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Soft X-Ray Spectromicroscopy

Materials Characterization

on a

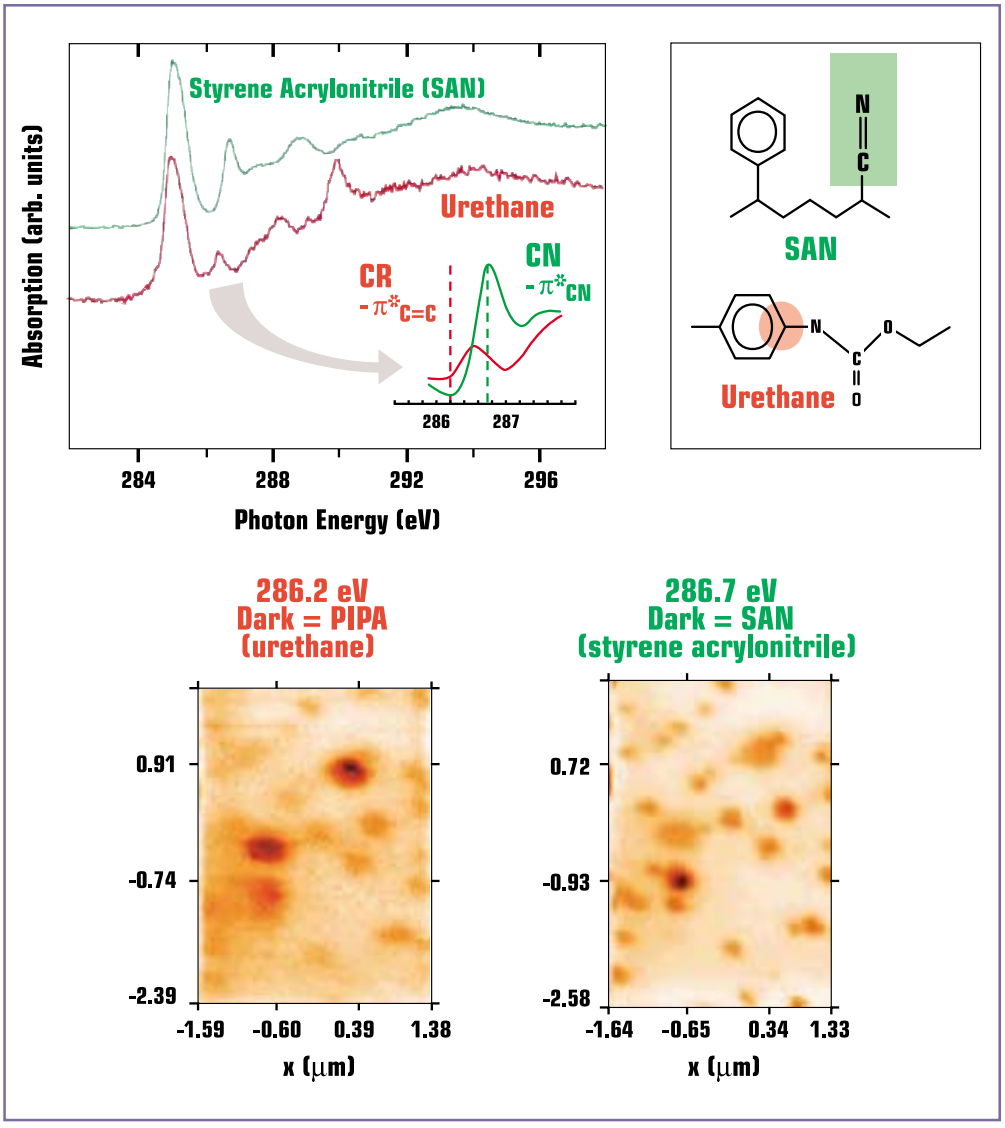
Microscale

Cover Photo:
The new Advanced Light Source building retains the dome that covered the first large accelerator at the Lawrence Berkeley National Laboratory, the 184-Inch Cyclotron built by Laboratory founder Ernest Orlando Lawrence during World War II.

A New Opportunity

Spectro Microscopy

X-Ray Microanalysis Of High-Tech Materials



X-ray microscopy at photon energies near characteristic x-ray absorption peaks for carbon bonds makes it possible to differentiate submicron particles of styrene acrylonitrile (SAN) from particles of urethane-based polyisocyanate-polyaddition (PIPA) in a polyurethane matrix. [Images made at the ALS. Courtesy of A. Hitchcock, McMaster University; E. Rightor and W. Lidy, Dow Chemical; and T. Warwick, ALS. Spectra taken at the National Synchrotron Light Source. Courtesy of H. Ade, North Carolina State University.]

In today's world of high-tech materials, researchers need to examine samples on a local scale often measured in fractions of a micrometer, rather than observing averages over large areas. The patterns of integrated circuits provide the most obvious example. Domains in magnetic recording media constitute another, as does the interleaved structure of metal, ceramic, or polymer composite materials. Moreover, many of the additives that scientists often mix in to improve the performance of permanent magnets, superconductors, and structural materials end up distributed as second-phase particles with a different structure and composition than the matrix in which they are imbedded.

While x-ray absorption and photoelectron spectroscopy of atomic core levels are well-developed tools for obtaining elemental composition, chemical state, and structural information from complex materials and their surfaces, they have not traditionally provided significant spatial resolution. But now, owing to the dramatically enhanced brightness of the newest synchrotron radiation sources, such as the Advanced Light Source (ALS) at the Lawrence Berkeley National Laboratory, researchers are able to carry out these x-ray spectroscopies on a microscopic scale (spectromicroscopy), thereby providing the spatial resolution required to address a wide range of materials microcharacterization problems, including analysis of the intricate sub-micrometer features on a microcircuit chip, in-situ chemistry and metallurgy of composites, and measurements of spatially inhomogeneous chemical reactions.

At the ALS, we are looking forward to a wealth of new applications resulting from the union of established x-ray spectroscopy techniques with imaging. In the following pages, we will specifically illustrate the potential of soft (long-wavelength) x-ray spectromicroscopy for providing spatially resolved information about materials and surfaces. Along the way, we will review the properties of synchrotron radiation that make it suitable for this kind of materials microcharacterization, summarize spectroscopy techniques, and describe how spatial resolution and imaging are achieved. We will finish by describing how to obtain information about the ALS and how to initiate a research program.

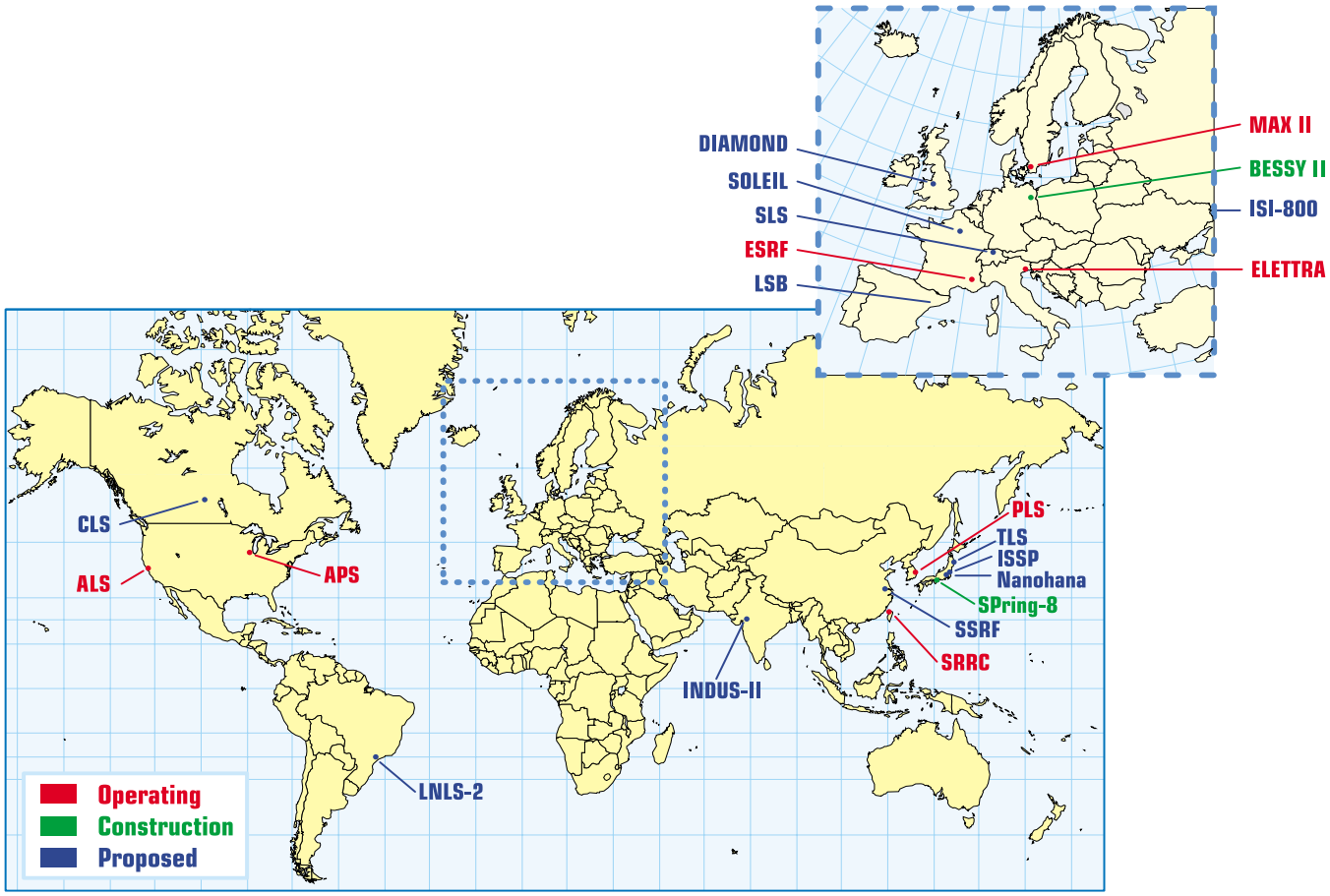
For a summary of the spectromicroscopy facilities currently available at the ALS, please turn to the loose-leaf pages inserted in the pocket of the inside back cover of this brochure.

The Advanced Light Source (ALS) at the Lawrence Berkeley National Laboratory is located in the hills above Berkeley, California, adjacent to the campus of the University of California, in the midst of the varied high-tech industrial infrastructure of the San Francisco Bay Area.

Synchrotron Radiation

Brightness

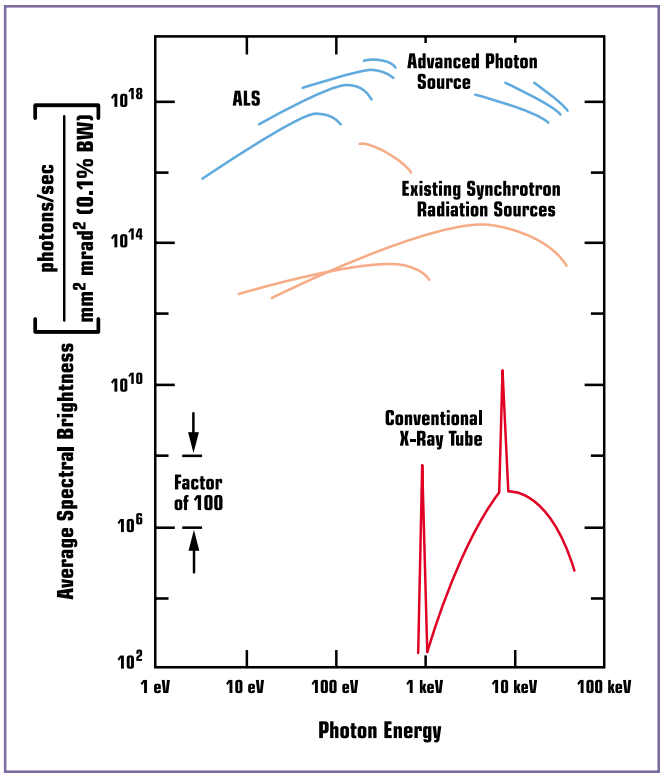
The Competitive Frontier of Synchrotron Radiation



The locations of existing and planned ultrabright third-generation synchrotron sources in countries around the world illustrate the importance of state-of-the-art materials microcharacterization tools in an economically competitive global economy.

In the 1960s, scientists interested in the properties of materials and surfaces began to exploit the ultraviolet and x rays emitted by electrons in synchrotrons built and operated for high-energy physics research. By the early 1980s, new facilities based on electron storage rings were springing up in the major industrialized nations. Not only were these dedicated to synchrotron radiation, but many were also designed to achieve much higher brightness than previously obtainable.

Users of synchrotron radiation now recognize that brightness—defined as the flux of photons per unit source area and per unit solid angle of emission—is a critical performance specification (see box on page 10). In short, with a high-brightness source, x-ray optical systems can use the photons efficiently to get the highest possible flux onto the sample. For these reasons, within a decade, work began on the third generation of facilities, with great expectations for benefits from still higher brightness.



These performance curves demonstrate the orders-of-magnitude jump in the brightness available from the ALS in the soft x-ray spectral region (and those of its sister facility, the APS, in the hard or short-wavelength x-ray spectral region) over that available from second-generation synchrotron sources and from conventional continuum and line sources in the laboratory.

The ALS is one of the first third-generation facilities, with construction completed in March 1993. Designed explicitly for the production of soft x-ray and ultraviolet synchrotron radiation with the highest possible brightness, the ALS is now being operated by the Lawrence Berkeley National Laboratory of the University of California for the U. S. Department of Energy as a national user facility that is available around the year for research by scientists from industrial, academic, and government laboratories.

The ALS organization is committed to the rapid development and application of spectromicroscopy techniques that exploit the brightness of the source in collaboration with academic and industrial users. The industrial connection to synchrotron radiation is particularly important, and it is one reason countries around the world are building high-brightness synchrotron sources. In addition to two in the United States (the ALS and the Advanced Photon Source at Argonne National Laboratory near Chicago), Europe and Asia together have five in operation, and more are planned or under construction.

Phase Space & Brightness

The statistical-mechanical concept of phase space applies to the source of the synchrotron radiation (the electron beam) and to the radiation itself. For electrons, the phase-space area (the emittance) is the product of the beam size and divergence. Emittance is somewhat like a temperature, so the ALS storage ring is said to have an ultralow-emittance or ultracold beam. The emittance sets a lower limit for the phase-space area of the light beam, defined as the product of the effective source size and radiation cone angle. Brightness is the density of photons in this phase space. Although optical systems can manipulate the source size and angle (e.g., by focusing), they cannot reduce their product without losing some of the light, as with an aperture, and therefore cannot increase the brightness. Optical systems that transport the radiation in general collect light in only a portion of the phase-space area of the incident radiation, The useful phase-space area (the acceptance) is given by the product of the maximum source size and radiation angle that the system can collect. Similarly, there is a sample acceptance defined by the spot size and beam divergence required at the sample. Since the highest flux is delivered to the sample when the phase-space area of the photon beam matches the acceptances of the optical system and the sample, brightness is almost always more important than flux alone.

A Small Beam Emittance Maximizes The Brightness

The key parameter of the photon source for spectromicroscopy is its brightness. For a photon beam with a Gaussian density distribution, the brightness $B(\varepsilon)$ on the optical axis of an undulator is approximately

$$B(\varepsilon) = \frac{F(\varepsilon)}{(2\pi)^2 \Sigma_h \Sigma_v \Sigma_{h'} \Sigma_{v'}},$$

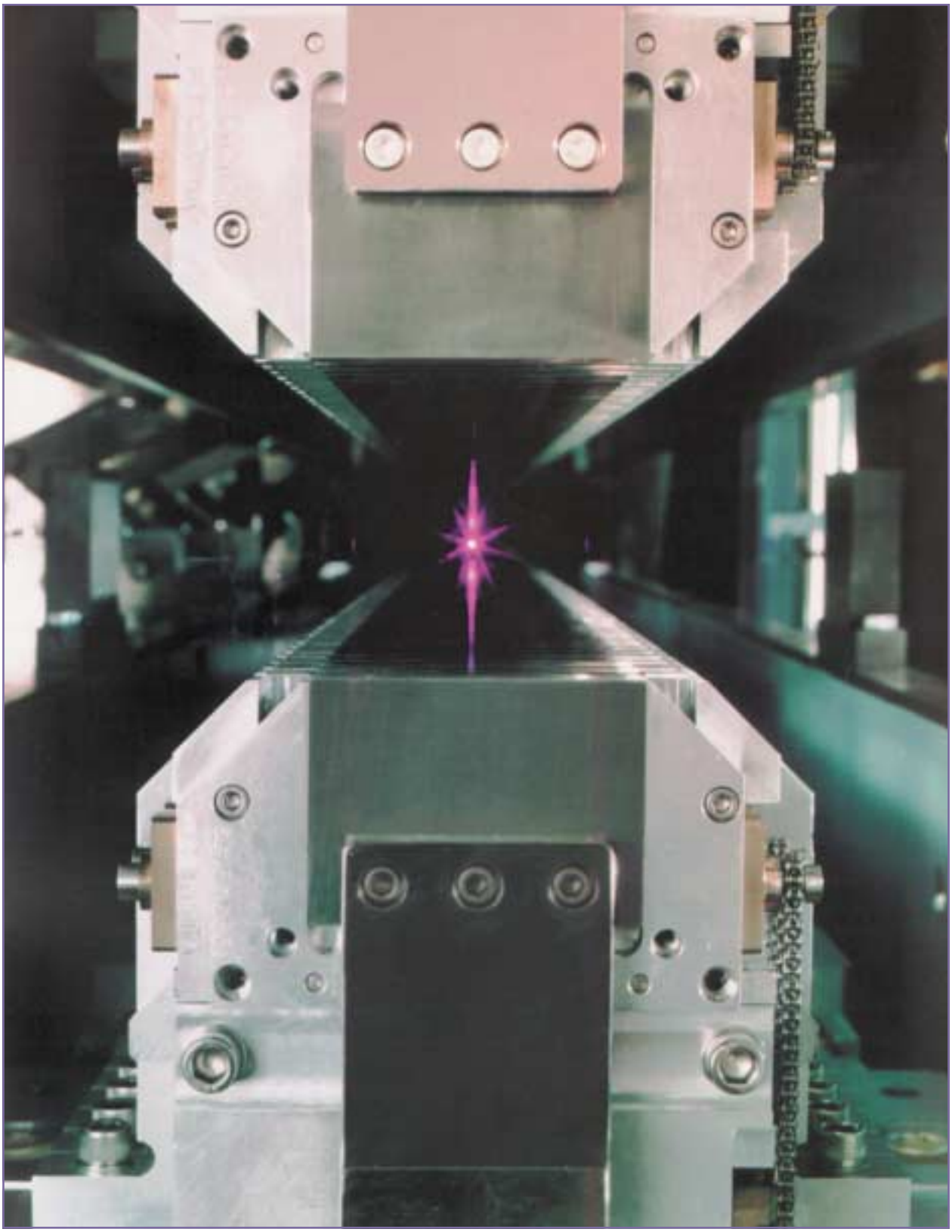
where $F(\varepsilon)$ is the on-axis flux for photon energy ε, Σ_h and Σ_v are the RMS horizontal and vertical source sizes, and $\Sigma_{h'}$ and $\Sigma_{v'}$ are the RMS horizontal and vertical radiation-cone half-angles.

The electron beam contributes to both the effective source size and divergence. For a single electron in an undulator radiating into a cone with an RMS half-angle $\sigma_{r'}$, there is a corresponding wavelength-dependent, diffraction-limited source size $\sigma_r = \lambda/4\pi\sigma_{r'}$. The effective source size Σ_h or Σ_v is the quadrature sum of σ_r and the size σ_h or σ_v of the Gaussian electron beam [e.g., $\Sigma_h = (\sigma_h^2 + \sigma_r^2)^{1/2}$]. Similarly, the effective divergences $\Sigma_{h'}$ and $\Sigma_{v'}$ are obtained from the divergence $\sigma_{r'}$ of the photon beam from a single electron and the divergences $\sigma_{h'}$ and $\sigma_{v'}$ of the electron beam.

Third-generation synchrotron light sources optimize the brightness by generating a beam with a small emittance (product of the electron-beam size and divergence). If the emittance is small enough relative to the wavelength λ, the radiation is diffraction limited (i.e., the phase space occupied by the electron beam is less than the phase space occupied by a diffraction-limited photon beam)

$$\varepsilon_h = \sigma_h \times \sigma_{h'} < \sigma_r \times \sigma_{r'} = \lambda/4\pi \qquad \varepsilon_v = \sigma_v \times \sigma_{v'} < \sigma_r \times \sigma_{r'} = \lambda/4\pi.$$

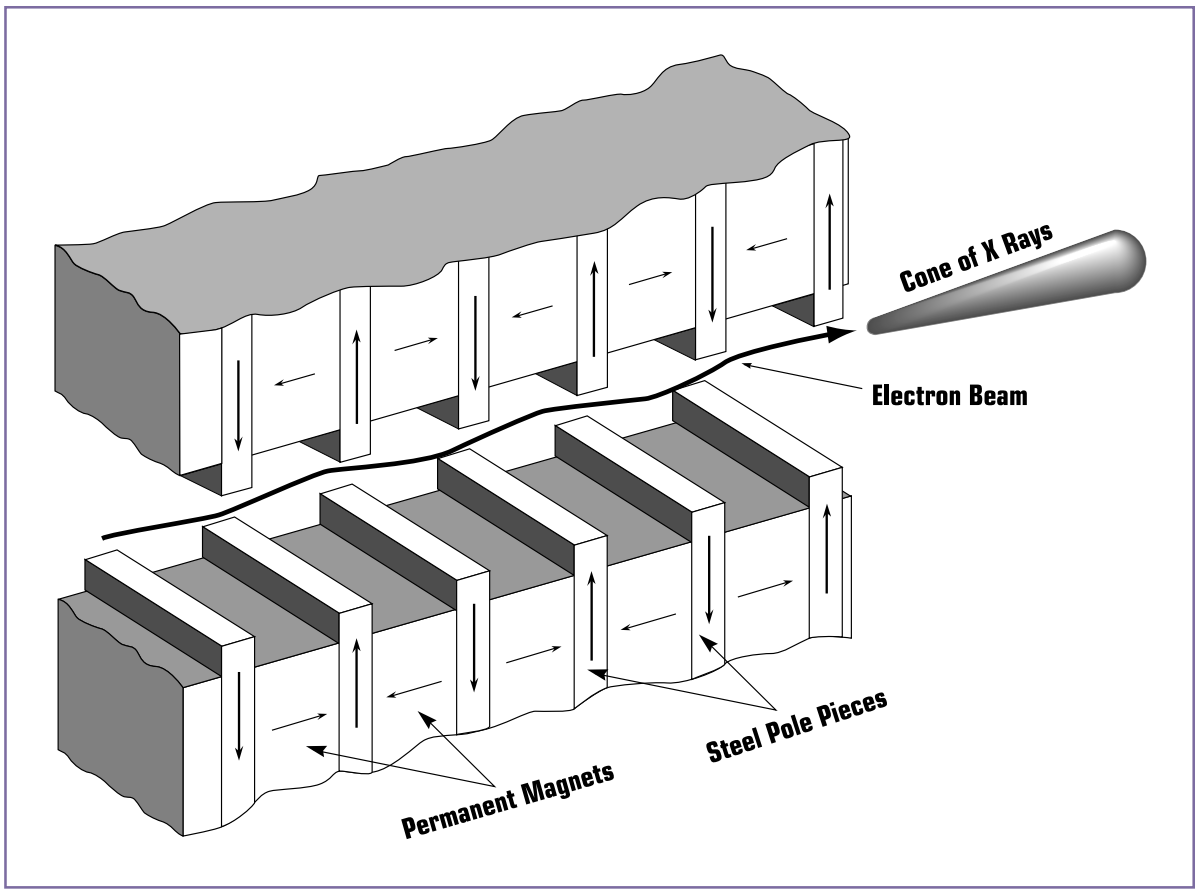
Under these conditions, the radiation has the spatial (transverse) coherence properties of a laser and it is possible to collect all of the light and focus it to the smallest possible size without loss. At the ALS, the vertical emittance ε_y is about 10^{-10} m·rad (1 Å·rad) while the horizontal emittance is about 4×10^{-9} m·rad. This means that the ALS has laser-like properties in the vertical direction at wavelengths as short as 10 Å.



Strobe-light simulation of the electron beam in the ALS immediately suggests the laserlike qualities of the ultrabright light produced by the beam.

Third-Generation Sources

The Emphasis Is On Undulators



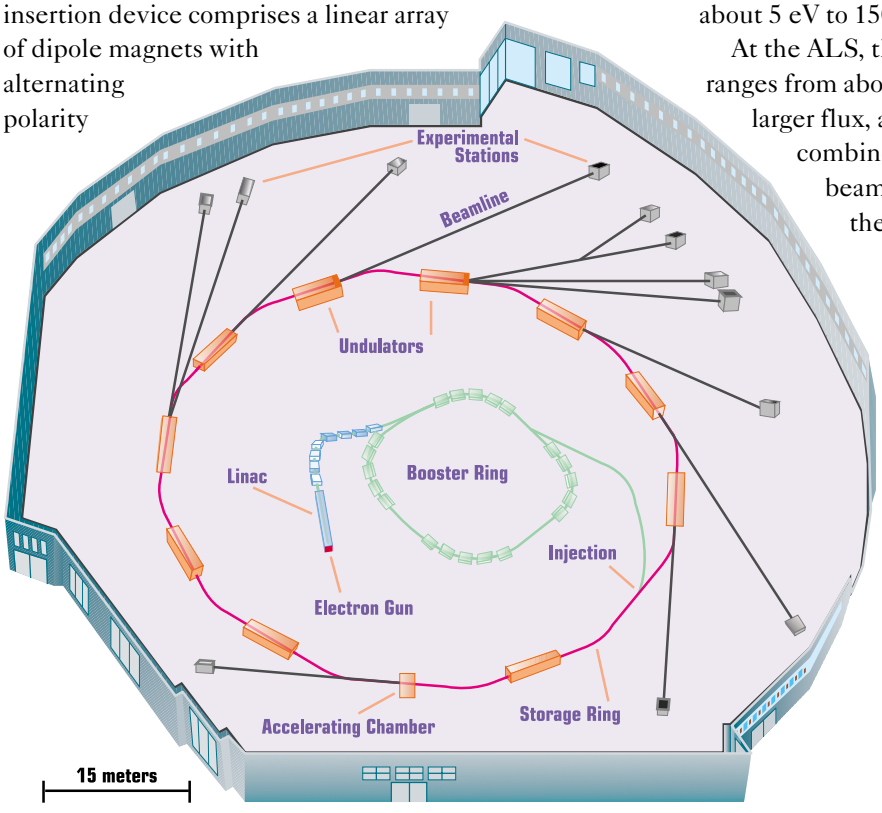
The undulator sources of the ALS are carefully engineered arrays of permanent magnets that produce precisely periodic magnetic fields to generate the brightest possible beams of soft x rays.

In the first synchrotron sources, the radiation came from the bend magnets in the curved sectors of an electron accelerator. Relativistic electrons emit synchrotron radiation when a magnetic field bends them into a curved trajectory. At any point on the trajectory, the synchrotron radiation emerges in a narrow cone tangential to the path of the electron with a flux and spectral range that depends on the electron energy and the magnetic field. As the electron sweeps around the curve, it generates a horizontal fan of light. In practice, an aperture determines how much of the horizontal fan is collected (typically, up to several milliradians). For the ALS, the highest beam energy is 1.9 GeV, where the bend-magnet field is about 1.35 Tesla, so that useful fluxes are available at photon energies above 10 keV.

In third-generation light sources, the storage ring is specifically designed to include special magnetic structures known as insertion devices (undulators and wigglers). Although designs differ, the most common insertion device comprises a linear array of dipole magnets with alternating polarity (i.e., N-S-N-S and so on). The array generates a sinusoidal vertical field that drives an electron into an oscillating trajectory in the horizontal plane with the same period as the field. Each dipole is a bend-magnet source radiating along the axis of the undulator.

The defining feature that separates an undulator from a wiggler is that the maximum angular excursion of an electron relative to the axis of the device is less than the natural opening angle of the synchrotron-radiation cone, so that the radiation emitted from each pole interferes constructively. As a result, the broad radiation spectrum from individual bends squeezes into a series of sharp spectral peaks comprising a fundamental and a set of harmonics. Mechanically opening and closing the vertical gap between the undulator poles adjusts the undulator field and thereby the photon energies (wavelengths) at which the peaks occur. For the values of the periods (5 to 10 cm) and maximum fields (less than 1 Tesla) of the ALS undulators, the spectral range is currently from about 5 eV to 1500 eV.

At the ALS, the number of poles in an undulator ranges from about 80 to about 180, resulting in a much larger flux, as compared to a bend magnet. This, combined with the low emittance of the electron beam, is the source of the high brightness of the ALS undulators.



The ALS comprises an accelerator complex (electron gun, linear accelerator or linac, booster synchrotron, and storage ring), an experimental area (beamlines and experimental stations), and a building to house them. The brightest sources of synchrotron radiation—and therefore best for spectromicroscopy—are the magnetic structures called undulators that reside in the straight sections of the storage ring.

Spectroscopy

The
ALS

New Power & Versatility For X-Ray Spectroscopy

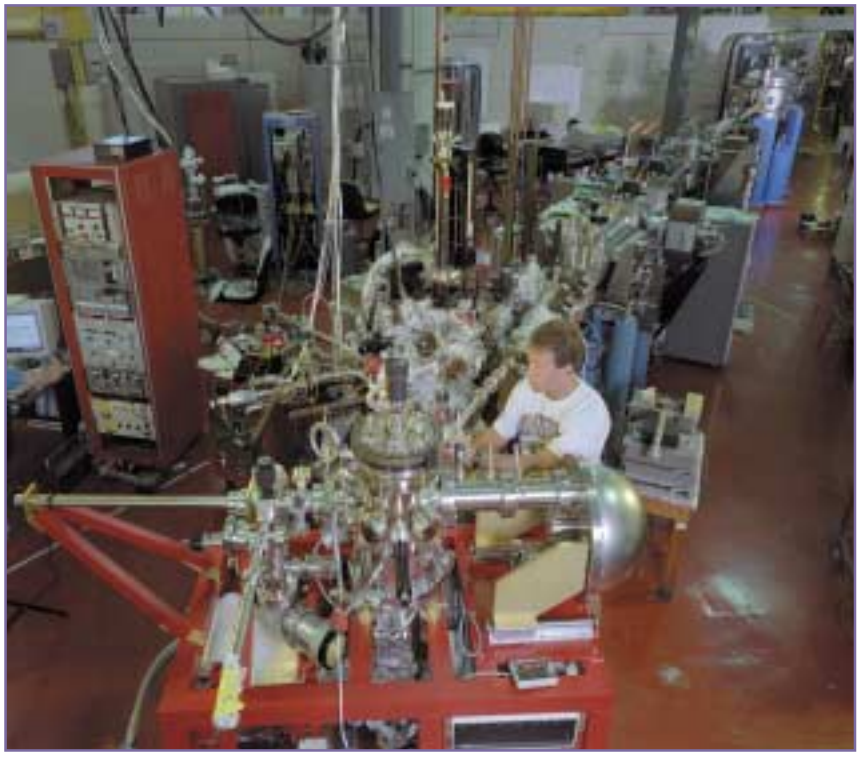
The magnetic force between the upper and lower rows of magnets in this ALS undulator ranges up to 40 tons, yet the placement of the magnet poles must be accurate to better than 20 micrometers over the 4.5-meter length of the device in order to achieve the highest brightness.

In addition to being at least 100 times brighter than sources available before, the ALS offers the traditional virtues associated with synchrotron radiation as compared to conventional laboratory sources of x rays: high flux and variable (tunable) photon energy, as well as tightly collimated beams and controllable polarization. These properties make the ALS an ideal source for soft x-ray spectroscopy based on the atomic core levels in complex materials.

Since the presence or absence of long-range order does not strongly affect core levels, they are particularly suited for probing short-range order and local properties (e.g., atomic coordination and oxidation states). Because of their localized nature, they inherently provide elemental identification in spectroscopy experiments. Complex materials can be dissected sequentially by tuning to the absorption edges of the constituent elements.

Among core-level spectroscopies, laboratories throughout the world use x-ray photoelectron spectroscopy (XPS) as an analytical technique for materials characterization. Typical XPS instruments available commercially include a Mg- or Al-Kα x-ray source, filtered to send a monochromatic beam into a spot possibly as small as 30 micrometers in diameter. In addition to elemental analysis, XPS can also determine the valence state and bonding environment of atoms near a sample surface; it can identify organic functional groups of polymers; and it can characterize very thin, layered structures. Already a powerful technique in the laboratory, XPS has greatly expanded capabilities when performed at a modern synchrotron radiation source, such as the ALS, where the high flux, high spectral resolution, and tunable photon energies can be fully exploited.

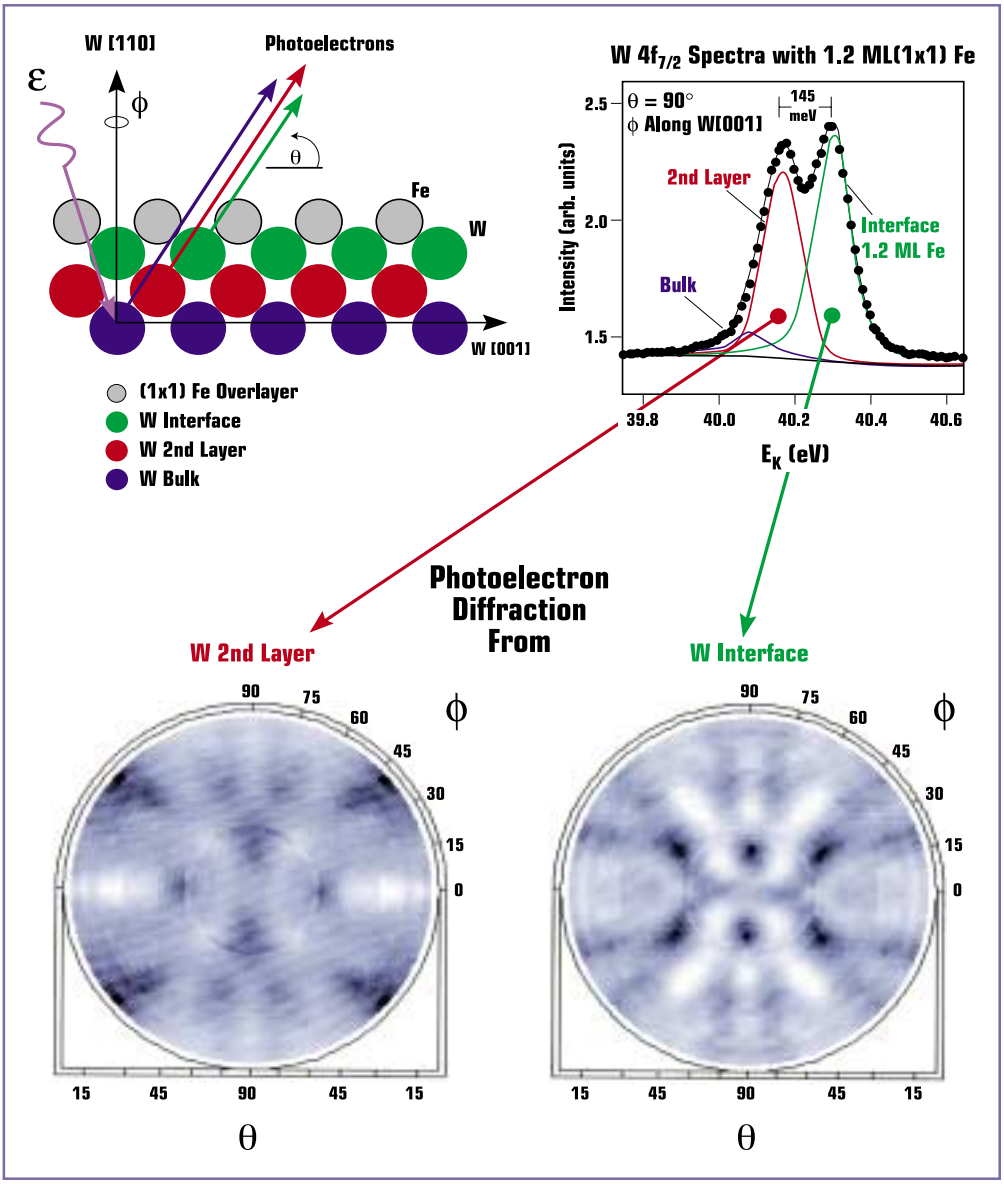
Another spectroscopic technique based on excitation of electrons from core levels, near-edge x-ray absorption fine structure spectroscopy (NEXAFS), provides spectra with signatures characteristic of the chemical bonding of the element excited by the x rays. It is easy to perform when the x-ray source is tunable. Consequently, the high flux and tunable photon energy available at the ALS not only make this technique practical but extend its capabilities to include real-time studies of the dynamics of localized surface chemical changes. And because it makes efficient use of every photon and thereby minimizes radiation damage, NEXAFS is also ideal for studies of large organic molecules.



Undulator beamline 7.0 at the ALS serves multiple experimental stations (foreground and immediately behind) capable of NEXAFS and XPS spectroscopies.

Photoelectron Spectroscopy

High Flux, High Resolution, & Tunable Photon Energy



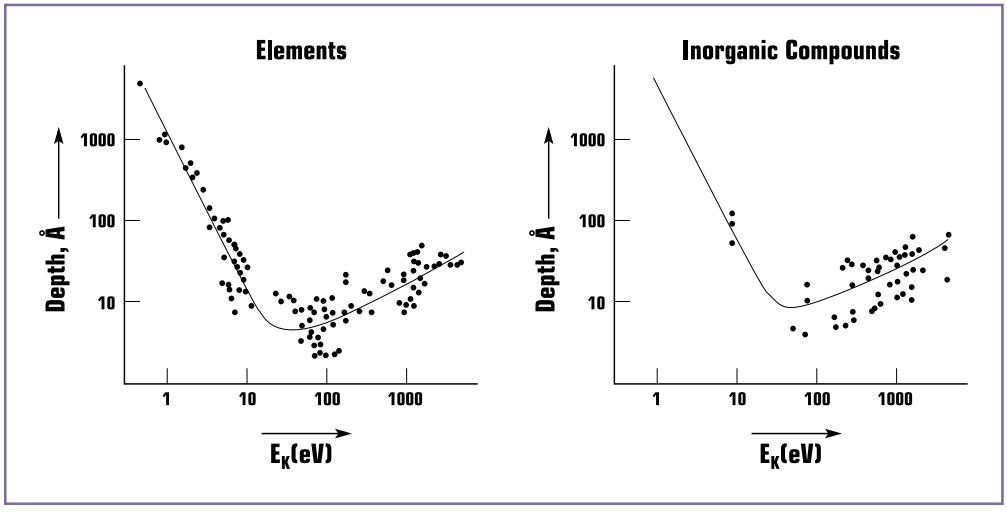
Photoelectron diffraction gives the atomic geometry of a monolayer of iron deposited on a tungsten (110) surface. The XPS spectrum for tungsten 4f photoelectrons has three primary components: one peak due to atoms at the interface next to iron, a second peak due to next-neighbor atoms in the second layer below the iron, and a weak feature due to deeper atoms. Comparison of experimental diffraction patterns, such as those shown for interfacial and next-neighbor atoms, with calculated diffraction patterns for candidate structures shows that the iron sits in a bridge site 2.16 Å above the interfacial tungsten atoms. [Data taken at the ALS. Courtesy of E. Tober, IBM Almaden Research Center, and C. Fadley, University of California, Davis.]

The intense flux of tunable synchrotron radiation that the high-brightness undulator beamlines at the ALS supply to a sample brings enormous benefits to x-ray photoelectron spectroscopy (XPS). For example, with the substantial count rates resulting from high flux, researchers can exploit the fine energy resolution available from ALS beamlines (typically 1 part in 5000). High resolution facilitates fitting complicated multiple spectral peaks for quantitative analysis.

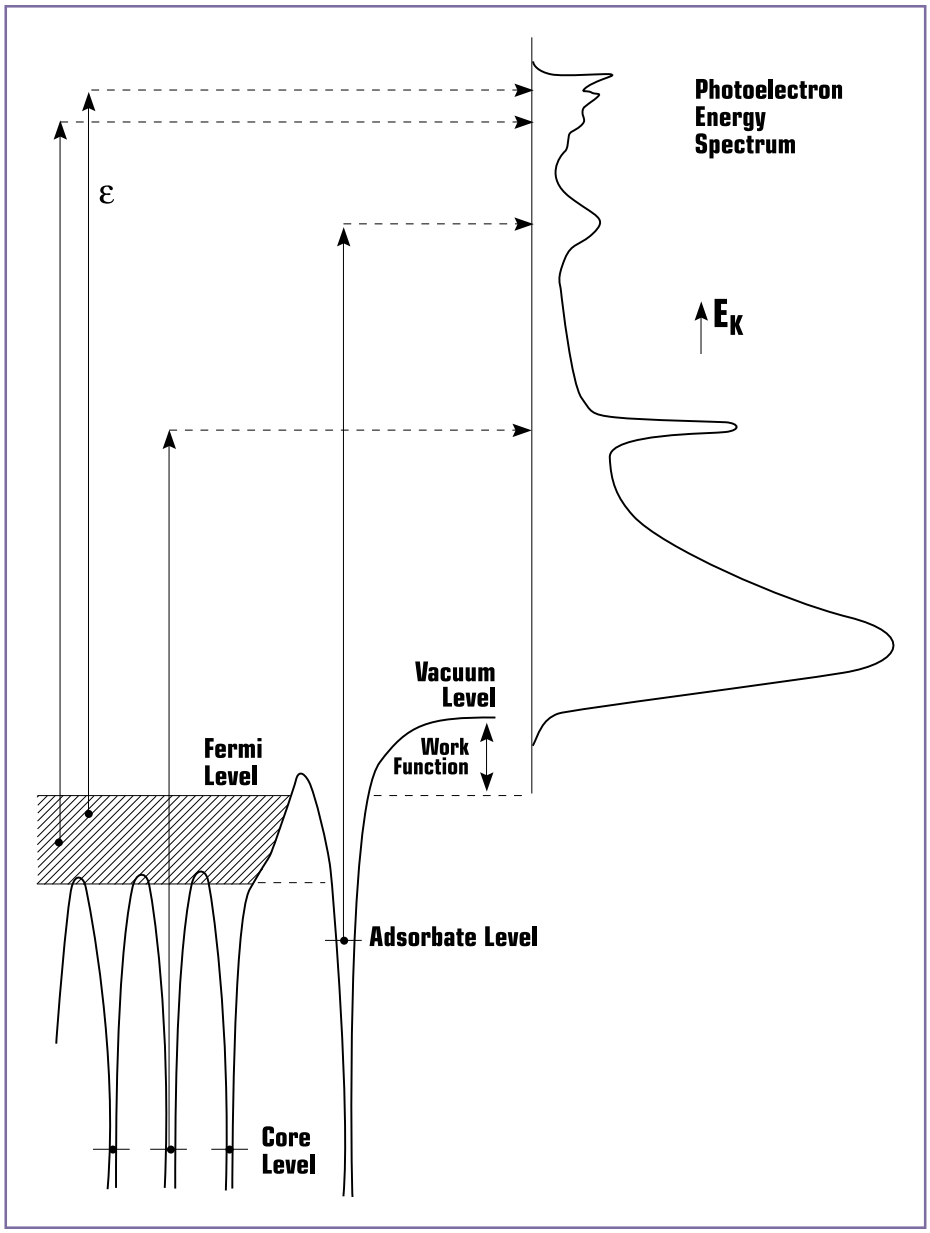
The photon-energy tunability of synchrotron radiation provides additional benefits because the cross section for x-ray absorption is much higher when the photon energy is near an absorption edge, resulting in a significantly enhanced photoemission signal that can make marginal experiments practical. For example, the cross section for absorption by carbon 1s electrons is about 100 times larger for excitation just above the carbon K edge with 320-eV photons than with aluminum-Kα radiation.

Still another aspect of tunability is the ability to adjust the surface sensitivity of the experiment. X rays can penetrate deeply into a solid before being absorbed, so that a photoelectron has some distance to travel in order to escape from the surface. The distance the photoelectron can travel with no energy losses due to scattering depends on its kinetic energy. The minimum escape depth is a few angstroms for kinetic energies near 50 eV and is deeper at both higher and lower energies. Experimenters using synchrotron radiation can therefore tune the x-ray photon energy to produce photoelectrons from very near the surface or farther down in the bulk, a feature that turns core-level photoelectron spectroscopy into a technique with variable depth sensitivity.

These features combine to enhance studies of surface structure by means of the angular distribution of the intensity of a peak in the photoelectron spectrum. This distribution constitutes a diffraction pattern of the atoms surrounding the emitter (photoelectron diffraction). Selecting the emission energy isolates a particular atomic species, and binding-energy shifts due to chemical bonding or lattice location can further specify the emitting atom. The angular-distribution data then contains an immense amount of information about the crystal structure close to the surface.



Some photoelectrons lose kinetic energy on their way to the surface because of inelastic collisions. The mean free path between inelastic scattering events for a photoelectron depends on its kinetic energy in much the same way for elements and inorganic compounds. This functional dependence combined with tunable photon energies provides a way to control the depth below the surface from which electrons contributing to the XPS spectrum come. [Adapted from C.R. Brundle, C.A. Evans, Jr., and S. Wilson, eds., *Encyclopedia of Materials Characterization*, Butterworth-Heinemann, Stoneham, MA, 1992, p. 293.]



This schematic diagram shows that peaks in the photoelectron spectrum of a metallic solid correspond to the quantum states from which the electrons are emitted. The diagram shows core states in atomic potential-energy wells, the portion of the valence band occupied by electrons (i.e., below the Fermi level), and a state in a potential-energy well associated with the surface. There is also a large secondary-electron tail due to inelastically scattered electrons. [Adapted from N.V. Smith and F.J. Himpsel, "Photoelectron Spectroscopy," in E.-E. Koch, ed., *Handbook of Synchrotron Radiation*, Vol. 1B, North-Holland Publishing Company, Amsterdam, 1983, p. 908.]

How XPS Provides Chemical Specificity

In x-ray photoelectron spectroscopy (XPS) of solids, an electron excited from a core level by x-ray absorption makes a transition to an unoccupied state at energies above the ionization threshold. From there, the electron can migrate to the surface and escape into the vacuum. If the electron suffers no collisions on the way, its kinetic energy E_K on escaping is related to the binding energy E_B (minimum energy to escape) of the core level and the photon energy ε

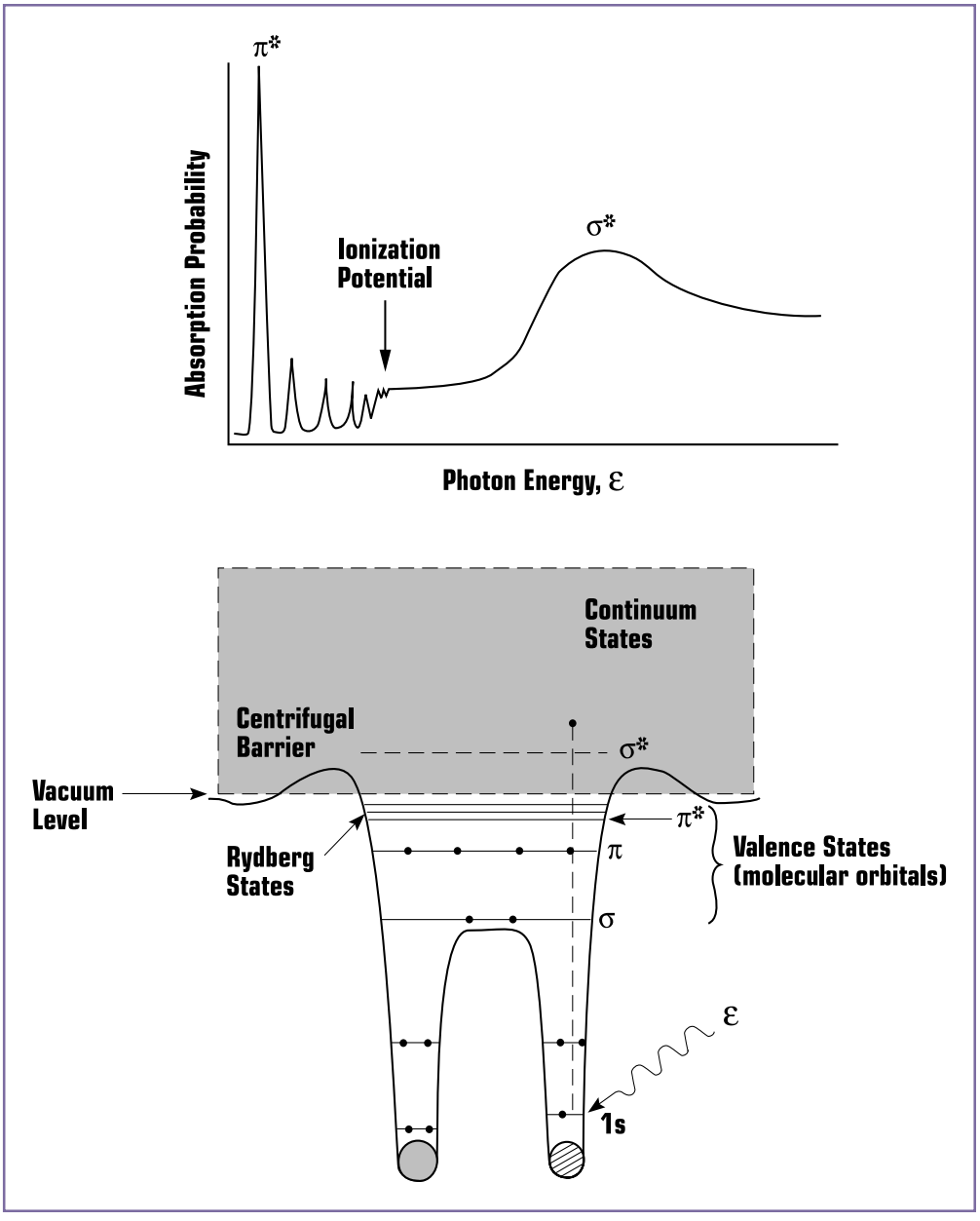
$$E_K = \varepsilon - E_B.$$

This relation shows that, for a fixed photon energy, the spectrum of photoelectron kinetic energies measured with an electron-energy analyzer reflects the distribution of occupied core states. Several other effects also contribute to the spectrum—Auger-electron emission occurs at a fixed energy independent of the photon energy when an electron from a higher lying state fills the core hole; satellite photoelectron peaks appear at lower kinetic energy than the main peak when excitation of a second electron to an unoccupied state below the ionization threshold drains away some of the incident photon energy; and the large background at low kinetic energies is due to multiply scattered (secondary) electrons.

To a first approximation, the binding energy of the core level is independent of the environment, so it identifies the atomic species. However, there are small "chemical shifts" in the binding energy that depend on the local chemical bonding. For example, at the interface between silicon and silicon dioxide, the binding energy of the important silicon $2p_{3/2}$ core level varies approximately linearly with the number of oxygen atoms bound to silicon, shifting about 1 eV per oxygen atom from the 99.2-eV binding energy for pure silicon. It is these energy shifts that provide chemical-state information.

X-Ray Absorption

A Chemical Fingerprint From Near-Edge Spectra



This schematic diagram of a diatomic molecule shows that peaks observed in the near-edge x-ray absorption spectrum correspond to unoccupied valence states to which core electrons are excited. These valence states may be modified by crystal-field effects in the solid state and by their participation in chemical bonding. The NEXAFS spectrum is thus a striking signature of the structural environment and chemical state of the absorbing atom. [Adapted from J. Stöhr, *NEXAFS Spectroscopy*, Springer-Verlag, Berlin, 1992, p. 85.]

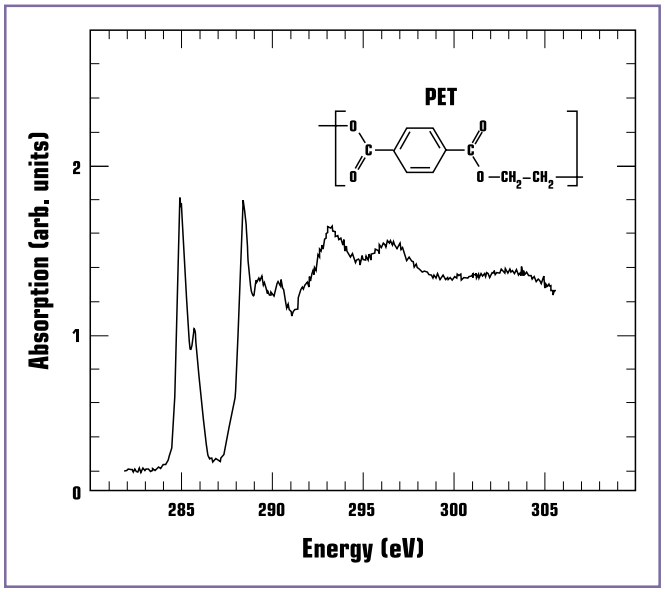
Chemical information can be derived from the distinctive spectral features of the absorption cross section in the region of an x-ray absorption edge. This technique is known as near-edge x-ray absorption fine-structure spectroscopy (NEXAFS, but sometimes called XANES for x-ray absorption near-edge spectroscopy).

If the excitation energy is near an absorption edge, an electron excited from a core level by x-ray absorption can make a transition to an unoccupied valence state at energies near the ionization threshold. If the lifetime of the core hole is long, then the intrinsic energy width of a core level is narrow, and the absorption spectrum (peak position, shape, and intensity) maps the energy distribution of unoccupied states to which the electron is excited. As it happens, most of the sharpest core levels of interest for NEXAFS spectroscopy have binding energies well within the VUV and soft x-ray range of the ALS undulators.

The density of states is a signature of the chemical bonding and surrounding crystal structure of the absorbing atom. For example, in organic compounds, there may be a distinctive spectrum due to strong carbon absorption into orbitals determined by the chemical bonds in which the carbon atoms are participating. In transition metals, local symmetry (e.g., octahedral or tetrahedral) and the strength of the crystal field strongly affect the absorption. The near-edge absorption therefore serves as a chemical and structural fingerprint.

Measurement of the transmitted intensity is the most obvious way to do absorption spectroscopy, if the sample is thin enough. The penetration depth for soft x-ray photons is usually in the range 0.1 to 1 μm, so transmission measurements give information about the interior of materials. For thicker samples, one can monitor the emission of either electrons or fluorescence photons from a solid surface, both of which are a measure of absorption probability.

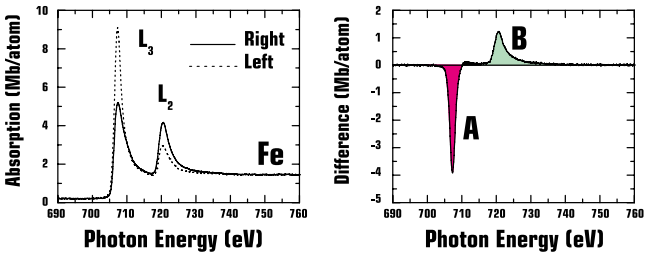
A simple photocurrent measurement of the total yield of electrons (sum of photoelectrons, Auger electrons, and scattered secondary electrons of a few electron volts) monitors electron emission, whereas an energy-resolving x-ray detector directly measures the fluorescence. Electron emission and fluorescence have different sampling depths, typically 5 nm for total electron yield and up to 1 μm for fluorescence. One can enhance the surface sensitivity by biasing the sample to eliminate the low-energy electrons (partial electron yield) or by monitoring the Auger electrons directly with an electron-energy analyzer.



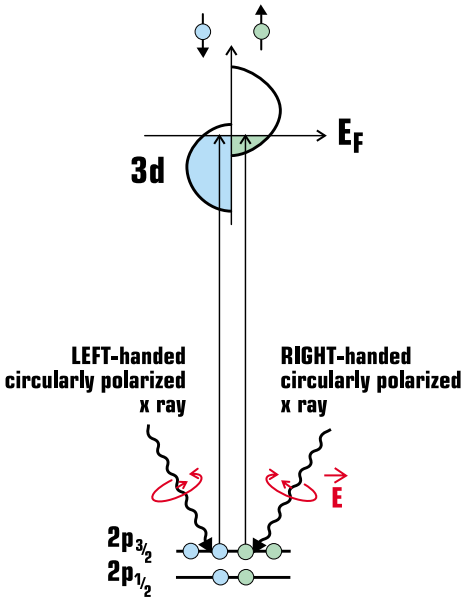
NEXAFS spectra of organic molecules at the carbon K edge show peaks characteristic of the molecular orbitals bonding the carbon atoms. Absorption at different sites with correspondingly different bonding yields spectral signatures of the various molecules. The spectrum of polyethylene-terephthalate (PET) is shown here. As a database of characteristic spectra builds up, the NEXAFS technique is becoming a very important tool for polymer analysis. [Data taken at the National Synchrotron Light Source. Courtesy of H. Ade, North Carolina State University.]

Polarization

Enhancing The Capability of X-Ray Spectroscopy



- **Orbital Magnetic Moment**
  $m_{orb} \propto (A+B)$
- **Spin Magnetic Moment**
  $m_{spin} + m_D \propto (A-2B)$

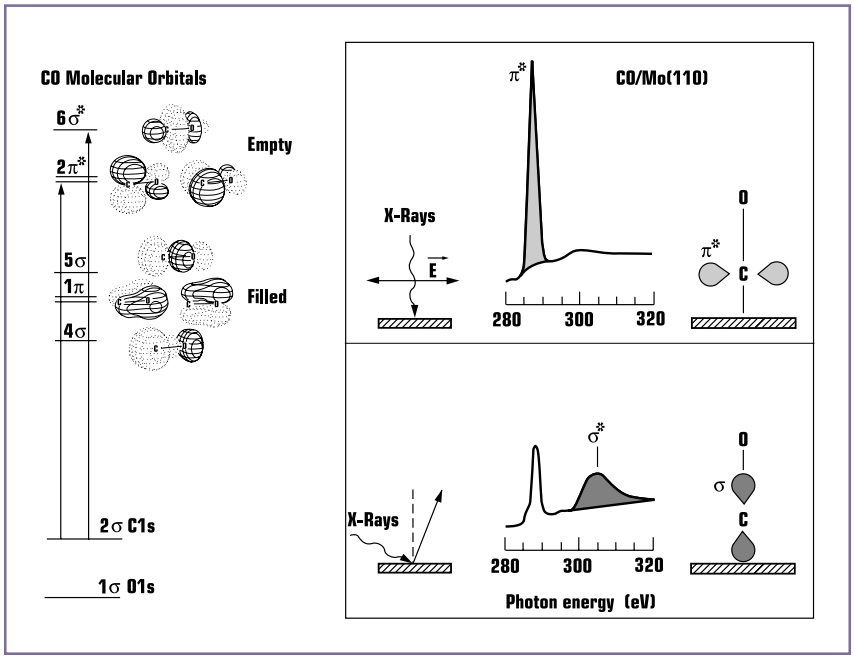


In an elemental ferromagnet, the differential absorption of left and right circularly polarized x rays propagating parallel to an applied magnetic field results from an imbalance in the spin occupancy of the partially occupied valence band and from quantum-mechanical selection rules that apply to the absorption transitions. From the dichroism, it is possible to extract the separate spin and orbital contributions to the total local magnetic moment. The spin density m_D in the figure is an orientation-dependent term that vanishes in isotropic materials and certain experimental geometries.

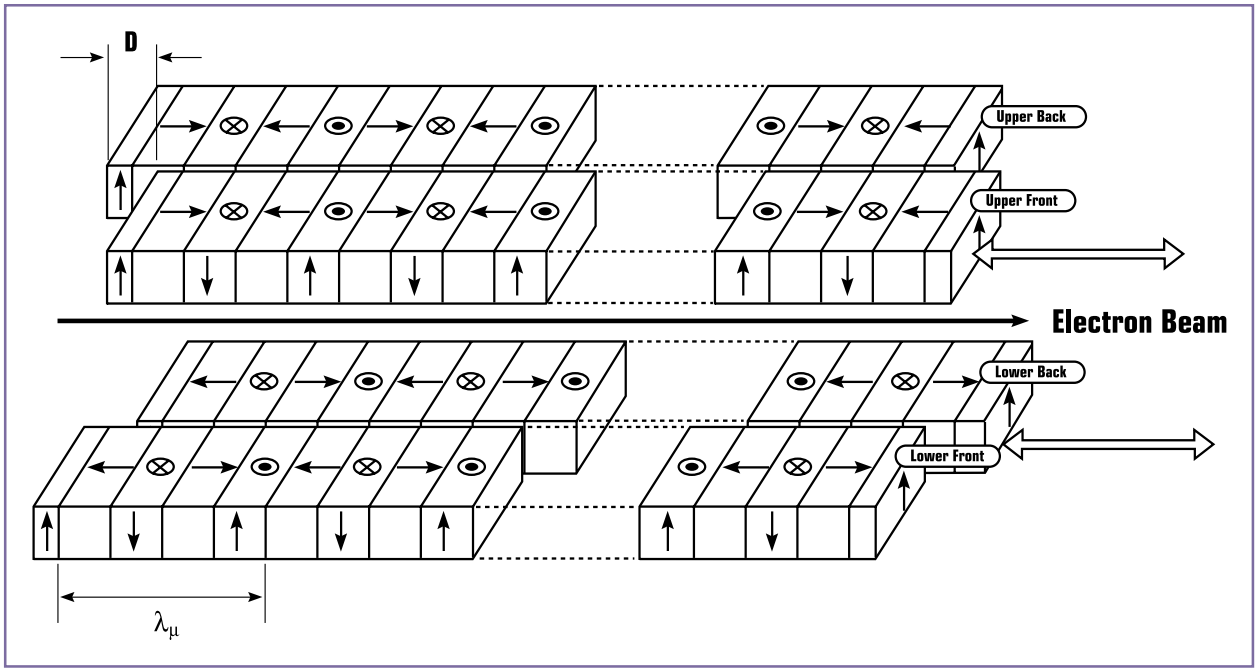
Synchrotron radiation is naturally linearly polarized in the horizontal plane of the electron orbit. Since quantum-mechanical transition probabilities depend on the relative orientation of the electric-field vector of the exciting radiation and the anisotropic wave functions of the quantum states involved, polarization provides symmetry selectivity that is lacking in processes excited by unpolarized light. For example, in the case of core-level excitation of molecular species, the absorption is proportional to the cosine squared of the angle between the electric-field vector and the orbital to which the electron is excited. Scientists have used this relation with NEXAFS spectroscopy to determine the orientation of molecules chemisorbed on a solid surface and the orientation of polymer fibers in complex structures.

The use of circularly polarized synchrotron radiation is growing in materials research to study spin-dependent magnetic phenomena. Experimental techniques at the ALS that make use of circularly polarized x rays are often based on NEXAFS spectroscopy. In x-ray magnetic circular dichroism spectroscopy (XMCD), one measures the absorption spectra in a magnetized sample using left- and right-circularly polarized x rays. For example, in the elemental ferromagnets iron, nickel, and cobalt, the valence band splits with the majority electrons (those whose spin magnetic moment is parallel to the applied magnetic field) lying lower in energy relative to the minority electrons with antiparallel spin, so that the unoccupied states in the valence band are primarily of minority spin. Together with the angular-momentum selection rules appropriate for circularly polarized x rays, this means that the absorption cross section for excitation of electrons from core levels into the valence band depends on the direction of the field and the helicity of the photons.

Analysis of the difference between the absorption spectra for reversed helicities or field orientations (the dichroism) yields element-specific magnetic properties, such as the size and orientation of magnetic moments. If a given element exists in more than one oxidation state or site symmetry, the spin orientations of these individual species may be distinguishable. There is also the possibility of extracting the separate spin and orbital contributions to the total local magnetic moment. With the use of polarizing optics and linearly polarized synchrotron radiation, similar information about magnetic materials comes from measuring the rotation of the plane of polarization of soft x rays transmitted through (Faraday rotation) or reflected from (Kerr rotation) samples in magnetic fields.



Linearly polarized synchrotron radiation is a useful tool for determining the orientation of adsorbed molecules on a surface. Here, the vertical orientation of a carbon monoxide molecule on a molybdenum surface is verified by the strong absorption due to the π^* orbital when the electric vector is parallel to the surface and by the absorption due to the σ^* orbital when the electric vector is nearly normal to the surface. [Adapted from J. Stöhr, *NEXAFS Spectroscopy*, Springer-Verlag, Berlin, 1992, p. 171.]



Schematic diagram of an elliptically polarizing permanent-magnet undulator shows how mechanically adjusting the relative positions of the magnet rows controls the polarization. The arrows indicate the direction of magnetization in each block, so that four blocks constitute one period λ_u. The displacement D between the upper front and lower rear rows relative to the upper rear and lower front rows gives a row phase = D/λ_u. The degree of polarization depends on the phase shift.

How To Obtain Polarized Synchrotron Radiation

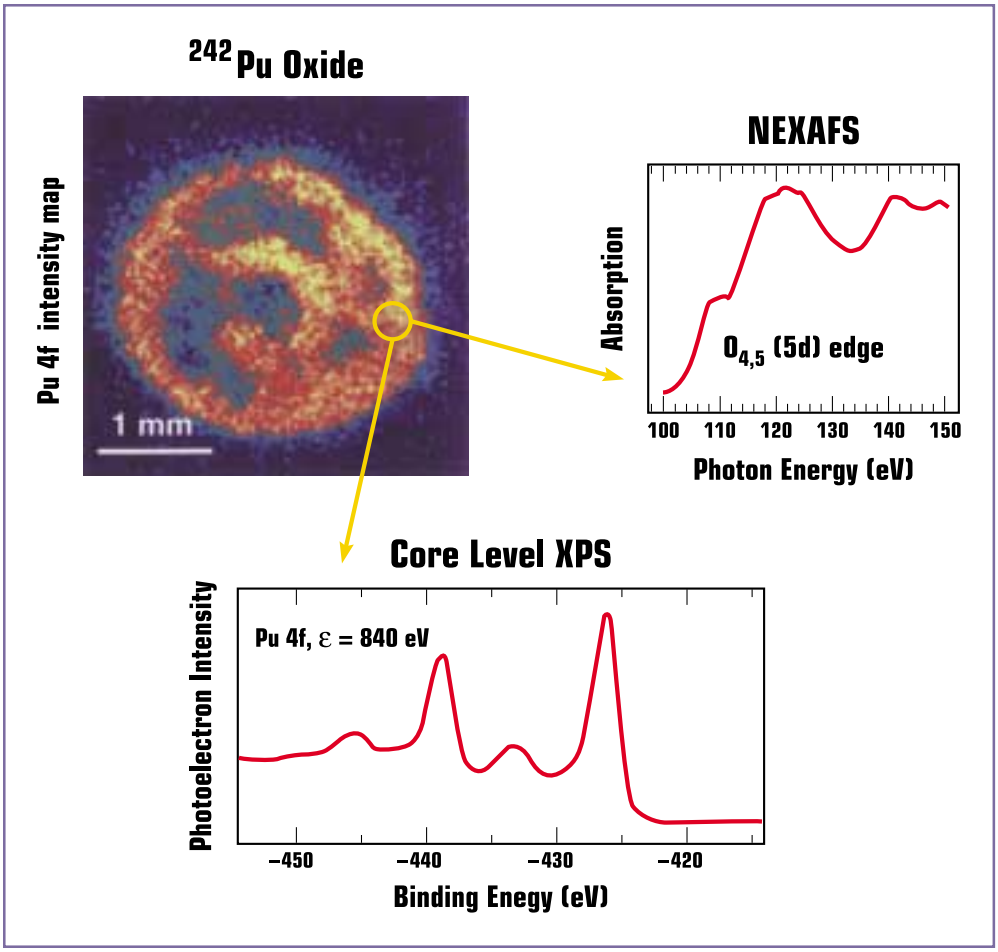
Bend-magnet radiation out of the horizontal plane of the electron orbit is elliptically polarized; that is, the electric-field vector consists of horizontal and vertical components that are 90° out of phase. The relative amplitudes of the horizontal and vertical components depend on the observation angle. In the horizontal plane, the vertical component is zero, so that bend-magnet radiation is linearly polarized with the electric-field vector in the plane. As the observation angle above or below the plane increases, the amplitude of the vertical field component approaches that of the horizontal component, but the intensity of the radiation decreases. In practice, one chooses an observation angle that maximizes the product of the square root of the flux and the degree of circular polarization. At this maximum, the degree of circular polarization is typically about 70 percent. The sense of the polarization can be reversed by changing the viewing angle from above to below the plane and vice-versa by means of movable apertures.

To increase the flux of circularly polarized photons over that available from a bend magnet, the ALS is developing an elliptically polarizing undulator (EPU). In the usual linear undulator, the circular components of the radiation from successive poles reverse direction, so the net effect is linearly polarized radiation. In the EPU, four identical rows of permanent-magnet blocks are arranged in two pairs above and below the plane of the electron orbit. If the upper front and lower rear rows (or upper rear and lower front rows) are moved longitudinally in the same direction by the correct distance with respect to the fixed rows, a helical magnetic field—and hence electron trajectory—is created. Moving the rows in the opposite direction creates a field of the opposite helicity. This motion is termed a row phase shift. Depending on the phase shift, the undulator radiation is polarized linearly in the vertical direction, right or left circularly polarized, or linearly polarized in the horizontal plane as in a conventional undulator.

Spectromicroscopy

A New Combination

Putting X-Ray Microscopy & Spectroscopy Together

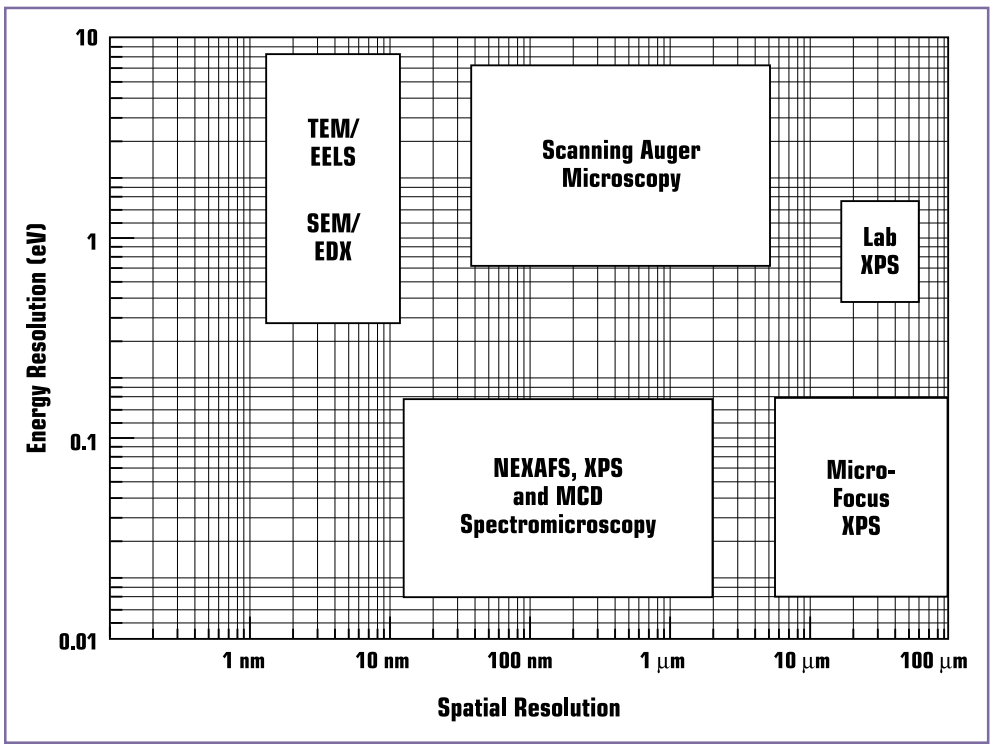


XPS imaging of a very small amount of plutonium oxide (4 μg) demonstrates that spectromicroscopy can analyze otherwise hazardous materials without special environmental chambers. Here, imaging is accomplished using an x-ray spot 50 μm in diameter by moving the substrate in the fixed x-ray focal spot. XPS spectra of the plutonium 4f electrons used in the imaging show oxide chemical shifts. NEXAFS spectra also show the plutonium 5d edge. [Data taken at the ALS. Courtesy of D.K. Shuh, Lawrence Berkeley National Laboratory, B.P. Tonner, University of Wisconsin–Milwaukee, S.D. Kevan, University of Oregon, and T. Warwick, ALS.]

Owing to the dramatically enhanced brightness of the newest synchrotron radiation sources, such as the ALS, x-ray spectroscopy and microscopy can now be combined. Spectromicroscopy provides all the power of x-ray spectroscopic techniques (e.g., chemical sensitivity, variable excitation energy, minimum radiation damage, and variable polarization) with spatial resolution ranging from around one micrometer to as fine as a few nanometers. With soft x-ray spectromicroscopy at these small length scales, scientists can address a very wide range of materials microcharacterization problems involving complex microstructured systems and surfaces.

For example, the semiconductor industry now fabricates devices with features a fraction of a micrometer in size, and the ability of x-ray spectromicroscopy to perform chemical analysis at this spatial scale is attracting attention. Similarly, materials analysts can use these techniques to examine the chemical, structural, and magnetic properties of small-scale structures in magnetic recording media and devices. And they can investigate composite materials with phases of the order of 1 μm in size, even when radiation-sensitive, organic components are involved.

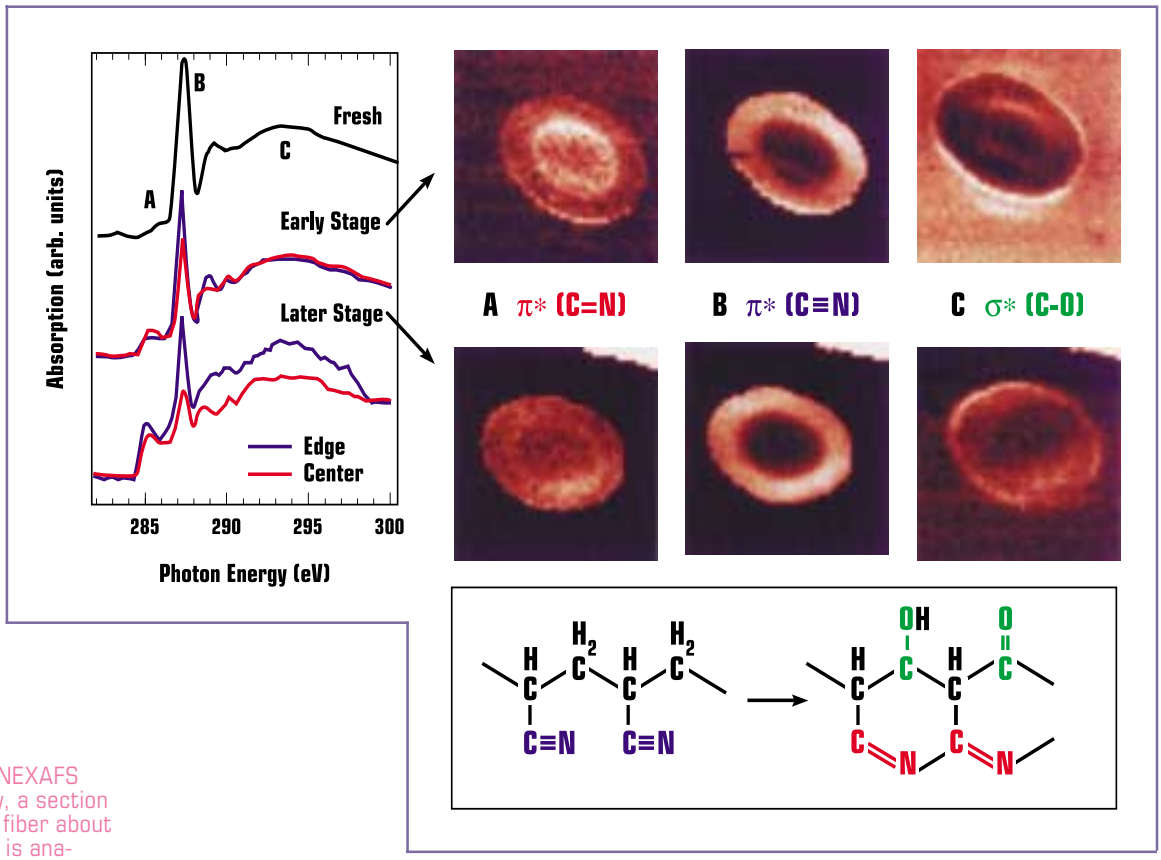
At the ALS, near-edge x-ray absorption fine-structure spectroscopy (NEXAFS) and x-ray photoelectron spectroscopy (XPS) are being developed as spectromicroscopies in the soft x-ray region of the spectrum for materials microcharacterization. Although soft x-ray spectromicroscopy is in its infancy, microscopes at the ALS have already demonstrated 100-nm resolution in the early stages of a continuing program aimed at the development of several instruments and techniques residing on multiple beamlines with a spectrum of capabilities.



This "phase space" diagram showing the spectral and spatial resolutions achievable with electron-beam and x-ray analytical techniques highlights the ability of soft x-ray spectromicroscopy to obtain spatially resolved chemical information.

Scanning Microscopy

Versatility & High Spatial Resolution



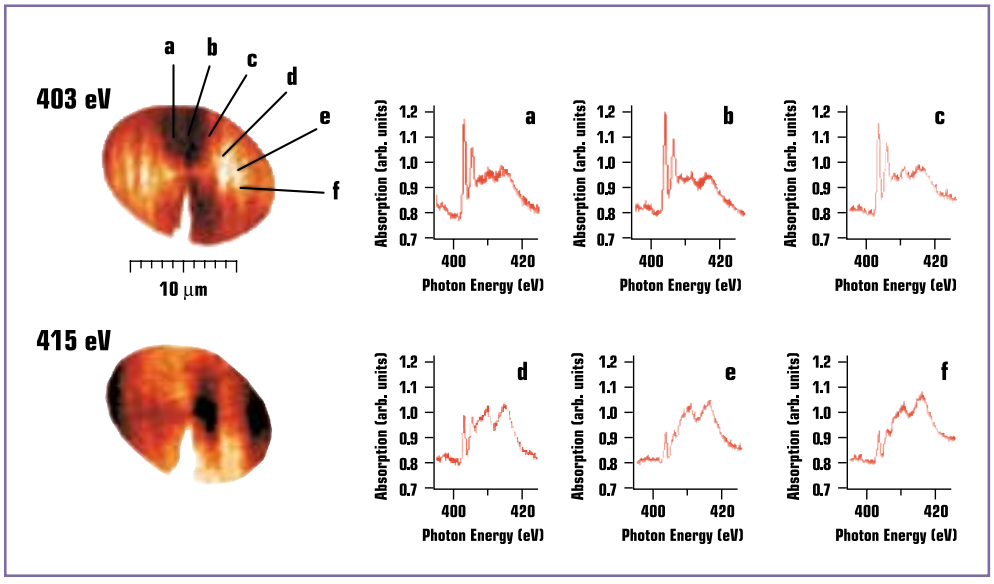
In this example of NEXAFS spectromicroscopy, a section of polyacrylonitrile fiber about 10 μm in diameter is analyzed by x-ray absorption near the carbon K edge. Images taken after a heat-treatment process to enhance the fire-retarding capabilities of this material show contrast due to the distribution of carbon-nitrogen and carbon-oxygen bonds (peaks A, B, and C). Annealing in air causes triple bonds (C≡N), which exist uniformly throughout the homogeneous PAN fiber before treatment, to become depleted in the interior, giving rise to a rim structure. [From B.P. Tonner, et al., *J. Elect. Spectros. Rel. Phenom.*, **75**, 309–332 (1995).]

In a scanning x-ray microscope, optics focus the x-ray beam, and a detector senses photons or electrons as the x-ray spot moves across the sample surface (or as the sample rasters through a fixed x-ray spot). In either case, the image builds up pixel by pixel during a scan that typically lasts about a minute at the ALS. The mechanical design of the sample stage fixes the spatial scanning range. For example, the scan range of piezo drives limits the maximum scanned areas to about 100 μm × 100 μm. However, the image field can be positioned anywhere within a much larger area. The primary advantages of scanning microscopy are its guaranteed spatial resolution and the versatility resulting from the variety of useful signals.

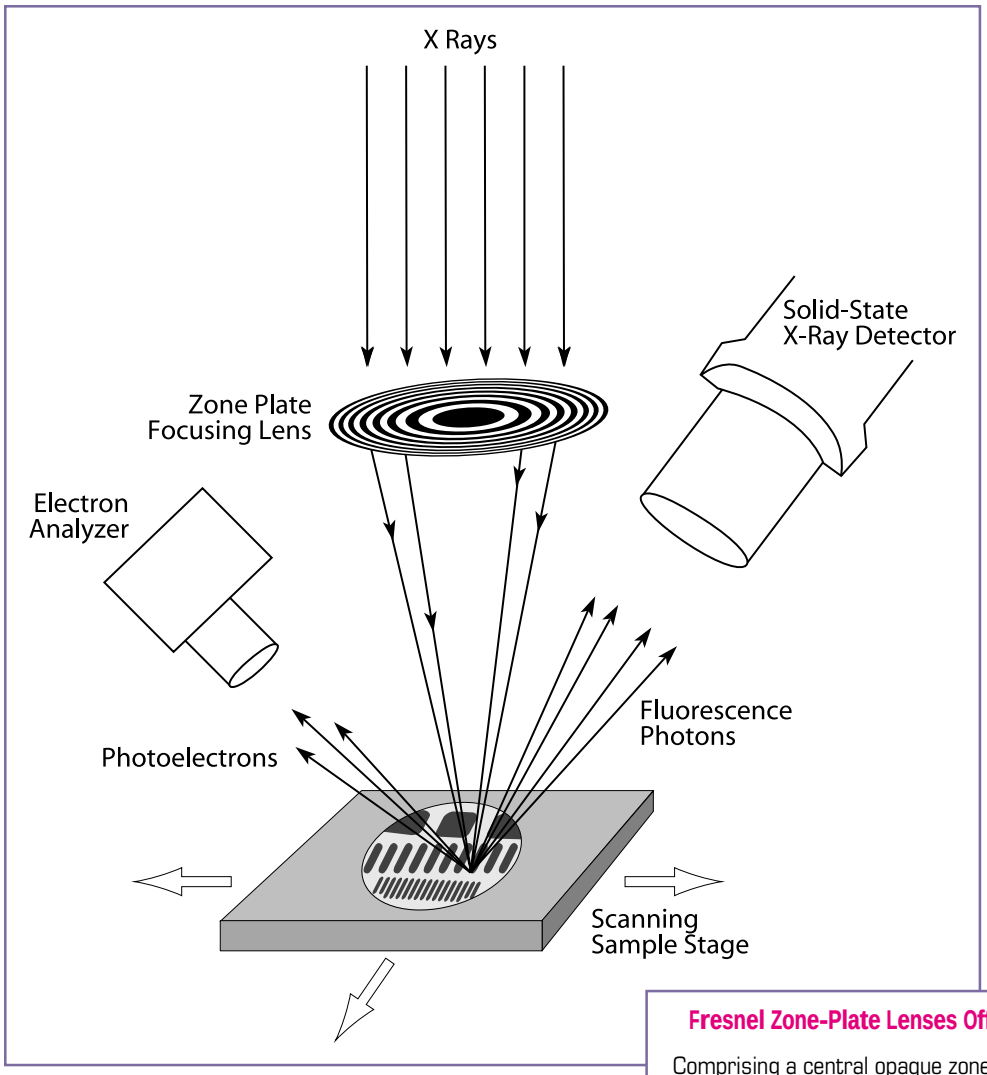
The size of the focused spot determines the spatial resolution of the instrument. Spot sizes of the order of 100 nm are routine. Achieving such a small spot depends on special diffraction-limited x-ray optical systems. In particular, scanning microscopes have been implemented using both Fresnel zone-plate lens systems, whose focusing action is like that of a circular diffraction gratings, and reflective focusing from spherical mirrors in the Schwarzschild configuration. Because of the low emittance of the ALS storage ring, undulator radiation comes from a nearly diffraction-limited point source, resulting in acceptance of a large fraction of the x rays by the diffraction-limited optics. The low-emittance also makes it practical to obtain significant coherent flux from bend magnets by means of apertures.

Images can be obtained from any signal as a function of position on the sample surface. This signal could be the count rate of transmitted photons, the total or partial yield of photoelectrons, the yield from a particular photoelectron peak in an XPS spectrum , or the yield of fluorescence photons at peaks in a NEXAFS spectrum. Each type of signal will give different elemental, chemical, magnetic, or surface/bulk information. Scanning spectromicroscopy has been applied to many materials, including biological systems, organic polymer blends, magnetic media, and semiconductors.

Scanning microscopy is a serial acquisition method with inherent speed restrictions. A typical scenario might consist of a conventional spectroscopic examination to locate characteristic spectral features, followed by a mapping of the distribution of those features at coarse resolution, a finer mapping of areas of interest, and a detailed spectroscopic examination of the most important features.



Transmission NEXAFS images of a sectioned Kevlar fiber with radially oriented hydrogen-bonded sheets between adjacent polymer chains shows the sensitivity to the relative orientation between the x-ray polarization plane and the π^* molecular orbitals of nitrogen in the molecule. Nitrogen 1s electrons can be excited into these orbitals only when the orbitals are oriented parallel to a component of the electric field of the linearly polarized light. The magnitude of the parallel component rises and falls around the fiber, giving rise to the distinctive image. [Data taken at the ALS. Courtesy of H. Ade, North Carolina State University, and B.P. Tonner, University of Wisconsin–Milwaukee.]



Fresnel zone plates are diffrac-tive devices that can focus the x-ray source to a spot size approximately equal to the width of the outermost zone. As a result of their proven record of achieving high spatial resolution and the flexibility of multiple de-tection methods, they are widely used in x-ray micro-scopes at several synchro-tron radiation facilities.

Fresnel Zone-Plate Lenses Offer High Resolution & Tunability

Comprising a central opaque zone surrounded by alternating trans-parent and opaque zones of increasing radius and decreasing width, zone plates are circular diffraction gratings with a diffraction angle increasing linearly with radius, so that they focus incident radiation to a small spot. For a lens of diameter D with a total of N opaque and transparent zones and a numerical aperture NA, the focal length f is given by

$$f = D/2(NA) = 4N(\Delta r)^2/\lambda,$$

where Δr is the width of the outermost opaque zone. Lenses with Δr as small as 30 nm and N equal to several hundred have been made. The depth of focus Δf (the longitudinal distance occupied by the fo-cused spot) is also wavelength dependent

$$\Delta f = 1.22\lambda/(NA)^2 = 4.88(\Delta r)^2/\lambda,$$

The finest spatial resolution δ of a zone-plate with monochromatic and spatially coherent illumination and perfect zone placement is

$$\delta = 0.61\lambda/NA = 1.22\Delta r.$$

It is anticipated that, with improvements to zone-plate fabrication technology based on electron-beam lithography, resolutions of 25 nm or less will ultimately be possible. Although zone plates can focus any wavelength, the amount of light they collect varies as λ^2, so that signal intensity is highest at long wavelengths and low photon ener-gies. Phase-contrast zone plates are also available with transmissive zones designed to give the appropriate phase shift at a specific wavelength for higher efficiency.

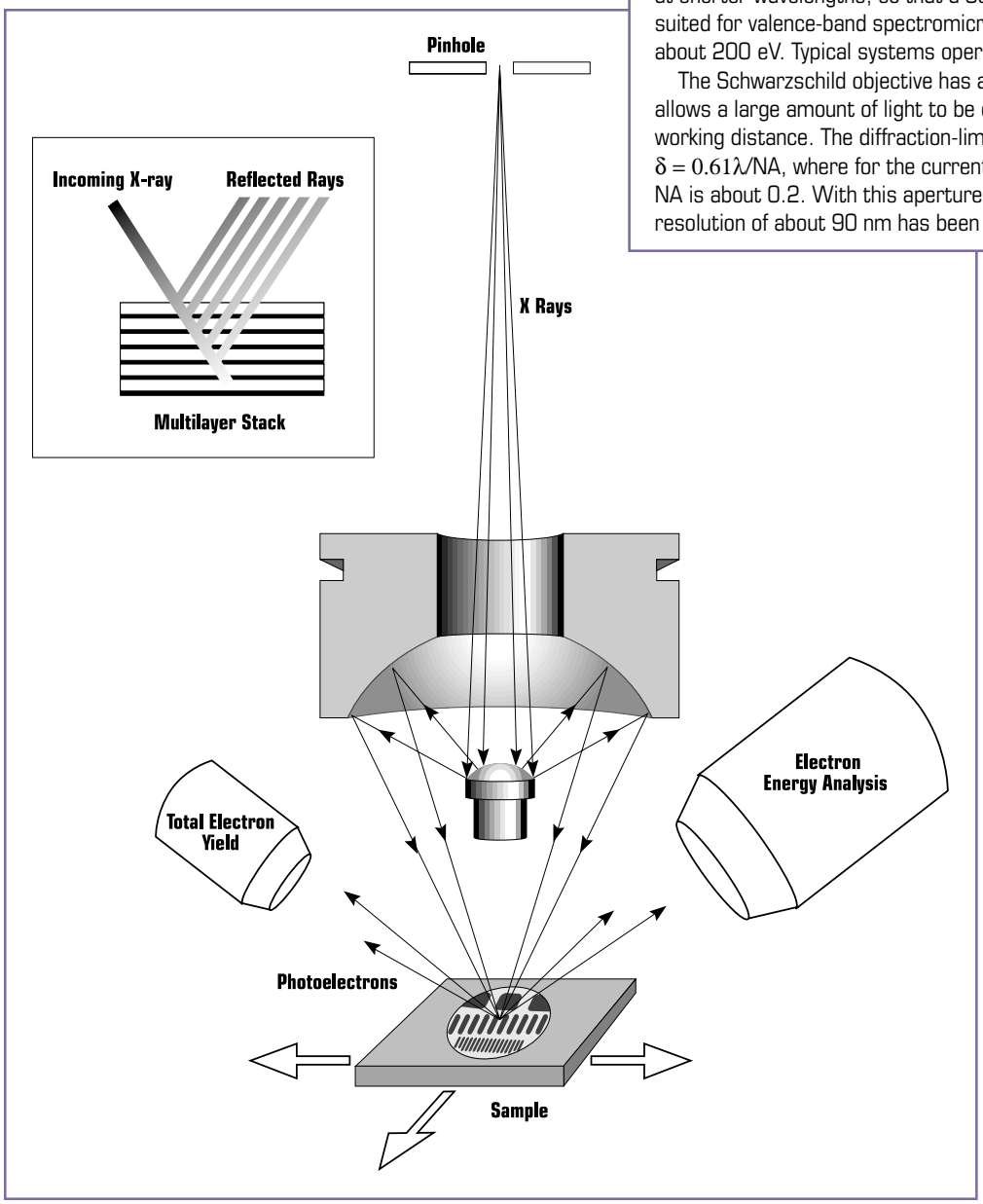
Schwarzschild Objectives—Efficient At Lower Photon Energies

The Schwarzschild objective comprises two concentric spherical mirrors, the first presenting a convex face to the incident radiation and the second a concave face, so that the net effect is to focus the light to a small spot. Since the normal-incidence reflectivity of mirror materials at soft x-ray wavelengths is small, recourse is made to multilayer coatings. These comprise alternate layers of x-ray transmitting and absorbing materials, resulting in a coherent addition of the reflectivities at each interface. The increased reflectivity, which may be as high as 60% for each mirror, comes at the expense of a limited bandwidth of a few percent around a central wavelength given by a Bragg equation for reflection from the layers

$$\lambda = 2d \sin\theta,$$

where d is the layer spacing and θ is close to 90°. The limited bandwidth for a coating with N layers ($\Delta\lambda/\lambda \approx 1/N$) means that multiple objectives are needed for work over a range or wavelengths. Multilayer coatings can be designed with a variety of materials and spacings for a wide range of wavelengths, but the reflectivity decreases at shorter wavelengths, so that a Schwarzschild instrument is best suited for valence-band spectromicroscopy up to a photon energy of about 200 eV. Typical systems operate at 95 or 130 eV.

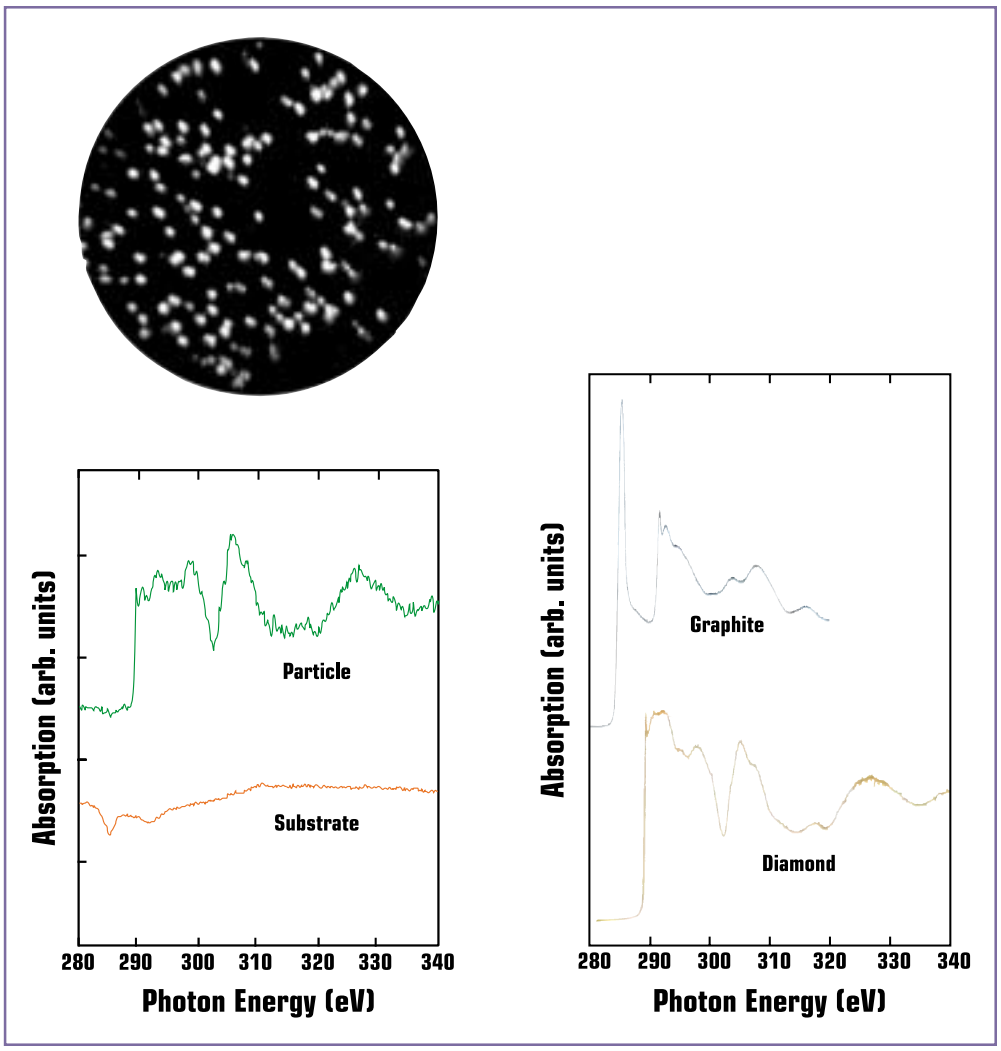
The Schwarzschild objective has a large numerical aperture, which allows a large amount of light to be collected and results in a long working distance. The diffraction-limited image size δ is given by $\delta = 0.61\lambda/NA$, where for the current-generation x-ray microscopes, NA is about 0.2. With this aperture and a wavelength of 13 nm, a resolution of about 90 nm has been experimentally measured.



Mirrors in the Schwarzschild geometry can focus the x-ray source to a submicron spot. Multilayer interference coatings enhance the normal-incidence reflectivity at soft x-ray energies. The long working distance of this objective lens is conven-ient for XPS scanning spectromicroscopy.

Full-Field Imaging

Spectromicroscopy In Real Time



Thin, hard coatings based on sp^3 bonding in carbon have many uses. Here, micron-sized particles of chemical vapor-deposited diamond on a silicon substrate are easily seen in the X-PEEM image taken at 292 eV near the carbon K edge. Moreover, NEXAFS spectra from the particles reproduce the structure seen in spectra for diamond. In particular, they do not have the characteristic resonance below the K edge associated with sp^2 bonding in graphite and other carbon forms but absent in the purely sp^3 bonding of diamond. [Data taken at the Stanford Synchrotron Radiation Laboratory. Courtesy of A. Cossy, S. Anders, and A. Garcia, ALS. [Reference spectra from J.F. Morar et al., *Phys. Rev. Lett.* **54**, 1960 (1985) and L.J. Terminello et al., *Chem. Phys. Lett.* **182**, 491 (1991).]

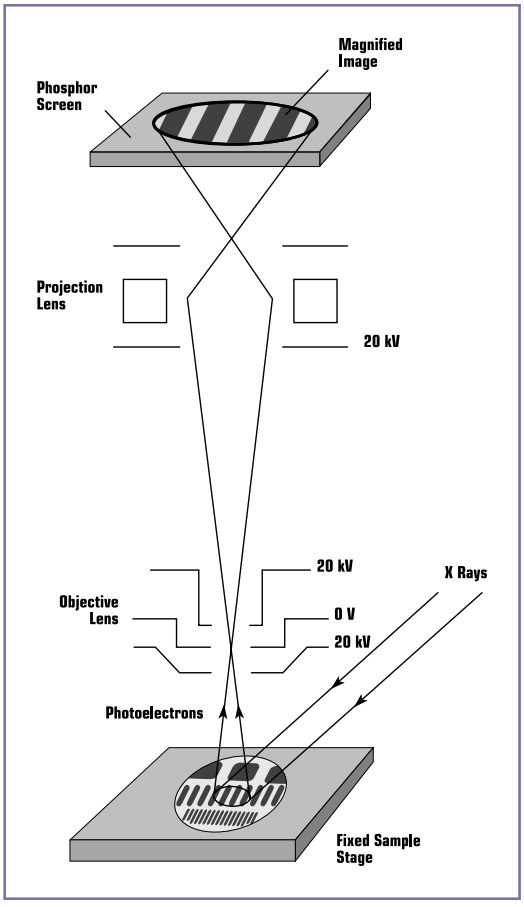
For materials microcharacterization, imaging spectromicroscopy is based on electron optical techniques. In this approach, photon optics condense the beam to the desired field of view, and an electron microscope images the sample surface using photoemitted electrons. The first photoelectron microscopes (UV-PEEM) used an ultraviolet source and imaged the electrons with a series of electrostatic lenses, obtaining both topographical and work-function contrast.

Using higher energy photons from a synchrotron light source to excite core levels, it is possible to obtain chemical-state information with an X-PEEM. The yield of electrons over the entire illuminated area is imaged at high magnification onto an image-intensified camera. By recording frames sequentially with an incrementally increasing photon energy, spatially resolved electron yield, and hence absorption, can be measured in the near-edge region, thereby producing a NEXAFS spectrum for each image point. In principle, the addition of an electron-energy analyzer allows XPS spectromicroscopy, as well.

At present the chromatic aberration resulting from the wide energy distribution of the imaged electrons limits the spatial resolution of the X-PEEM. For a microscope with a 10-kV accelerating voltage, a resolution around 300 nm has been measured in a field of view 50 μm in diameter. The field size itself is dependent on the magnification of the microscope, since the size of the detector is fixed. Instruments now under design, in which an electron-mirror removes the chromatic aberration, ultimately should achieve 5-nm resolution.

For full-field imaging microscopy, the goal is to get the highest possible flux in the field of view, hence the beam need only be demagnified enough to illuminate the area to be imaged. Since there is no focusing to a small spot, the illuminating radiation need not be diffraction limited, so both undulators and bend magnets at the ALS can serve as high-quality sources.

Moreover, the full-field photoelectron microscope is inherently fast, and the evolution of surface processes, such as the propagation of surface-reaction fronts at low coverage, can be measured at video rates. As compared to a scanning instrument, however, the microscope is less versatile, owing to the exclusive use of electron detection, and suffers from the added complexity of the electron imaging system and the need for a high-vacuum environment.



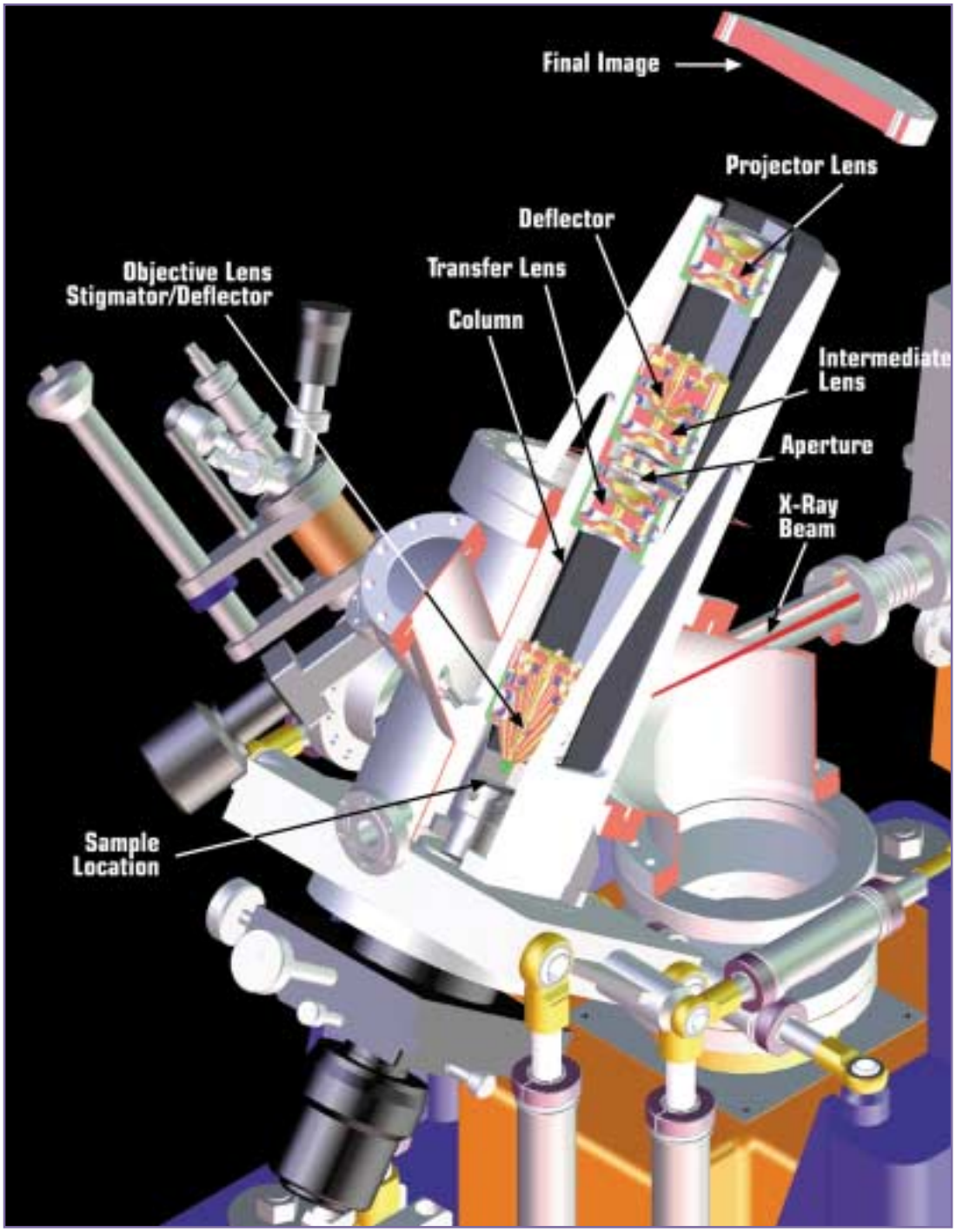
Schematic of a simple two-stage X-PEEM showing electrostatic objective and projective lenses that create a magnified image of the photoemitted electrons from the sample surface. A pinhole in the back focal plane of the objective lens limits the angular acceptance to reduce spherical aberration.

Advanced Electron Lenses Reduce Chromatic Aberration

New concepts in electron optics are making possible an advanced x-ray photoemission electron microscope (X-PEEM) for chemical-state imaging that has very high spatial resolution and greatly improved transmission, as compared to previous instruments. One breakthrough is the invention of electrostatic hyperbolic-field mirrors for the correction of electron-optical aberrations, and chromatic aberrations in particular.* The use of aberration-correcting mirrors has the potential for pushing down the spatial-resolution limit of the X-PEEM to 5 nm and possibly even to 2 nm.

In the first of an evolving series of microscopes leading to incorporation of this concept at the ALS, photoemitted electrons will be imaged by means of an optical system consisting of a three-element high-voltage objective lens, a transfer lens, intermediate lens, and deflector lens. With this system, electrons will be imaged at high magnification onto an image intensifier and CCD camera. Next, coupling lenses, a chromatic-aberration correcting electron mirror, and a beam separator will be added. The expected improved spatial resolution would take synchrotron-based spectromicroscopy into the regime previously accessible only by using electron-beam probes in transmission and scanning electron microscopes. Eventually, lens designers plan to refine the electronic optics and incorporate energy filtering (i.e., use an electron-energy analyzer) so that multiple electron spectromicroscopies (i.e., NEXAFS and XPS) will be possible with the same instrument

*A number of groups are working on this problem; we are following the electron-mirror correction method suggested and demonstrated by Rempfer at Portland State University, Oregon.



CAD drawing of a new 30-kV x-ray photoemission electron microscope (X-PEEM) now under construction at the ALS for full-field imaging shows the multiple lens system whose high voltage, small aperture, and beam-guiding deflectors should, according to extensive computer modeling, give a spatial resolution around 30 nm. A future version of the X-PEEM will incorporate an electron mirror for correction of chromatic aberrations and still better resolution.

Getting Started

The
ALS

A Growing Complement Of Tools & Techniques

New research tools often have unforeseen benefits. For example, soft x-ray fluorescence spectroscopy at undulator beamlines 7.0 and 8.0 (shown here) has blossomed into a powerful probe of electronic structure at surfaces and in the bulk.

The characteristics and capabilities of soft x-ray spectromicroscopy reviewed in the previous pages show that applications to the microcharacterization of materials and surfaces are potentially widespread and limited only by the imagination and creativity of those with problems to solve.

But the question may arise: why another form of microscopy? There are already a plethora of microscopies based on electron and ion probes that have excellent spatial resolution and even elemental sensitivity. In addition, the new generation of scanning probe techniques (STM, AFM, etc.) offer a wealth of information. The answer is that no single technique can provide all the answers that materials scientists seek, so spectromicroscopy should be viewed as bringing a major new capability to materials microcharacterization, rather than as replacing established techniques. Moreover, the field of spectromicroscopy has only recently been born, and we are just setting out to develop applications and the techniques to carry them out.

Because the ALS is a relatively new facility, our complement of experimental instrumentation continues to grow as additional research opportunities become defined and funding becomes available. Accordingly, we are providing information about specific spectromicroscopy facilities and successful demonstrations of their applications in the form of loose-leaf inserts in the pocket on the inside back cover of this brochure. These inserts are modified or added as new information becomes available, so that they represent the most up-to-date information about spectromicroscopy opportunities at the ALS.

Participation

How To Conduct Research At The ALS

On the Web

For a general facility overview & late-breaking news about operations & science, check the ALS home page on the World Wide Web. The URL is:

http://www-als.lbl.gov/als/

Publications & Proposal Submission

For publications or information about submitting a proposal, contact:

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Spectromicroscopy Opportunities

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. . . or spokespersons for the spectromicroscopy PRTs listed in the data sheets in the back pocket of the brochure.

The ALS provides synchrotron radiation year around, except for scheduled shutdowns (usually one major shutdown each year lasting several weeks) for installation of new experimental facilities and for accelerator maintenance. The current operating schedule provides 15 shifts per week (8 hours/shift) for users. As a U.S. Department of Energy national user facility, the ALS is available without charge to personnel from university, industrial, and government laboratories for nonproprietary research intended for publication in the open literature. Proprietary research is also welcome but is subject to a nominal cost-recovery charge for provision of beam time. Proprietary users have the option to take title to any inventions made during the proprietary research program and treat as confidential all technical data generated during the program.

Whether nonproprietary or proprietary, there are three modes of conducting research at the ALS: as a member of a participating research team (PRT), as an independent investigator, or as a collaborator with a PRT. Collaborative groups comprising research personnel from one or more institutions with common research interests, PRTs contribute to the construction, operation, and maintenance of experimental facilities at the ALS. In return for their contributions, PRT members are granted priority for a percentage of the operating time on their facilities. The remaining operating time on each beamline is available to independent investigators. The proportion of time allotted to independent investigators varies from beamline to beamline. Independent investigators may bring their own experimental stations to ALS beamlines or with PRT concurrence use PRT stations.

Proposals for the establishment of new PRTs are reviewed by the Program Advisory Committee. Those interested in collaboration with a PRT, which may be a productive mode of entry to the ALS for new users, should contact the spokesperson for the appropriate PRT. Proposals from independent investigators are peer-reviewed by the Proposal Study Panel twice a year with 1 June and 1 December deadlines for receipt of proposals. For details, consult the ALS Users' Handbook, which is available from the User Administrator (see box). Additional publications available include a safety handbook, a beamline-design guide, and an annual activity report describing the previous year's accomplishments.

Additional Reading

Publications in the literature relevant to spectromicroscopy for materials microcharacterization include:

H. Ade (editor), Special issue on spectromicroscopy with x-ray and VUV photons, *Journal of Electron Spectroscopy and Related Phenomena* **258** (1) (to be published in spring 1997).

H. Ade et al., "Chemical Contrast in X-Ray Microscopy and Spatially Resolved XANES Spectroscopy of Organic Specimens," *Science* **258**, 972 (1992).

H. Ade and B. Hsiao, "X-Ray Linear Dichroism Microscopy," *Science* **262**, 1427 (1993).

G.F. Rempfer, D.M. Desloge, W.P. Skoczylals, and O.H. Griffith, "Simultaneous Correction of Spherical and Chromatic Aberrations with an Electron Mirror: An Electron Optical Achromat," *Microscopy and Microanalysis* **3**, 14 (1997).

G. R. Harp, Z. L. Han, and B. P. Tonner, "X-Ray Absorption Near Edge Structures of Intermediate Oxidation States of Silicon in Silicon Oxides During Thermal Desorption," *Journal of Vacuum Science and Technology A* **8**, 2561 (1990).

J. Kirz, C. Jacobsen, and M. Howells, "Soft X-Ray Microscopes and Their Biological Applications," *Quarterly Review of Biophysics* **28**, 33 (1995). Despite the emphasis on biology, a good summary of microscopy principles.

W. Ng. et al., "High Resolution Spectromicroscopy with MAXIMUM: Photoemission Microscopy Reaches the 1000 Å Scale," *Nuclear Instruments and Methods in Physics Research A* **347**, 422 (1994).

J. Stöhr et al., "Element-Specific Magnetic Microscopy with Circularly Polarized X-Rays," *Science* **259**, 658 (1993).

B. P. Tonner, G. R. Harp, S. F. Koranda, and J. Zhang, "An Electrostatic Microscope for Synchrotron Radiation with X-Ray Absorption Microspectroscopy," *Review of Scientific Instruments* **63** (1), 564 (1992).

B. P. Tonner, "The Role of High Spectral Resolution in Soft X-Ray Microscopy," in *X-Ray Microscopy and Spectromicroscopy*, J. Thieme, G. Schmahl, E. Umbach, and D. Rudolph, eds., Springer-Verlag, Heidelberg, to be published in 1997. This is the proceedings of XRM '96 International Conference on X-Ray Microscopy and Spectromicroscopy, Würzburg, Germany, 19–23 August 1996.

T. Warwick et al., "Soft X-Ray Spectromicroscopy Development for Materials Science at the Advanced Light Source," in the special issue on spectromicroscopy with x-ray and VUV photons, *Journal of Electron Spectroscopy and Related Phenomena* **258** (1) (to be published in spring 1997). Also published as Lawrence Berkeley Laboratory Report LBNL-38906, August 1996.

***Synchrotron Radiation News* reports on developments in synchrotron radiation at facilities around the world. Articles on x-ray spectromicroscopy for materials analysis include:**

H. Ade et al., "Industrial Applications of X-ray Microscopy," *Synchrotron Radiation News* **9**, 31 (No. 5, September/October 1996).

H. Ade, "NEXAFS Microscopy of Polymeric Samples," *Synchrotron Radiation News* **7**, 11 (No. 2, March/April 1994).

W. Ng et al., Study of Surfaces and Interfaces by Scanning Photoemission Microscopy," *Synchrotron Radiation News* **7**, 25 (No. 2, March/April 1994).

B. P. Tonner, "Photoemission Spectromicroscopy of Surfaces in Materials Science," *Synchrotron Radiation News* **4**, 27 (No. 2, March/April 1991).

Recent proceedings of major x-ray microscopy conferences include:

X-Ray Microscopy and Spectromicroscopy, J. Thieme, G. Schmahl, E. Umbach, and D. Rudolph, eds., Springer-Verlag, Heidelberg, to be published in 1997. This is the proceedings of XRM '96 International Conference on X-Ray Microscopy and Spectromicroscopy, Würzburg, Germany, 19–23 August 1996.

X-Ray Microscopy IV, V. V. Aristov and A. I. Erko, eds., Begorodski Pechatnik Publishing Company, Moscow, 1995.

X-Ray Microscopy III, D. A. Michette, G. Morrison, and C. Buckley, eds., Springer Series in Optical Sciences Vol. 67, Springer-Verlag, Berlin/New York, 1992.

National and international conferences on synchrotron radiation are held every two and three years, respectively. Recent proceedings include:

G. K. Shenoy and J. L. Dehmer, Eds., "Proceedings of the Ninth National Conference on Synchrotron Radiation Instrumentation," *Review of Scientific Instruments* **67** (9), (1996). Available only on CD.

J. B. Hastings, S. L. Hurlbert, and G. P. Williams, Eds., "Proceedings of the Fifth International Conference on Synchrotron Radiation Instrumentation," *Review of Scientific Instruments* **66** (2), (1995).

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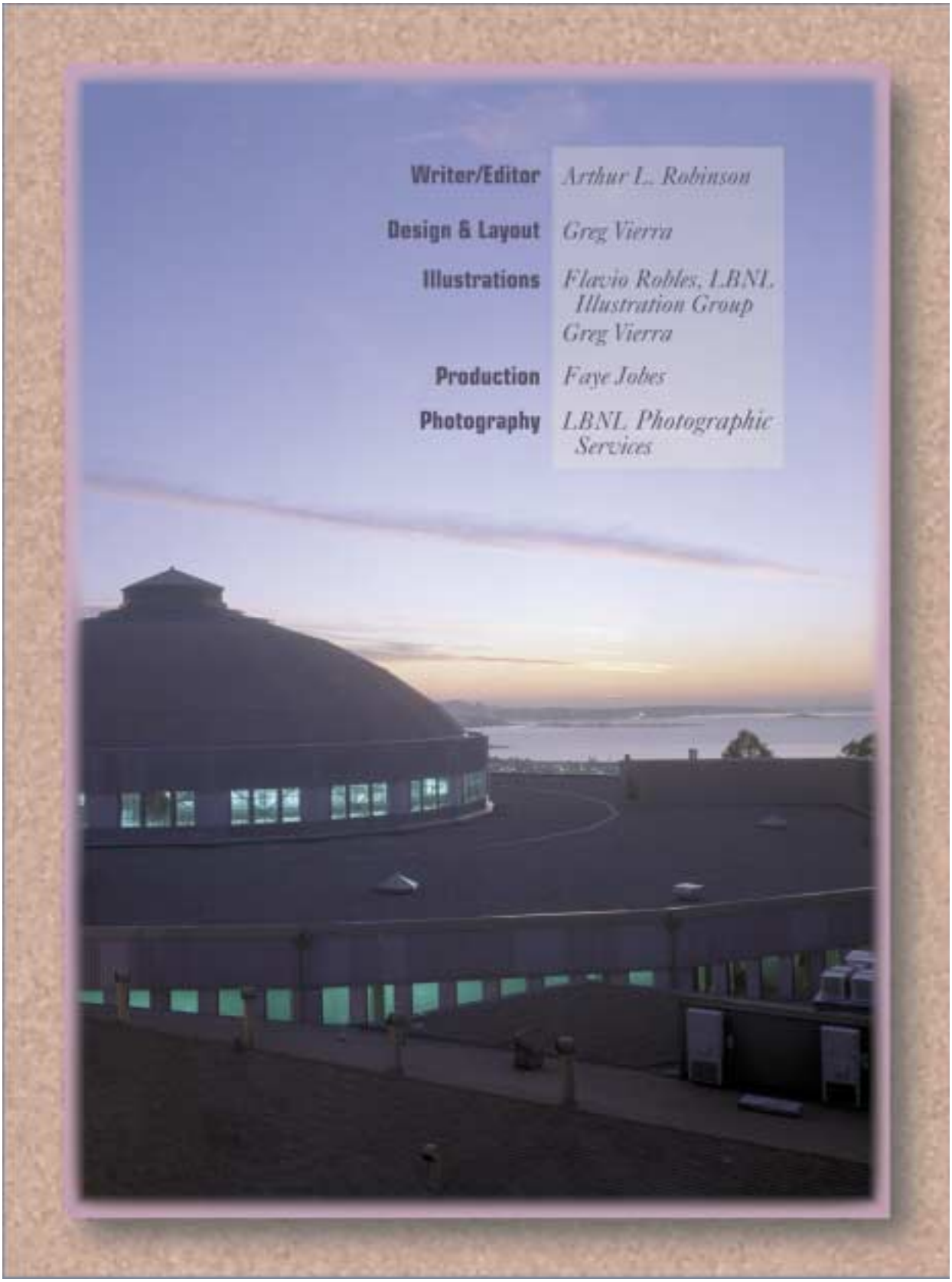
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