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Accelerator and Fusion Research Division
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

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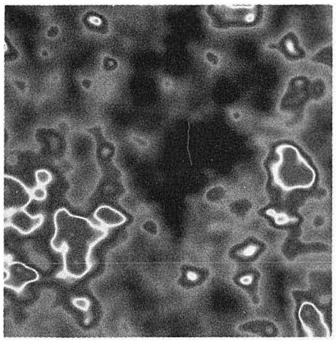

CENTER FOR X-RAY OPTICS

IN 1990 AND 1991, THE CENTER FOR X-RAY OPTICS (CXRO) continued its two complementary roles: demonstrating the capabilities and usefulness of the x-ray and ultraviolet regions of the spectrum and developing equipment and techniques to make those capabilities widely and readily available.

High-resolution x-ray microscopy continues to be prominent among our activities. Soft-x-ray microscopy based on Fresnel zone-plate lenses has provided images of features as small as 300 Å in experiments at the Berlin Electron Synchrotron (BESSY). In the hard-x-ray regime, our microprobe, based on multilayer-coated reflective optics, has achieved 2-μm spatial resolution at the National Synchrotron Light Source (NSLS) and has been used in a large number of applications in the life and physical sciences.

In the long-term effort to develop high-reflectivity multilayer coatings for extreme-ultraviolet and soft-x-ray optical elements, such as mirrors and gratings, we continued investigating the structure and stability of various multilayer pairs and developed a new, highly versatile reflectometer based on a laser-plasma x-ray source and a high-throughput monochromator.

These ongoing efforts in soft-x-ray imaging led to the initial funding of an Advanced Light Source/CXRO program in projection lithography. This joint initiative brings researchers from CXRO and the University of California at Berkeley campus together with representatives of the semiconductor industry. The goal is to further the use of x-rays in the fabrication of computer chips with feature sizes of order 0.1 μm. The photon beams from the ALS are well suited to this research.

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CXRO won two 1990 R&D 100 awards from *Research and Development Magazine* and one 1991 award—the group's fourth and fifth consecutive years of winning in this contest. One award was for a CXRO-developed apparatus that uses soft-x-ray scattering for various applications. Another award recognized a high-fluence X-ray source, developed in a joint project with Sandia National Laboratories at Livermore. The third was for a scanning photoelectron microscope, developed jointly by several collaborators. Also, we tested a new, more portable version of our X-ray microprobe, a previous R&D 100 winner.

Soft-X-Ray Imaging with Zone-Plate Lenses

Extending high-resolution visible-light and ultraviolet imaging techniques into the soft-x-ray region of the spectrum offers several special advantages. The relatively short wavelengths, ranging from several angstroms to perhaps one hundred angstroms, permit users to both "see" and "write" smaller patterns. Furthermore, the associated photon energies, ranging 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 1990 and 1991 we advanced the technology of soft-x-ray imaging and demonstrated some potential applications in the physical and life sciences. Features as small as 300 Å may be seen in our best images.

One of these lenses is at the heart of the High-Resolution Scanning Photoelectron Microscope at Brookhaven National Laboratory's National Synchrotron Light Source (NSLS). The microscope, installed in the X1A beamline at the NSLS, was developed by a collaboration involving the State University of New York at Stony Brook, BNL/NSLS, IBM, and CXRO. It won an R&D 100 award in 1991. It currently offers a peak two-dimensional resolution of 0.4 μm; upgraded beamline optics and improved nickel zone plates may improve resolution to about 50 nm.

Development of Fresnel Zone-Plate Lenses

In microscopy with soft x-rays, the key optical component that ultimately determines performance is the objective lens. Ordinary refractive lenses like those used for visible light, which transform the phase of a wavefront without changing the amplitude, cannot be used at x-ray wavelengths because available materials do not give enough phase shift and are not sufficiently transparent. Reflective optics can be used in the low-energy part of the soft-x-ray region (≈ 100 eV), where efficient high-reflectivity multilayer coatings can be fabricated. To date, however, their resolution in this spectral region has not been as high as that of Fresnel zone plates. Zone plates are thus the lenses of choice for the highest spatial resolution, particularly for energies greater than 200 eV.

Accordingly, one of our major areas of research has been the development of zone plates that have smaller and more-accurately located zones. We are also pursuing the use of materials such as nickel to achieve greater diffraction efficiency. The lenses are fabricated by means of electron-beam writing techniques.

In pressing toward the fundamental diffraction limit of lens performance, accurate placement of the zones (alternate circular bands of transmissive and opaque material) is important. The maximum placement error should be less than a small fraction—25% or so—of the smallest zone width on the lens. This is a formidable challenge, since our highest-resolution lenses, like the one shown in Figure 4-1, have zone widths of 300 Å. Achievement of the required accuracy at these dimensions, especially across large (50-µm-diameter) lenses, is at the frontier of microfabrication. One of our current efforts is aimed at characterizing and reducing placement errors.

In collaboration with researchers from the University of Göttingen, we have been using and characterizing the 300-Å zone plates in the Göttingen x-ray microscope at the Berlin Electron Synchrotron Facility (BESSY). Although measurements of the microscope's optical performance indicate that the diffraction limit has not been reached, images of test patterns show that features as small as 300 Å are visible. Because of our efforts to reduce errors in the placement of the zones, our newest set of 300-Å nickel zone plates should be even better in both spatial resolution and diffractive efficiency.

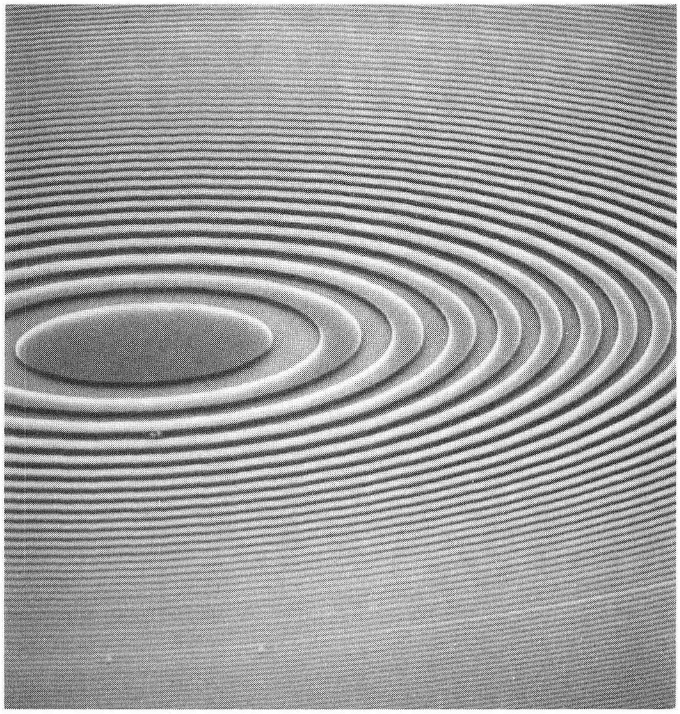

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Figure 4-1. A high-resolution Fresnel zone-plate lens is shown during processing. The tri-level resist has been etched and is ready for electroplating with nickel. Our new nickel-coated zone plates are designed for greater diffractive efficiency than their gold-plated predecessors, and more-precise zone placement will give better spatial resolution. The outermost zone (not visible in this photo) is 300 Å wide. The lenses are produced in an ongoing collaboration in which a CXRO scientist works in IBM's Nanostructure Technology Group at the Thomas J. Watson Research Center in Yorktown Heights, New York.

CXRO-ALS X-Ray Lithography Program

The attempt to "write" ever-smaller patterns on silicon chips has been a natural application for x-ray techniques and technologies, such as those developed by CXRO. As research and applications start to converge toward industrially useful x-ray projection lithography (*sidebar*), a unique environment for collaboration is emerging. CXRO, the University of California at Berkeley, LLNL, and Silicon Valley semiconductor companies are working together toward an advanced lithography R&D program at the ALS. A large measure of impetus came from successful projection printing of features smaller than 0.1 µm by an AT&T group working at Brookhaven National Laboratory's National Synchrotron Light Source. This early success, combined with the realization that the brightness and spectral properties of ALS radiation would be ideally suited for testing techniques and optics for lithography, led to this initiative.

A formative workshop was held in January 1991 to help researchers in government and industry work together toward a strategy for developing an advanced manufacturing capability in the U.S. It familiarized researchers from the semiconductor lithography industry with the capabilities of the ALS and user access to it, and also helped us to understand industry's needs. Technical discussions resulted in consensus on three key issues:

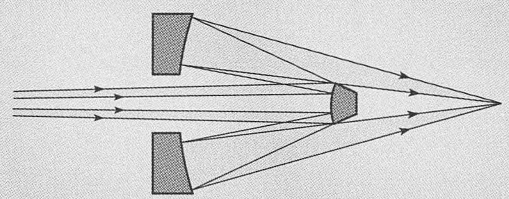
- The x-ray lithography community would benefit from ALS beams from both a bending magnet and a high-brightness undulator. This combination could supply a registration and metrology station and an easily accessible facility for basic testing of components, resists, and masks. There would also be sophisticated testing capabilities for optical elements, optical systems, and various projection technologies that have yet to be developed.
- The time frame for research activities at the ALS (that is, beginning in 1993) is well-matched to industry's needs for pattern transfer below 0.15 μm in the year 2000 and beyond.
- Development of production technologies for structures of these dimensions will require a nearby supporting infrastructure for nanostructure pattern generation, masks, optical coatings, resists, and the requisite synthesis and processing operations.

Smaller Features, Bigger Challenges

One technique for writing a mask pattern onto a chip is proximity printing (shadow casting), which is very much like making a contact print of a photographic negative, albeit with a small gap. This is the more immediately available technique, because no x-ray optics are required. However, the features on the mask, the "negative" that serves as the master for the circuit pattern, must be very nearly as small as those on the chip itself. Such a mask is obviously difficult and expensive to make and repair. Further, the proximity, on the order of a few μm, can result in mask damage in a production environment where silicon wafers must be "stepped" through the system rapidly.

Circuit patterns can also be printed by projection lithography, a technique closely analogous to printing a photograph with an enlarger, but in reverse (see the illustration *below*). AT&T has made great progress in this technique in their experiments at the NSLS. A major obstacle to projection lithography is the need for focusing optics that give sufficient resolution and breadth of field, corresponding to a small, uniformly good pattern across a large chip.

In collaboration with colleagues from the University of Wisconsin Synchrotron Radiation Center, we are developing normal-incidence optics with multilayer coatings for use in the Schwarzschild configuration. (Multilayer-coated optics are described in the next section.) The experimental work will use a Schwarzschild objective, coated with Mo/Si multilayers by CXRO. Optics suitable for manufacturing will be far more complex and demanding.



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An advisory group including industry, university, and national-laboratory representatives met six times in 1991 to develop the specific focus of the program. Their efforts culminated in a "white paper" that calls for a major investment in specialized facilities and infrastructure at LBL. These efforts recently resulted in a notice of initial funding from the Defense Advanced Research Projects Agency.

Multilayer Reflective Optics

Multilayer coatings are good reflectors of x-rays over a broad wavelength range. 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 fabricating multilayers via sputtering techniques, advancing the applications of multilayers in a variety of forefront experiments, and conducting fundamental research into multilayers themselves to improve them and to elucidate their performance limits.*

Multilayer-coated mirrors fabricated in our laboratory have been incorporated into a wide variety of x-ray optical systems in the U.S. and abroad, operating at photon energies ranging from the XUV to the hard-x-ray regions of the spectrum. Our long-wavelength multilayer characterization capabilities were recently enhanced by the development of a self-contained XUV reflectometer. The innovative device is based on a broadband laser-plasma x-ray source and a high-throughput, easily tunable monochromator.

Improving Multilayer Reflectance

Multilayer-coated optics have high reflectance at near-normal incidence, leading to proven and potential applications that range from x-ray astronomy to nanoelectronics. At wavelengths near the multilayer's Bragg peak, these optics provide orders of magnitude more reflectivity than bare surfaces in the XUV and soft-x-ray regions. Molybdenum/silicon multilayers have been demonstrated to have normal-incidence reflectance values exceeding 50% for a limited range of wavelengths longer than 124 Å, which corresponds to the $L_{2\text{-}3}$ edges of silicon. Mo/Si multilayers reflect well in this range, partly because Si is relatively transparent at energies below its absorption edge. At shorter wavelengths, multilayer normal-incidence reflection falls rapidly, presumably because the wavelengths are closer to the size of structural imperfections associated with the layers and interfaces, and the reflectance diminishes.

We are pursuing several means of improving multilayer reflectance at shorter wavelengths. One of them is an ongoing search for new material combinations; we are experimenting with multilayers that alternate ruthenium with carbon, boron, or boron carbide. Near the boron edge, Ru/B_4C multilayers hold great promise as normal-incidence reflectors; multilayers grown and measured in our labs have shown reflectance in excess of 15% at a wavelength of 70 Å. In addition to measuring the normal-incidence reflectance of these structures, we use techniques such as transmission electron microscopy and nonspecular x-ray scattering to study the structural imperfections that are thought to reduce reflectance.

Multilayer Phase Retarders for the Extreme Ultraviolet

Many techniques for examining samples with x-rays call for optical elements that have specific polarization properties, such as linear polarizers and phase retarders. Such optical elements can also be used to generate beams with specific polarization states and to modulate those states. Multilayer phase-manipulating optics are important recent subjects of investigation for CXRO. Developing such optics is a great challenge, but it holds the potential for providing a simple, inexpensive route to polarization control with existing sources.

The physical basis for these devices is the polarization dependence of electromagnetic scattering, combined with the geometry-dependent reflectance of multilayers. When the Bragg reflectance peak is very close to 45° (for a total scattering angle near 90°, as shown in Figure 4-2), the component of the radiation that has an electric vector in the scattering plane is extinguished by the extremely small reflectance at that angle. The result is a linear polarizer. Such devices have been investigated by a number of groups.

A differential phase change between two polarization components of a radiation beam upon interaction with a material can be achieved with a multilayer by using either the reflected or the transmitted beam. Calculations show that such a phase retardation can approach 90° at an energy of 100 eV in the case of the molybdenum-silicon multilayers we are studying. Such a device could be used as a quarter-wave plate to convert linear polarization to circular polarization and *vice versa*. The major challenge is the development of high-quality free-standing multilayers* that can work in the transmission geometry as well as in the reflection geometry. Recent collaborative measurements with the group at Tohoku University, using free-standing multilayers made at CXRO, confirmed the theoretical predictions. As the multilayers were not ideal, improvements in performance can be expected.

At shorter wavelengths, the magnitude of phase retardation in the transmission mode decreases along with the optical constants of the materials and the quality of the multilayers. These limits have not yet been established. From 300 to at least 3000 eV, no optical means of achieving significant phase retardation have been demonstrated.

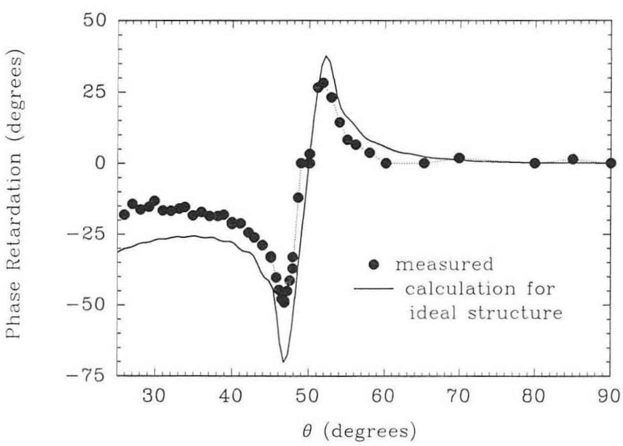
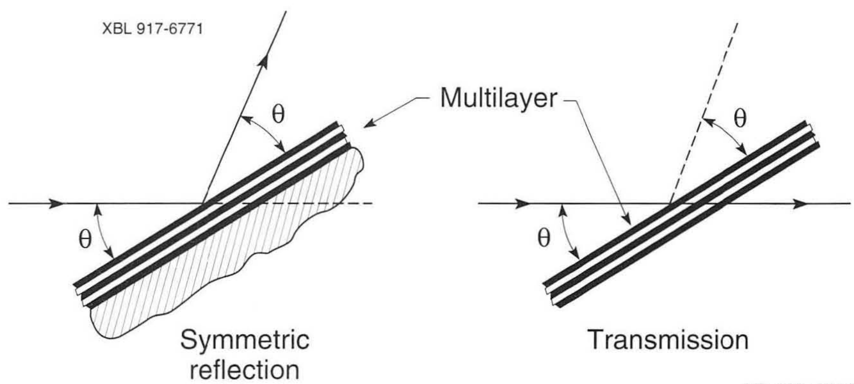


Figure 4-2. Phase retardation of soft x-rays can be obtained in both the reflection and the transmission geometries when the Bragg peak is near 45° (total scattering angle near 90°). The graph shows data from the first measurement of soft-x-ray phase retardation. The experiment used a free-standing Mo/Si multilayer at 97 eV. Also shown is the retardation predicted for an ideal multilayer structure with the same parameters. These measurements were made at Tohoku University.



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* The multilayer *mirrors* used in some of CXRO's other programs are made up of multilayers on a substrate.

XUV Reflectometer

Development of means for characterizing optical elements must go hand in hand with the development of the optics themselves. We recently completed a soft-x-ray/extreme-ultraviolet reflectometer based on laser-induced x-ray emission from a plasma. The plasma is generated by a neodymium:yttrium-aluminum-garnet (Nd:YAG) laser, as diagrammed in Figure 4-3. The reflectometer uses a unique high-throughput monochromator designed and built at CXRO that varies the wavelength transmitted to the optical element being tested. (A monochromator of the same design was used in the High Fluence Laboratory XUV Source that we developed together with Sandia National Laboratories in Livermore, a 1990 R&D 100 award winner.) One of the immediate applications of our new reflectometer will be characterization of our short-wavelength multilayer optics. A recent measurement of the Ru/B_4C multilayer's reflectance is shown in Figure 4-4.

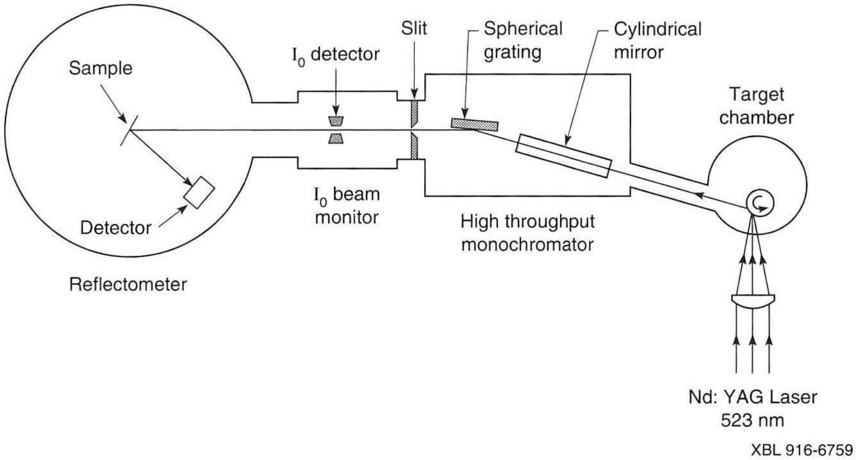


Figure 4-3. The new CXRO extreme-ultraviolet reflectometer uses a laser-produced plasma; the desired wavelength is selected from its broadband x-ray output with a unique high-throughput monochromator designed and built at LBL. (Conventional reflectometers use an x-ray tube of fixed wavelength.) The wavelength range extends from about 30 to 400 Å. One of the immediate applications is characterization of our short-wavelength multilayer optics, such as the Ru/B_4C multilayer.

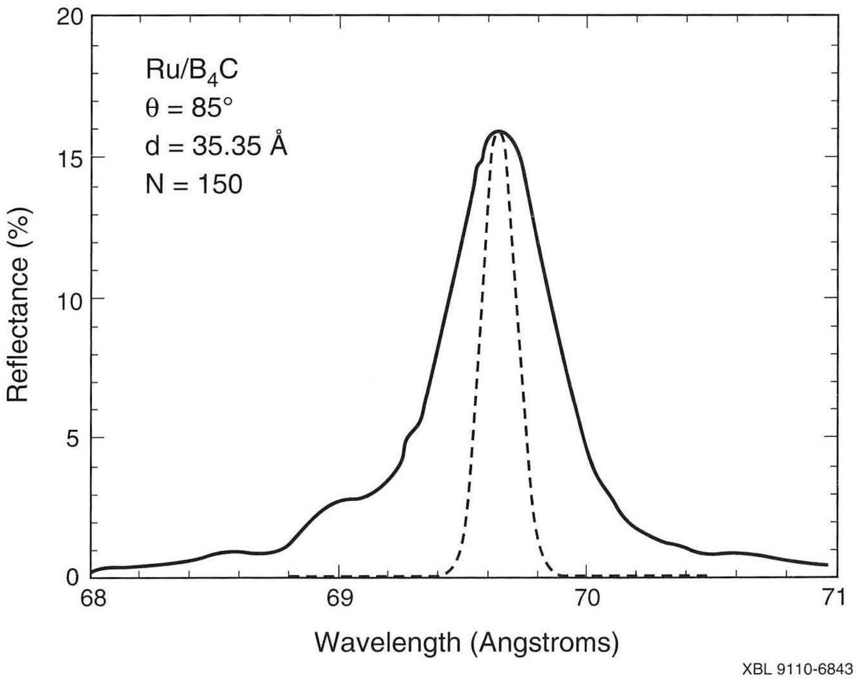


Figure 4-4. With our new XUV reflectometer, we measured this reflectance profile for a Ru/B_4C multilayer designed to reflect at a wavelength of about 70 Å. The dashed curve shows the resolution of the monochromator at these wavelengths.

Soft-X-Ray Small-Angle Scattering

Another application of multilayer-coated optics may fill a significant gap in the coverage of present-day techniques for small-angle soft-x-ray scattering measurements. The SXSA-447 is a combination sample chamber, focusing apparatus, and detector for analyzing particulates and thin films with small-angle diffraction of soft x-rays. Through mathematical analysis of the interference patterns that result from scattering of x-rays by the sample, a variety of characteristics of the sample can be determined, including the sizes of micrometer and sub-micrometer particles within it, the structure of the particles (solids, hollow shells, and so forth), and, in the case of thin films, periodic or nonperiodic structures. The incident beam is gathered and focused with one of the periodic multilayer-coated mirrors that are among CXRO's specialties. The apparatus won a 1990 R&D 100 award, given annually by *Research and Development Magazine* to recognize the year's 100 most-significant technical innovations.

Hard-X-Ray Microprobe

The latest development of our hard x-ray microprobe (Figure 4-5) is a portable version that can be readily moved from one synchrotron-light source to another. It is similar in operating principles to its predecessor, and appears to be comparable in performance as well, providing both micrometer spatial resolution and femtogram elemental resolution with 30-second exposures. The data the microprobe provides—what elements a sample contains and where they are concentrated—are useful in many scientific disciplines.

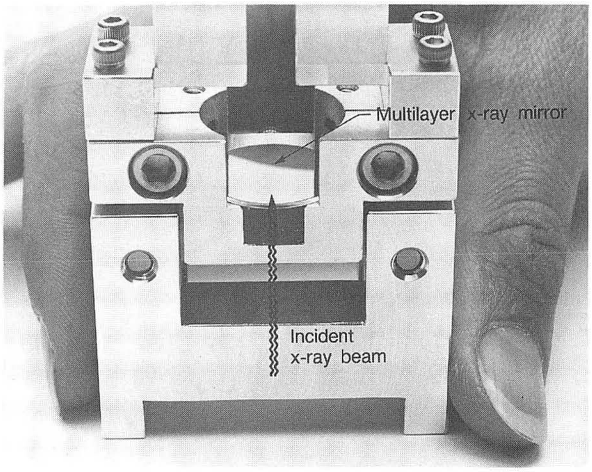
The new microprobe is based on a pair of concave, spherical multilayer-coated mirrors that serve both as focusing elements and as monochromators for the incoming x-ray beam. Following the mirrors, we have a scanning stage to raster-scan the sample; an optical microscope for prealignment of the sample; an x-ray fluorescence detector; and a beam-intensity monitor. A computer controls the system and provides rapid analysis and display of the recorded data on elemental concentrations. Figure 4-6 shows a typical set of data: the concentration of titanium in a silicon carbide ceramic. The false-color display makes it easy to visualize complicated quantitative data.

The portable microprobe has thus far achieved a spot size of 2 μm × 2 μm at a bending-magnet beamline at the NSLS. By tuning the mirrors to energies just below the K absorption edge of major elements in a sample, the elemental sensitivity for lighter trace elements is greatly improved, as compared to electron-stimulated techniques.

Recently we used our system on a high-energy x-ray undulator beamline at the Cornell High Energy Synchrotron Source (CHESS). In this collaborative experiment with Oak Ridge National Laboratory, the University of Chicago, Argonne National Laboratory, and Cornell University, we studied different x-ray optical elements for potential use on microprobe beamlines at the new third-generation x-ray facilities. Our multilayer mirrors were able to provide the best focus of a 250 μm × 250 μm undulator beam, achieving a 4 μm × 8 μm spot. Working with several user groups, we are currently developing plans for dedicated microprobe beamlines that use focusing optics on the NSLS, the ALS, and the Advanced Photon Source (APS).* With the lower emittance of the ALS and APS, we will be able to achieve a beam spot size of 1 μm × 1 μm. The APS, where we will have access to a hard-x-ray

* A 7-GeV "third-generation" synchrotron-radiation source being built at Argonne National Laboratory.

Figure 4-5. The hard-x-ray microprobe has been used to achieve 2-μm spatial resolution, along with femtogram elemental sensitivity, at Beamline X26 of Brookhaven National Laboratory's National Synchrotron Light Source. The heart of the microprobe is a pair of W/C multilayer mirrors used at grazing incidence in Kirkpatrick-Baez geometry. They focus the beam of synchrotron radiation to a small, intense spot on the object, which fluoresces with x-rays characteristic of its elemental composition. The fluorescence x-rays are detected and analyzed by a lithium-drifted silicon detector, which is placed orthogonally to the incident beam to reduce the scattered background. The vertical and horizontal spatial resolutions shown here were obtained by scanning a sharp knife edge across the beam. The resolution in both cases is 2 μm full width at half maximum.



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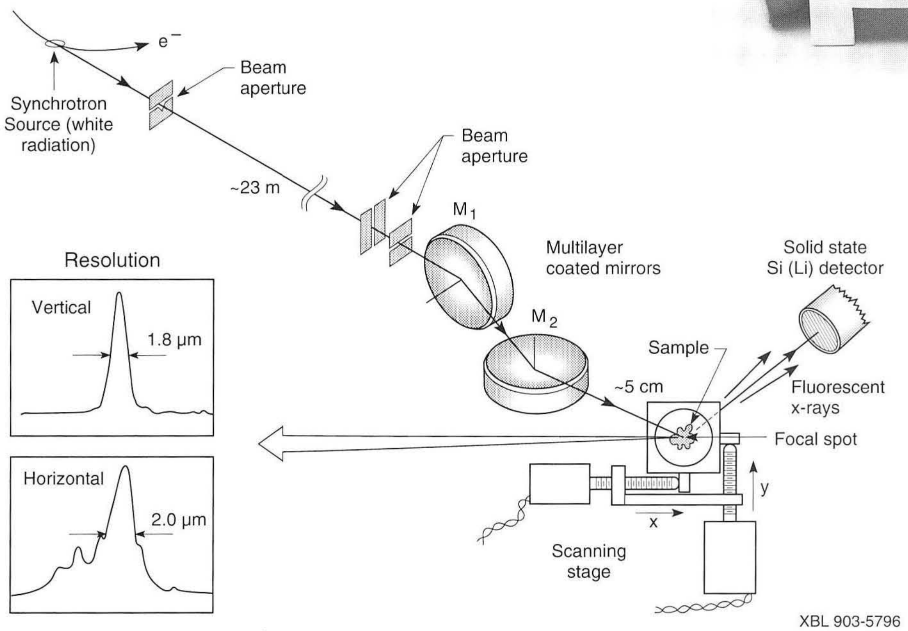

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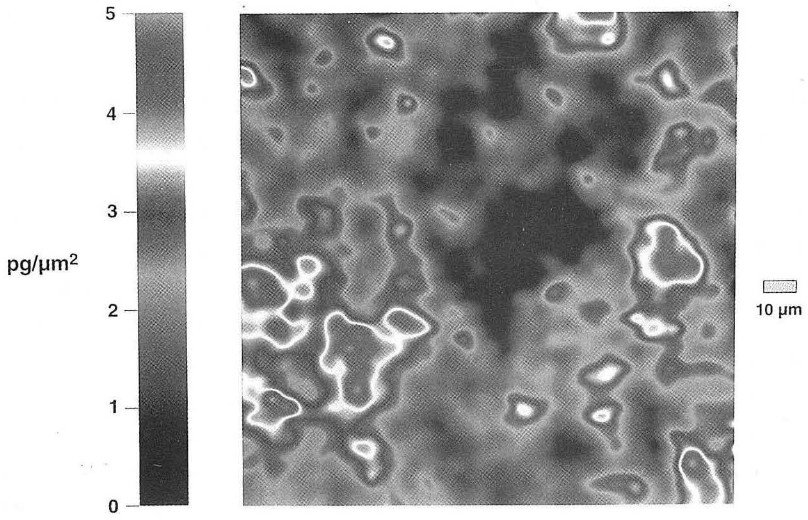

Figure 4-6. Data from the hard-x-ray microprobe shows the concentration of dilute amounts of titanium in a silicon carbide ceramic. In this false-color plot (rendered here in black and white), the color bar quantifies the range of concentrations. Such ceramics exhibit characteristic tensile and fractile strengths based on the uniformity of titanium through the ceramic. This can be controlled by the temperature of the material during the sintering process. The measurements were made at the NSLS.

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undulator, will also provide higher intensity. The microprobes at these facilities will use elliptically curved multilayer mirrors to improve the focus, and will scan the beam instead of the sample for greater experimental flexibility.

Spectroscopy with X-Rays

The Center for X-ray Optics has always been involved in the design, construction, and implementation of new types of x-ray and XUV spectroscopic instrumentation, both for synchrotron radiation research and for other applications. We have continued our efforts to develop new spectroscopic instrumentation with desirable properties such as high resolution, high throughput, simplicity, and low cost.

XUV Monochromators and Spectrometers

To achieve high spectral resolution without compromising throughput, we have continued our investigations into monochromators, spectrometers, and spectrographs using plane gratings with varied line spacing. Such gratings allow a flat (or erect) focal plane and can thus be scanned in wavelength without the complex scanning mechanisms required by more-conventional designs, such as spherical grating monochromators. One product of this work has been the High Resolution Streaked Spectrograph used for x-ray-laser studies at Lawrence Livermore National Laboratory's Nova facility. In addition, we have continued our studies of advanced monochromator and spectrometer designs for several synchrotron-radiation applications.

Earlier studies established that the varied-line-spacing designs can achieve resolution at least as high as conventional designs with the same tuning range. Recently we have developed computer codes that use ray-tracing techniques to simulate synchrotron radiation and estimate grating efficiency. In a theoretical study of such a monochromator for a U5.0 beamline at the ALS, we confirmed that it would provide high spectral resolution, high throughput, and small spot size with a simpler and less-costly tuning mechanism. To verify these results experimentally, we are planning to build a new varied-line-spacing plane-grating monochromator for use at BESSY.

In another application-oriented program, we are investigating design options for dividing a soft-x-ray beam among several users. This is challenging, because an undulator source in a modern, low-emittance storage ring puts out a very thin, quasi-monochromatic beam that does not lend itself readily to either spatial or spectral beamsplitting. The possibilities that we are examining include wavefront splitting, high-efficiency timesharing, and splitting according to diffraction-grating order. The findings will be especially relevant for beamlines at third-generation synchrotron-light sources. Present studies are focused on the ALS U3.9 undulator beamline, which is intended for biological microscopy and research in coherent optics.

A high-throughput, high-resolution spectrograph based on varied-line-space gratings, designed and built by CXRO, is installed on the two-beam x-ray laser chamber at Nova. The instrument continues to provide time-resolved spectral profiles for x-ray emissions in the wavelength range of 155-210 Å, as illustrated in Figure 4-7. It is now fully operational and achieves a spectral resolving power ($\lambda/\Delta\lambda$) in excess of 20 000. The line-profile data have proved to be invaluable for understanding physical processes in the Nova x-ray laser facility and are in demand for diagnostic measurements of other kinds of laser-produced plasmas. CXRO is involved

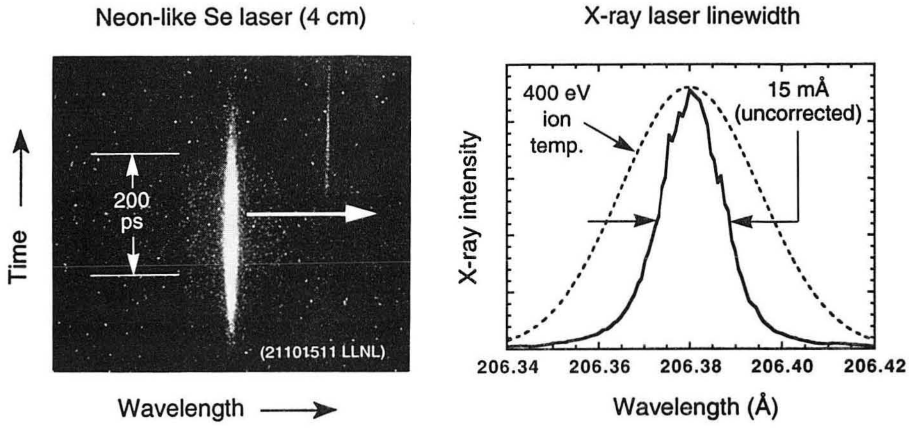

Figure 4-7. Using the CXRO time-resolved high-resolution spectrometer, x-ray-laser line narrowing has been measured for several lasing lines at LLNL's Nova facility. Shown here is the 206.38-Å line of neon-like selenium from a target 4 cm long. The line width is 15 mÅ full width at half maximum. Though not yet corrected for instrumental broadening, this is significantly narrower than the 36 mÅ that would correspond to a 400-eV Doppler-broadened emission line in the same plasma.

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in design studies for a new instrument that will operate at 44.83 Å, which is the wavelength of the Ni-like-tantalum laser, and is considered ideal for holographic imaging of biological material.

Coronary Angiography

X-ray beams from a synchrotron source—monochromatic, well-collimated, and intense—provide unique opportunities for medical imaging. One of them is a new method of coronary angiography that uses venous injection of contrast agent, as opposed to arterial injection. For several years we have collaborated with colleagues from Stanford, Brookhaven, and elsewhere who are working on this new, safer method. A new step toward large-scale tests of clinical applicability was taken in 1990 with the completion of a dedicated medical-imaging facility on a special wiggler beamline at the NSLS. Three patients' hearts have been imaged there; the results are not yet equivalent to conventional angiograms, but they are approaching clinical usefulness, and the technique is dramatically easier and safer for the patients.

Progress toward Clinical Quality

Using an upgraded imaging system previously tested at the Stanford Synchrotron Radiation Laboratory, three patients were examined in late 1990 and early 1991. Figure 4-8 shows the resulting image from one of the patients. Shown next to it is an earlier angiogram from the same patient, taken with the conventional, highly invasive, and somewhat risky technique of x-ray angiography using arterial injection of contrast agent.

The images obtained thus far clearly indicate that large portions of both the left anterior and the right coronary arteries can be examined with this method. Various changes are now underway to further improve the image quality. Later, a medical research team will begin to use this technique on a large group of patients, comparing the new method to standard coronary angiography and also studying the effect of various medical treatments on the progression of coronary artery disease.

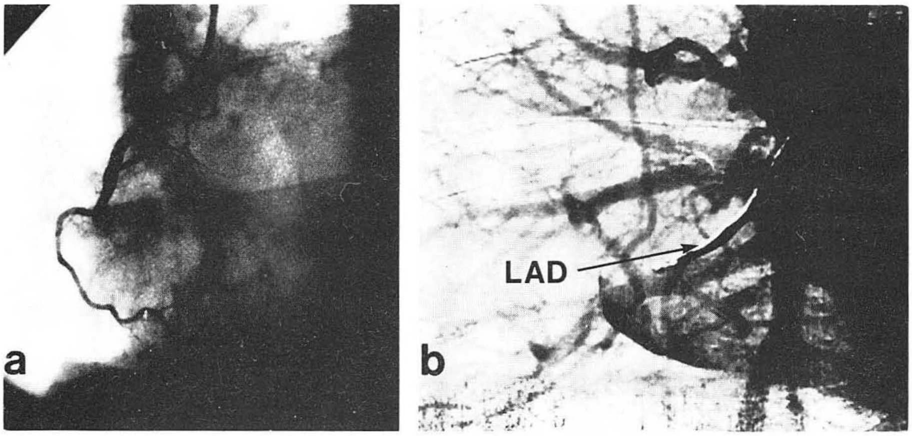

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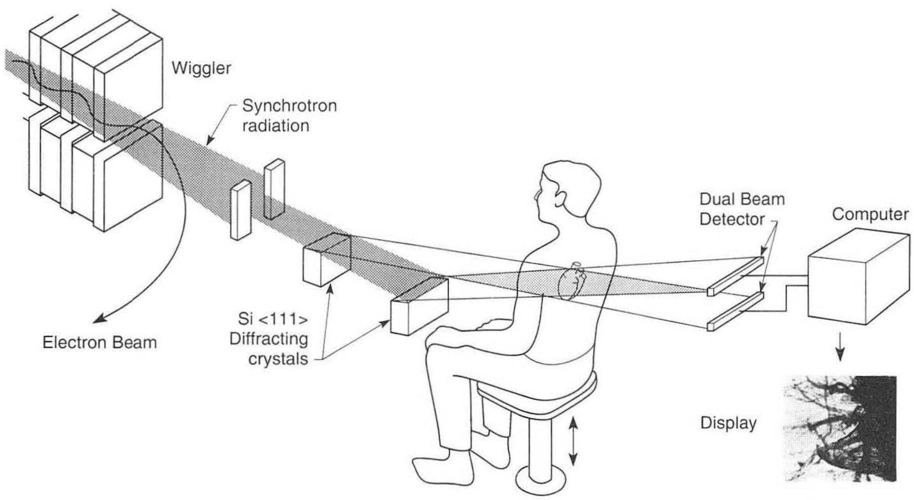
Figure 4-8. An angiogram made with arterial injection of contrast agent, an invasive and risky procedure, is shown at *left*. At *right* is an angiogram of the same patient taken with the synchrotron-radiation method, which uses venous injection. Clearly visible in both images is a total occlusion of the right coronary artery. The patient attended a Broadway musical the evening after the synchrotron angiogram—a recovery time unheard of with arterial angiography. This work is part of a long-term collaboration with Stanford University; Brookhaven National Laboratory; the North Shore Hospital on Long Island, NY; the Veterans Administration hospital in Palo Alto, CA; and the University of Tennessee. The images were obtained on the X17 beamline at the NSLS.



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