposited approximately 1060 J in 640 ps.

Figure 12 shows a time exposure of a typical target shot and a contour plot of the photograph's density. The most intense emission originates in a spherical

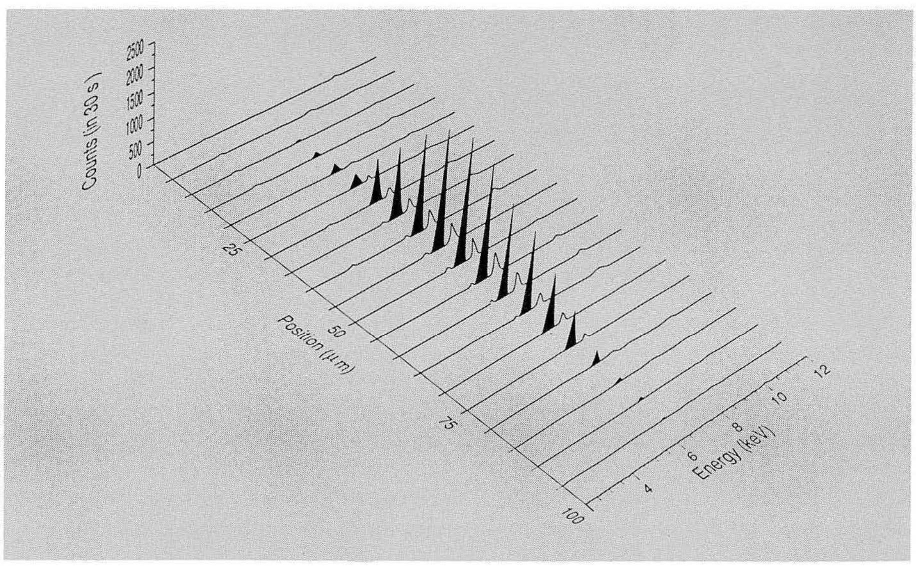
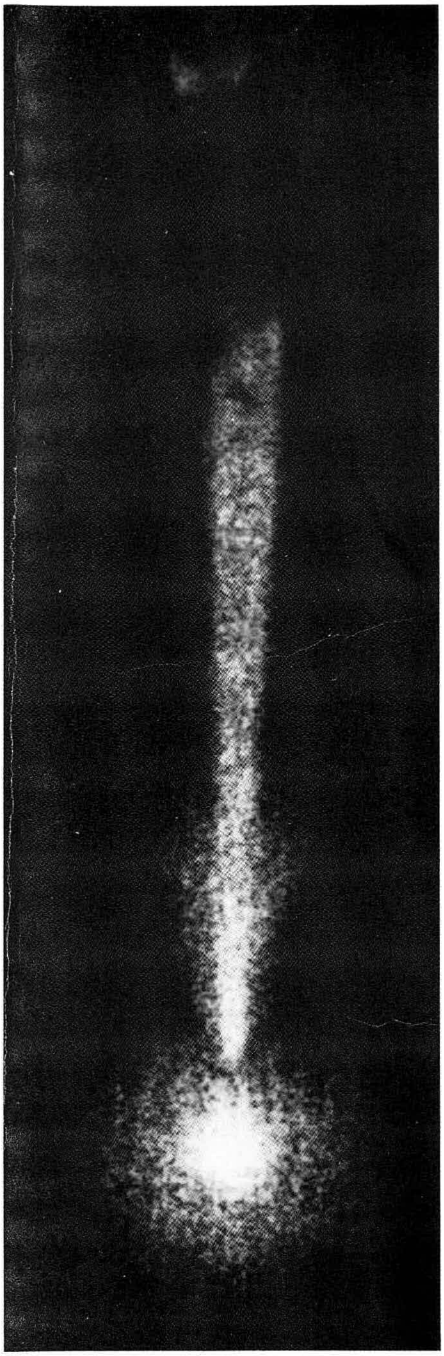


Figure 11. A linear x-ray-microprobe scan across a "fox" spot in a book 300–400 years old shows an elemental-iron distribution that is very localized (less than 50 μm in diameter) and quite concentrated (130 $\mu g/cm^2$).



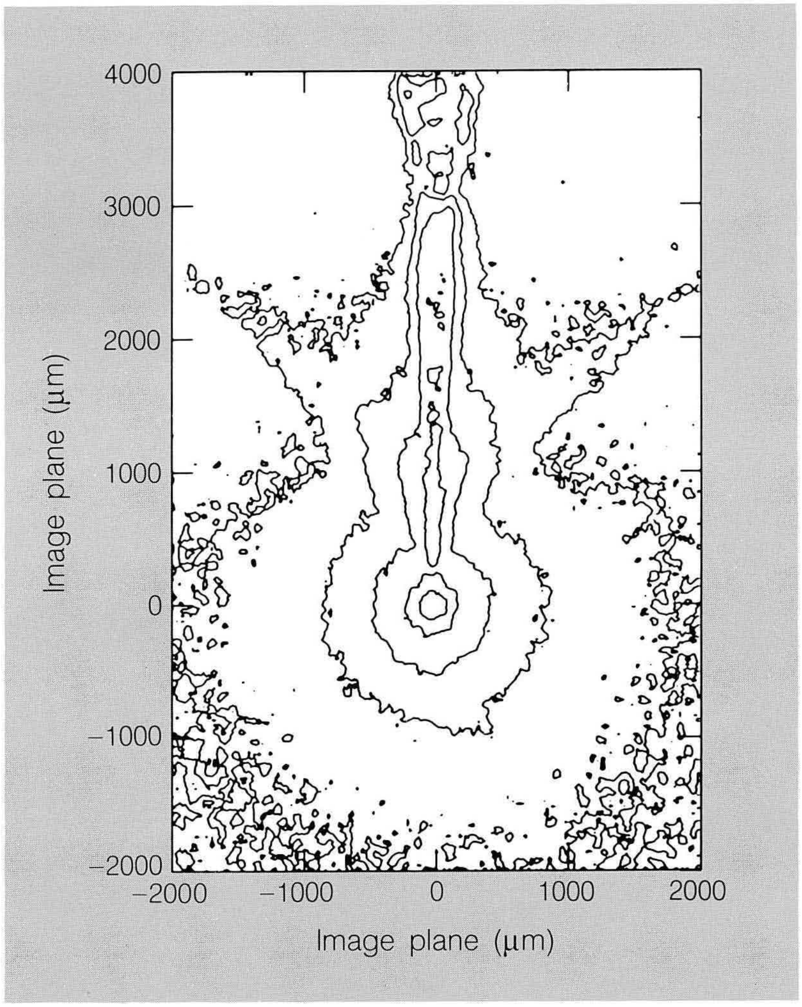


Figure 12. A photograph of a target shot (left) and a contour plot of the photograph's density show how a multilayer-coated spherical mirror can provide an x-ray observation tool for inertial-confinement fusion experiments. (The densitometer plot shows details in areas that would appear black in this reproduction and were therefore cropped out.) The scale length of the changes in the coronal emission recorded on the film is about 40 μm. Since the exposure is time-integrated, the upper limit to the resolution of the mirror is about three seconds of arc.

region of approximately 300-μm diameter. In addition, features associated with the target support system are clearly visible. The target was supported by a quartz "spider-web" fiber 300 μm long and less than 1 μm in diameter, which was in turn attached to a glass capillary tapered to 1 μm at the point of attachment. The scale length of the changes in the coronal emission recorded on the film is about 40 μm. Since the exposure is time-integrated, this gives an upper limit to the resolution of the mirror of about three seconds of arc. Narrow-spectral-band images of this kind constitute a valuable new diagnostic tool for the study of laser-produced plasmas and laser fusion.

Coherent XUV Sources

Free-electron lasers (FELs) are a promising source of intense, tunable coherent radiation with potential applications from basic research to national defense. As Figure 13 shows, there are two spectral regions in which FEL sources are especially interesting: those parts of the infrared that are not covered by conventional microwave techniques, and the short-wavelength end of the ultraviolet, where laser and harmonic-generation techniques are of decreasing importance. In several laboratories around the world, infrared FELs are operating or being built. FELs for the ultraviolet and shorter wavelengths are more challenging because of stricter requirements for electron-beam quality. In 1988 we began the study of several issues related to ultraviolet FELs, including accelerator options, the possibility of combining FELs with nonlinear optical techniques, and the understanding of temporal coherence.

Free-Electron-Laser Theory

In the past, we have investigated the properties of a high-gain ultraviolet (400-Å) FEL in a special long straight section, called a bypass, in an electron storage ring. In principle, such a bypass, containing a very long undulator, could be built in either an electron storage ring dedicated to FEL studies or in the storage ring of an advanced synchrotron radiation source, such as the Advanced Light Source (ALS). The high gain would allow the FEL to operate by the mechanism of self-amplified spontaneous emission (with no optical cavity), thereby obviating the need for high-reflectivity, long-lifetime mirrors, which are not yet available. Mirrors of moderate reflectivity offer some interesting advantages.

This past year, in collaboration with scientists from the University of California-Berkeley and from the Exploratory Studies Group here in LBL's Accelerator and Fusion Research Division, we began to study a new method for generating intense,

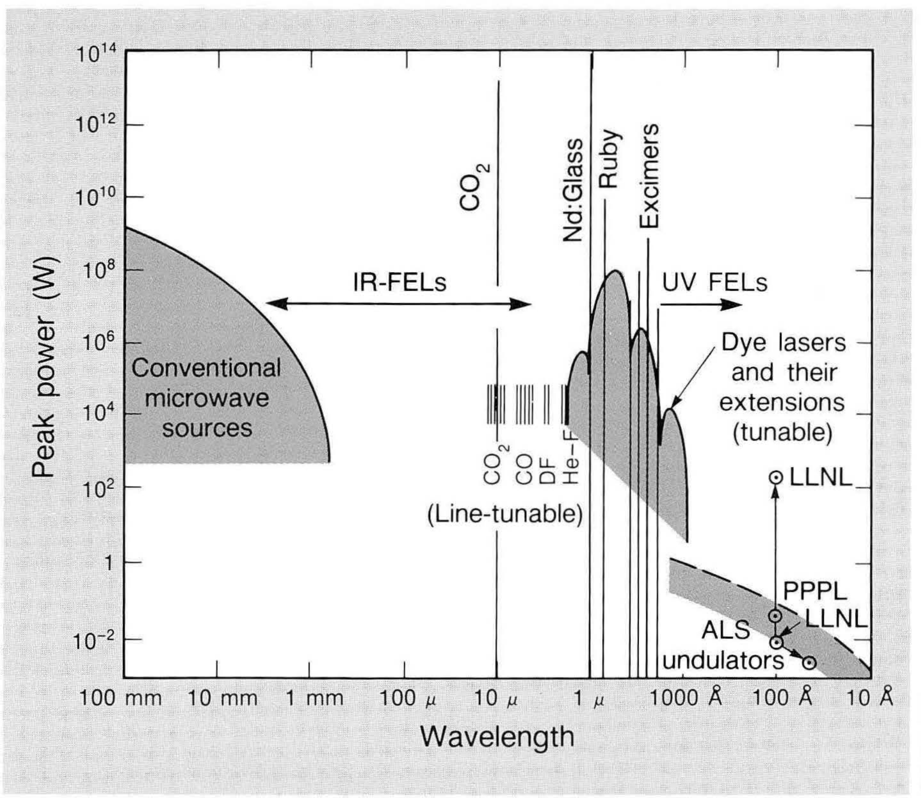


Figure 13. Two especially attractive spectral ranges for free-electron laser (FEL) applications are the infrared and the short-wavelength end of the ultraviolet where lasers and harmonic-generation techniques are of diminished importance.

coherent radiation at ultraviolet wavelengths from 1000 to 300 Å. The new method combines a high-gain FEL amplifier and optical harmonic-generation techniques. For example, conventional nonlinear optical techniques can be used to generate several kilowatts of coherent radiation at a wavelength of 1000 Å, which becomes the input to the FEL amplifier in the bypass. The amplifier increases the coherent power to the multimegawatt range. Finally, the wavelength is decreased to 400 Å by extending to shorter wavelengths the existing technique of four-wave mixing in a pulsed inert gas beam from a nozzle. In contrast to the earlier studies, the FEL is used as an amplifier rather than a generator of coherent radiation, and it operates at 1000 Å rather than 400 Å so the requirements on the quality of the stored electron beam are less stringent.

Electron storage rings offer both advantages and disadvantages for FELs. The natural damping mechanism of synchrotron radiation makes possible the high-brightness electron beams required for FEL operation at short wavelengths. But the long damping time—tens of milliseconds—limits the average coherent power of the radiation generated. For example, the previously investigated high-gain FEL in an ALS bypass could generate tens of megawatts of peak power, but it would require special operating conditions.

FELs based on linear accelerators are not limited by the damping requirements, but they require electron guns that can generate low-emittance (small-phase-space), bunched electron beams. The linac approach has become more promising with the recent development at other laboratories of a new type of electron gun that uses laser-induced electron emission in an rf cavity. We have studied the performance of this type of gun in collaboration with the Exploratory Studies Group and have derived simple analytical formulas for the gun emittance that agree well with the results of computer simulations.

Spectroscopy with X-Rays

Developing and demonstrating instrumentation for x-ray spectroscopy provide an objective test of the utility of new x-ray optical technology. A prime example is the high-resolution, erect-field spectrometer that we described last year. Because of a novel grating whose line spacing varies continuously, the spectrometer has both an ultrahigh spectral resolving power (35 000) and a simple wavelength-scanning mechanism, features that are normally mutually exclusive. The spectrometer's inventors received an R&D 100 award from Research and Development Magazine *for their achievement. In 1988 we completed a lengthy revision of a comprehensive and widely used set of tables of x-ray optical constants that are needed to design experiments and to calculate the performance of optical components. We carried out a systematic and detailed investigation of the properties of candidate materials for high-sensitivity photocathodes. And we demonstrated a low-angle x-ray scattering technique for determining the size of submicrometer particles.*

Interaction of X-Rays with Matter

To plan scientific experiments or predict the performance of optical components, one must know the optical constants that describe how radiation interacts with matter. For example, designing optical components and applying them to x-ray spectrometry of the new, intense synchrotron and high-temperature plasma radiation sources require analytical model descriptions that are based on these constants. (Typical optical components in a synchrotron radiation beamline include a first mirror, focusing mirrors, and a monochromator, which may include either a grating or a crystal dispersion element.)

For the XUV region of the electromagnetic spectrum, the optical constants for a material may be expressed in terms of the real and imaginary parts of the atomic scattering factor. We previously published tables of the atomic scattering factors of elements from Z = 1 to Z = 92; they covered the photon energy range of 30 eV to 10 keV for the imaginary part of the atomic scattering factor, f_2, and 100 eV to 2 keV for the real part, f_1. These tables are generally regarded as the best available in the low-energy x-ray region.

We have just completed revised tables that extend down to 10 eV for f_2 and up to 10 keV for f_1. For each element, at each photon energy, f_2 is directly determined from a compilation of measured mass absorption coefficients and recent calculations of the atomic photoabsorption cross sections. The real part f_1 is then calculated from the values of f_2 over the entire photon energy range by using Kramers-Kronig relations. The real and imaginary parts of the atomic scattering factor determined in this way for carbon are shown in Figure 14 as an example of the results. The new tables will be published and also made available in diskette form.

High-Efficiency Photocathodes

As part of a continuing effort to develop highsensitivity photocathode materials for time-resolved x-ray measurements, we recently reported extremely high yields of secondary electrons in condensed rare gases. This past year we completed a detailed and systematic investigation of x-ray-induced secondary electron emission from solid xenon. The optimum growth conditions were determined, and the films were shown to be stable under moderate vacuum conditions (about 10^{-7} Torr). A dramatic increase in the secondary electron yield was observed when an electric field was applied, as it would be in a real x-ray detector. Figure 15 shows the measured secondary electron yield for solid xenon at photon energies from 100 eV to 10 keV both with and without

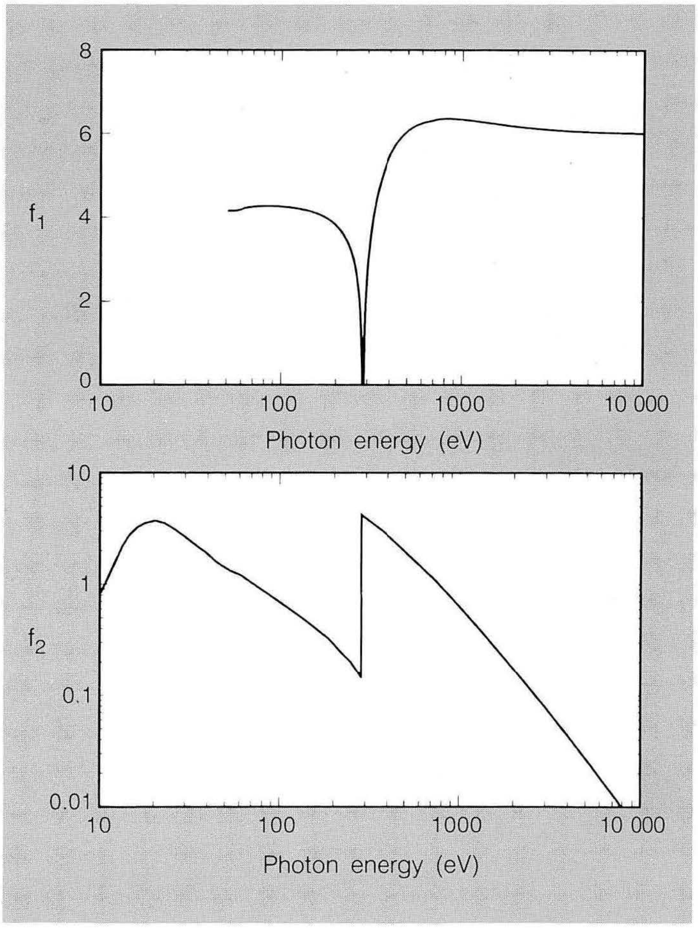


Figure 14 . The real and complex parts of the atomic-scattering factor as a function of energy for carbon, an example of the data generated and published by the Center for X-Ray Optics. The 1988 and early-1989 achievements included extension of the atomic-scattering-factor tables to both higher and lower photon energies.

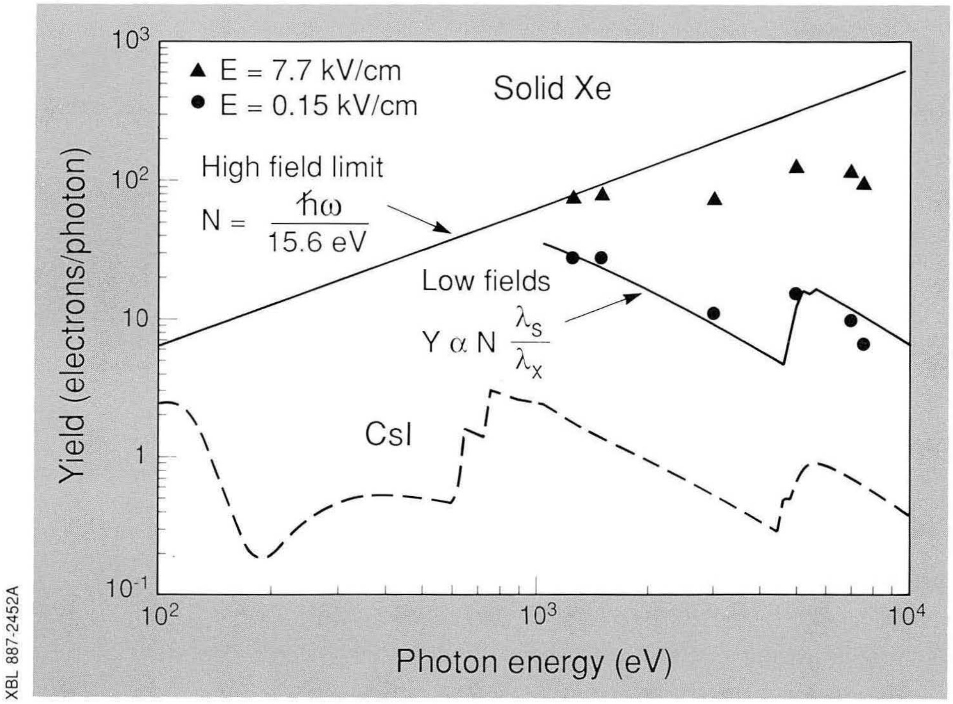


Figure 15. Secondary electron emission as a function of energy from solid xenon approaches the theoretical maximum (straight, solid line) when an electric field is applied. The line near the bottom shows the electron emission from cesium iodide, a standard high-quantum-yield photocathode material. Note especially the photon energies; today's photocathode materials, such as cesium iodide, give high quantum yields only at relatively low energies, whereas solid xenon can be used with hard x-rays at up to 10 keV.

an applied electric field. With an electric field, the yield approaches the maximum number of available photons per photon. It is believed that the enhancement is due to the electrons gaining energy as they drift in the field, so that the average electron energy becomes comparable to the electron affinity of solid xenon. The secondary emission studies demonstrate that solid xenon may be useful as a very sensitive hard-x-ray photocathode.

Particle Sizing with X-Rays

We have developed a novel technique of sizing submicrometer particles by means of low-angle soft-x-ray diffraction. These structures are intermediate in size between those observable with a light microscope (larger than 1 μm) and those observable with other x-ray techniques (approximately 0.1 μm). Solid polystyrene spheres of various known sizes were used as a scattering sample for carbon K_α (44.8-Å) and vanadium L_α (24.3-Å) radiation. Radiation passing through a single pinhole onto the sample is reflected by a spherical mirror coated with carbon-tungsten multilayers back through the sample and is focused onto an x-ray-sensitive film, where it forms a diffraction pattern. The sharp pattern arising from a hexagonal lattice structure of spheres and the diffuse pattern from a random arrays of spheres were found to compare well with the calculated pattern shapes and sizes. The success of this method suggests a new way to size particles in large sample areas with low-intensity radiation. For example, biological particles may be sized without extensive radiation damage to the particles, which may remain in their natural, aqueous environment.

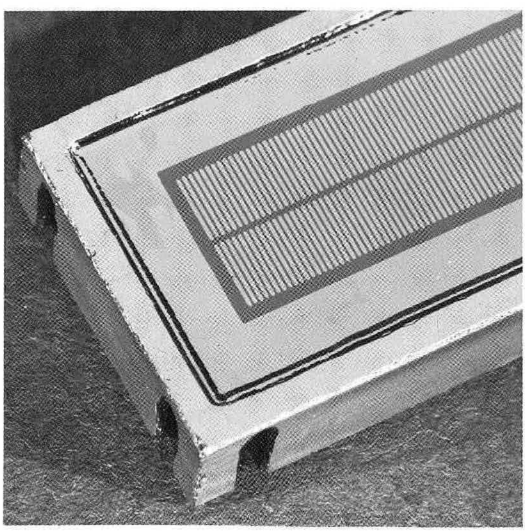
Detectors for Coronary Artery Imaging

For several years, we have joined with scientists from Stanford University, the Stanford Medical School, SSRL, and NSLS to investigate the use of synchrotron radiation to make x-ray images of human coronary arteries; the goal of this work is to develop a less-invasive and therefore safer means than the conventional x-ray technique of coronary angiography. In the alternative being explored, called transvenous coronary angiography with synchrotron radiation, an iodine-based contrast agent is injected into the venous system (rather than directly through a catheter into the arterial system near the heart, as in the conventional method). The transvenous technique is less invasive, but the contrast achieved is also lower, a deficit that is corrected by using a synchrotron x-ray source that provides the ability to select the wavelength of a high-intensity x-ray beam. The feasibility of this approach has been proven in early tests, and we are now approaching the ability to make clinical-quality images of coronary arteries in humans as we upgrade the imaging system.

Position-Sensitive X-Ray Detector

An important part of the newest system is a position-sensitive x-ray detector that simultaneously records the intensities of x-rays transmitted through the patient at two wavelengths, one just above and one just below the K edge of iodine. The difference between the two intensities is due to absorption by the iodine and is the information from which the image is built up by a computer. In the past year, we fabricated a 600-channel detector consisting of two rows of 300 elements integrated on a single silicon chip, one row for each wavelength (see Figure 16).

Figure 16. Higher resolution for transvenous coronary angiography is made possible by this LBL-developed multielement, position-sensitive x-ray detector. We also built a readout system that can read all 600 channels at intervals of 2 ms. With that speed and a 40 000:1 dynamic range, the readout system can provide high-resolution, frozen images, such as the accompanying image of the coronary arteries and other blood vessels in the heart of a live, anesthetized dog. (The labeled structures are the aorta, right coronary artery, left anterior descending artery, pulmonary veins, posterior descending artery, and part of the left ventricle).



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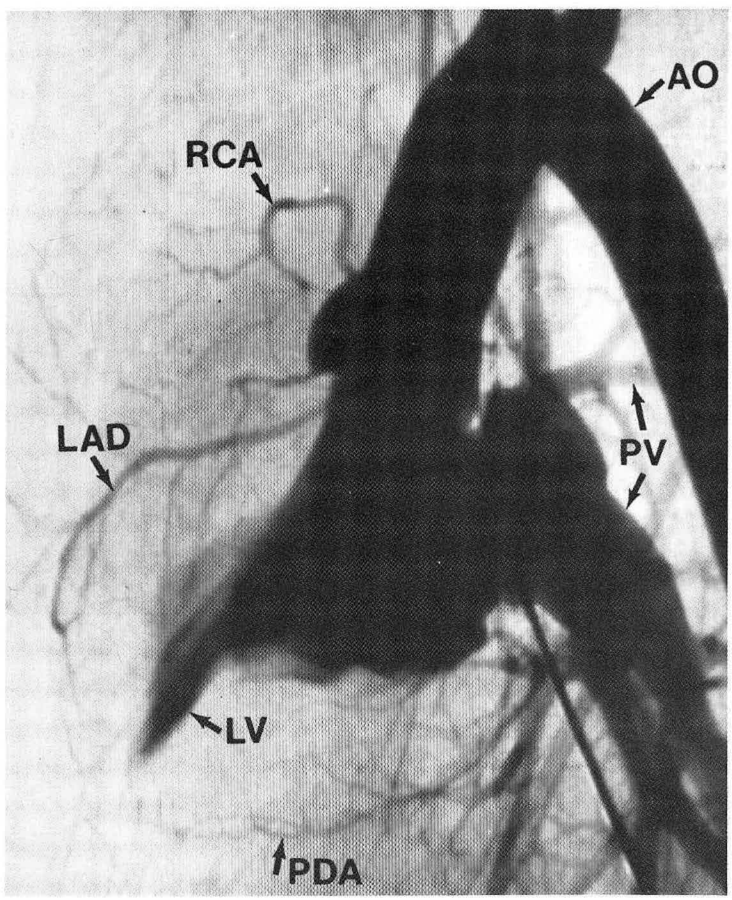

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Moving the patient vertically through the fan-shaped x-ray beam allows the information for the image to be recorded one line at a time. The 300 elements along each row of the detector have a center-to-center spacing of 0.5 mm, which determines the spatial resolution of the image. An electronic readout system for the detector was also constructed that provides separate readout electronics for each channel and the ability to read all 600 channels at intervals of 2 ms; the system is fast enough to freeze heart motion while one line of the image is being recorded, and has a dynamic range of 40 000:1. This detector will be part of a new imaging system to be used with human subjects in experiments at SSRL early in 1989.

Synchrotron-Radiation Optics

When electron synchrotrons and storage rings began to be used as light sources in the early 1960s, there was very little problem in obtaining the needed optical components. They were simply the same as the optics one would use with any other light source. This began to change in the 1970s as electron storage rings dedicated to the production of synchrotron radiation were built. Such machines had more powerful beams and higher brightness; that is, the spatial and angular extent of the source were both small. During the 1980s, the seriousness of all the challenges posed by dedicated synchrotron sources has been considerably multiplied by the use of wigglers and undulators in the straight sections of storage rings to generate still-more-powerful XUV beams. We have been active in developing techniques to meet the challenge of producing the optics needed for the coming generation of synchrotron radiation sources.

Water-Cooled Optics

From the point of view of the optical designer, the dedicated synchrotron radiation source poses a double challenge as compared to previous XUV sources. Greater optical quality is required to image the source, and the imaging has to be achieved in the presence of high photon-beam power and the concomitant thermal distortions. Moreover, the high fluxes achievable with the new sources make it worthwhile for experimenters to reduce the slit sizes of their monochromators in the hope of obtaining much higher spectral resolution. Such demands for higher-resolution instruments translate directly into a demand for higher-quality optics.

To meet the challenges posed by advanced synchrotron radiation facilities across the United States, we are supporting the development of optics for use with undulators (sources of high-brightness and partially coherent XUV radiation) and wigglers (high-power sources). In this program, we have designed and fabricated real-world systems for operation at the most demanding of the present generation of light sources:

- A mirror to receive the direct beam from the 54-pole wiggler at the Stanford Synchrotron Radiation Laboratory
- A similar mirror for the X-1A undulator beam at the NSLS (Figure 17)
- The world's first synchrotron radiation monochromator with directly water-cooled diffraction gratings (Figure 18)
- A water-cooled multilayer reflector to act as a pre-monochromator for the X-23 beamline at the NSLS

The technical strategy in all these cases has been to use water-cooled metal optics coated with electroless nickel. This approach solves many engineering challenges associated with cooling a heated surface, and also allows the needed structural design and fabrication work (multiple brazes and so forth) to be accomplished with reasonable cost and technical security. However, it departs from the well-tried optical fabrication techniques that have been so successful over the years in producing optics with high quality figure and finish in glassy materials. Although it is more difficult to achieve good optical quality in nickel, we have worked with vendors to push the technology to the limit. We have obtained firstclass results (comparable to glass optics) in figure, but not yet in finish. The surface finish one can get in the best glass optics is presently about twice as good as the best nickel surfaces, a gap we are attempting to narrow.

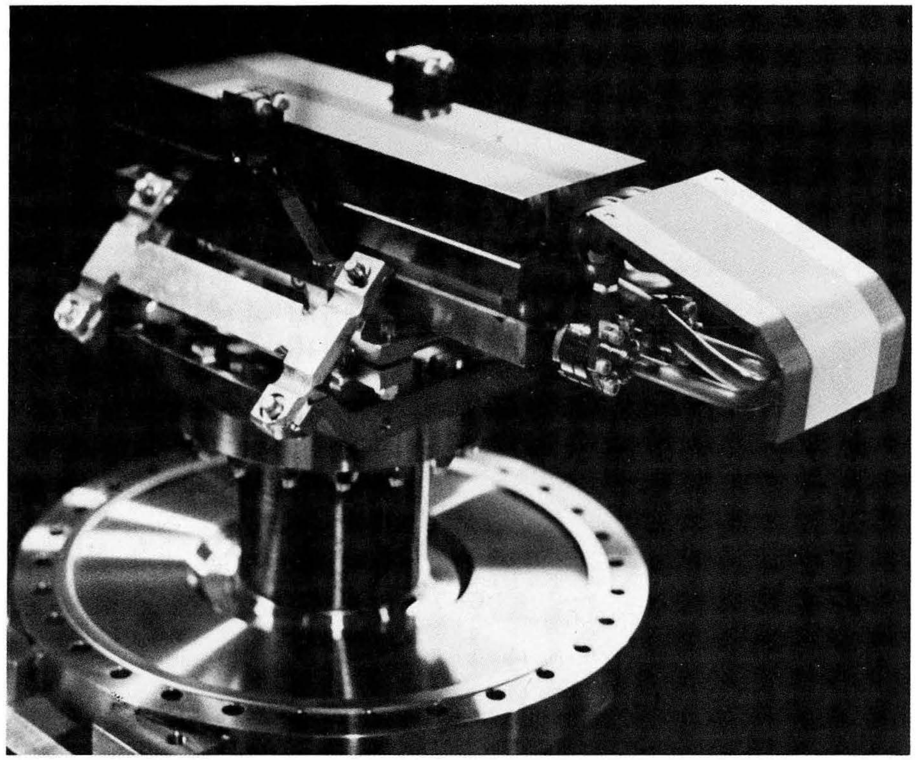

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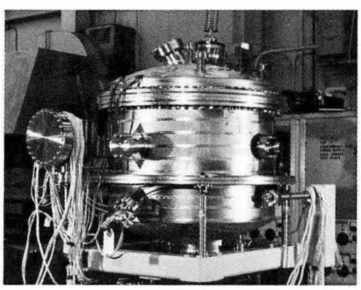
Figure 17. A water-cooled mirror now in use with the X-1A undulator beam at the National Synchrotron Light Source. X-rays come towards the left end of the mirror at an angle of about 3° from the horizontal.



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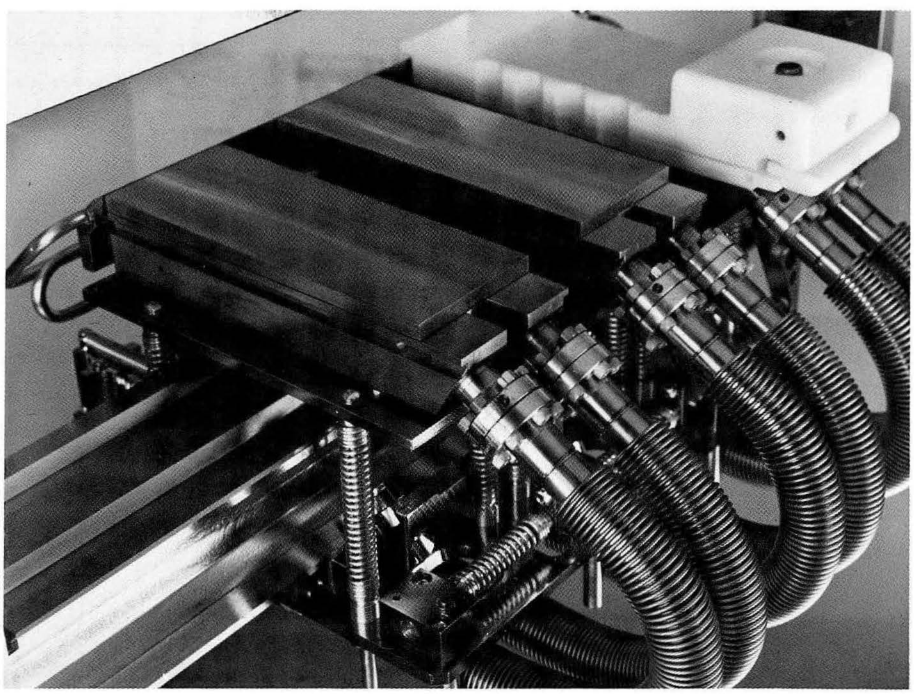

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Figure 18. LBL developed this innovative synchrotron radiation monochromator with directly water-cooled diffraction gratings. The subassembly containing the gratings themselves is shown; one of the three gratings has a protective cover installed. Inset: the complete assembly in its vacuum chamber, ready for installation in a beamline.

Support for the Advanced Light Source

The Center for X-ray Optics also made a strong contribution to the development of the scientific program at the Advanced Light Source by organizing a series of user workshops. (The ALS, a dedicated synchrotron-radiation user facility now under construction at LBL, features a 1-to-2-GeV electron storage ring that can provide high-brightness beams of ultraviolet light and x-rays from its wigglers, undulators, and bending magnets.) These workshops covered diverse areas of science and technology well matched to the spectral range and characteristics of the light from the ALS.

The second half of the year saw the formation of the Advanced Light Source Users' Association (ALSUA). Its first annual meeting in June was directed primarily toward identifying and initiating research teams. When a call for letters of interest went out, nearly three dozen responses were received from strong teams that covered a wide spectrum of research, confirming the user demand for the ALS and auguring well for an active scientific future at the facility.

ALS Workshops

In planning the ALS facility, it is important to understand the capabilities desired within the user community; it is especially critical to equip the limited number of straight sections appropriately. In 1987 and 1988 we held a series of seven topical workshops and symposia intended to develop information about the scientific and technological opportunities at the ALS and to determine the capabilities necessary for users to exploit them. The workshops and related investigations gave much useful information and also reaffirmed that the system's wide range of capabilities will be well used.

The workshops and symposia focused on experiments that take advantage of the high brightness and partial coherence of radiation from undulators in the ALS straight sections. This laserlike radiation is expected to enable experiments in materials and surface science and the life sciences that could not be done otherwise. The undulators will be complemented with wigglers, which also reside in straight sections. These wigglers would make very respectable sources of hard x-rays for crystallography and other applications because of the low emittance of the ALS electron beam. The wigglers will extend the range of photon energies up to about 10 keV. Broadband radiation will also be available from the ALS bending magnets; although this light will be lower in brightness and coherence properties, its photon flux is of interest to many groups; also, it is less complicated to use and less expensive to obtain.

Call for Letters of Interest

After the facility requirements and research opportunities were examined, the next step in forming participating research teams was to obtain concrete expressions of interest from scientists and engineers who have common objectives but who may collectively represent several institutions. Accordingly, the Laboratory issued a Call for Letters of Interest; it was sent to everyone on the ALS mailing list, published in several national and international scientific journals, and distributed at the ALSUA annual meeting.

The ALSUA annual meeting was largely devoted to six working groups (surface and materials science, chemical physics, life sciences, atomic and molecular science, x-ray lithography and quantum nanostructures, and earth sciences). The meeting attempted to bridge the gap between general and specific expressions of interest by identifying likely groups from which letters of interest would come. Note that letters of interest are less formal than proposals to establish research teams; proposals will be solicited in spring 1989.

The annual meeting was highly successful in this regard. By March 1989 there were 11 letters of interest from prospective undulator insertion-device teams and eight from wiggler insertion-device teams—more than enough to fill all the ALS straight sections if all were approved. Although insertion-device teams were the main focus of the meeting, seven letters of interest from bending-magnet teams were also received, as were seven miscellaneous inquiries from individual scientists. Nearly every strong group identified at the meeting has submitted a letter of interest.

On average, each letter of interest contained the names of 11 scientists. Their research interests include these:

- Materials science of interfaces
- Materials science of solids and buried interfaces
- Spatially resolved microscopy (spectromicroscopy) of materials and surfaces
- Spectroscopy of the actinide elements
- Time-resolved spectroscopy of solids and surfaces
- Infrared surface science
- Microscopy and coherent optics for the physical sciences
- Microscopy and spectroscopy of mineral surfaces
- Spatially resolved elemental analysis of rocks and minerals
- Molecular dynamics with laser/synchrotron-radiation techniques
- Gas-phase photoprocesses in atoms and molecules
- Gas-phase molecular spectroscopy
- X-ray microscopy for the life sciences
- X-ray crystallography of biological macromolecules
- Spectroscopy for the life sciences
- Circular dichroism of biological material
- Radiobiology
- Time-resolved studies of biomolecules

The responses came from academic, national-laboratory, and industry scientists from more than 20 states and several foreign countries.

Publications and Presentations

Soft-X-Ray Imaging

D. Attwood, "Partially Coherent Radiation at X-ray Wavelengths," paper presented at the International Laser Science Conference, Atlantic City, NJ, November 1–5, 1987.

D. Attwood, "Partially Coherent X-rays, Optics and Applications," tutorial presented at the annual meeting of the Optical Society of America, Rochester, NY, October 18–23, 1987.

D. Attwood, "X-ray Microscopy and the Advanced Light Source at Berkeley," paper presented at SR-88, Novosibirsk, USSR, August 1988.

D. Attwood, "X-ray Microscopy at the Center for X-ray Optics," paper presented to the Japanese Laser Society, Osaka, Japan, June 1988.

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