# SCIENCE WITH SOFT X RAYS

Synchrotron radiation with photon energies at or below 1 keV is giving new insights into such areas as wet cell biology, condensed matter physics and extreme ultraviolet optics technology.

### Neville Smith

"But, soft! What light ..." — William Shakespeare Romeo and Juliet

Researchers using synchrotron radiation fall into two fairly distinct camps: users of the soft x-ray and vacuum ultraviolet (VUV) region of the spectrum and users of the hard x-ray region. The distinction can be expressed quantitatively by comparing the energies of a photon and an electron whose wavelengths are 1 Å.

The scientific question posed by the users of hard x rays tends to be, Where are the atoms? The emphasis is on the determination of crystal structures and molecular structures using techniques such as x-ray diffraction. Therefore, the photon used as the probe should have a wavelength comparable to interatomic distances. With photon wavelength  $\lambda_{\rm ph}=1$  Å, we have

$$E ext{ (photon)} = hc/\lambda_{\text{ph}} \approx 12.4 \text{ keV}.$$

In the soft x-ray/VUV region, on the other hand, the question tends to be, What are the electrons doing as they migrate between the atoms? The emphasis is on studies of chemical bonding and valence band structures using techniques such as photoelectron emission spectroscopy. For users of this region, the probing photoelectron should have a wavelength comparable to interatomic distances. With electron wavelength  $\lambda_{\rm el}=1$  Å (and electron mass m), we have

$$E ext{ (electron)} = h^2/(2m\lambda_{el}^2) \approx 150 \text{ eV}.$$

This natural division of researchers in terms of two energy regions of interest is reflected in the establishment in the US of two new synchrotron radiation facilities: the Advanced Photon Source at Argonne National Laboratory, optimized for delivery of hard x rays, and the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory (LBNL), optimized for soft x-ray/VUV science (see PHYSICS TODAY, January 1994, page 18).

Between the two energy values just derived lies the soft x-ray region, with photon energies of 100–1000 eV. Such soft x rays are used in spectroscopy of inner shell (core) electrons, particularly for elements in the first row of the periodic table. These important elements have only one core level (the K-shell), and the core electron binding

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energy is in the soft x-ray region—for example, the binding energies of 1s electrons in carbon, nitrogen, and oxygen are 290, 400, and 530 eV, respectively. Core-level spectroscopy is a powerful technique both because of its elemental specificity and because it can reveal details of an atom's chemical bonding state, which has a significant effect on some spectral features just above the absorption edge. Also falling within the soft x-ray region are the Ledges of the transition metals, which come from the dipole-permitted transition from the 2p core level to the 3d valence level. Of particular interest are the elemental ferromagnets iron (with L-edge energy 710 eV), cobalt (780 eV), and nickel (850 eV). Fairly recently, the discoveries of cuprate high-temperature superconductors and manganate colossal magnetoresistance materials have propelled the L-edges of copper (930 eV) and manganese (640 eV) into prominence. The L-edge is attractive to researchers studying the new materials because of its ability to probe the 3d valence electrons that are responsible for their remarkable properties.

The following is a sample of recent science done using the soft x-ray and VUV part of the spectrum. For reasons of familiarity, the examples here are taken from work done at the ALS and at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. The new "third generation" facilities like the ALS were constructed to optimize radiation output (see Physics Today, April 1991, page 17), and the experiments described here are of the kind that profit from the higher brightness of the newer sources. Higher brightness gives experiments three principal advantages: higher spatial resolution, higher spectral resolution, and higher coherence (see the box on page 30). Despite the focus in this article on high-brightness sources, let me emphasize that similar work is being done at new and old sources both in the US and elsewhere (see also the article by Jens Als-Nielsen and Gerhard Materlik in PHYSICS TODAY, November 1995, page 34).

# High spatial-resolution microscopy

An important feature of the soft x-ray region is the "water window," the photon energy range between the carbon K-edge (290 eV) and the oxygen K-edge (530 eV). In this range, organic matter (that is, carbon) is absorbing, whereas water (that is, oxygen) is relatively transparent, thus providing a contrast mechanism that permits microscopic analysis of cells in their natural aqueous environ-

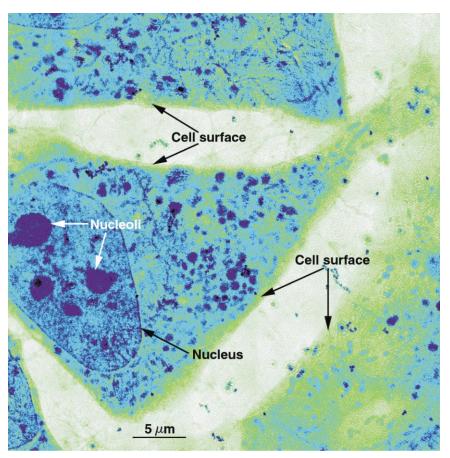


FIGURE 1. TRANSMISSION MICROGRAPH of mouse 3T3 fibroblasts, a type of connective-tissue cell. The data were taken in the "water window" (photon energy 290–530 eV) with a spatial resolution of 36 nm, and clearly show features—such as nucleoli and the sharp nuclear membrane—not resolvable with optical confocal microscopy. (Photo courtesy of C. Larabell.)

tial resolution is limited by the wavelength of light. A soft x-ray equivalent to this technique can be constructed using the Fresnel zone plate, a device based on the focusing properties of alternating transmitting and absorbing concentric rings. By employing such focusing elements, it is possible to use x rays with photon energies in the water window to see features 5–10 times smaller than what can be seen with the optical confocal microscope.

A team of cell biologists working under the leadership of Carolyn Larabell at LBNL is addressing the frontier problem of determining the localization, co-localization, and redistribution of proteins as they perform their functions in cells. Figure 1 shows mouse 3T3 fibroblasts (a type of cell from connective tissue), imaged using an x-ray

microscope¹ with a spatial resolution of 36 nm. The sample was taken fresh from culture and cryofixed (frozen with liquid nitrogen-cooled helium gas); as a result, the cells more closely resemble their native state than they would if they had been chemically fixed. Using this approach, the cellular ultrastructure is extremely well preserved and is revealed with a unique combination of high spatial resolu-

ment. Carbon near-edge spectra are rich in features and provide a chemical fingerprint that serves as another contrast mechanism for the microscopic investigation of systems such as polymer blends, which are of critical importance to the chemical industry.

In wet cell biology, confocal microscopy in the visible region is a widely used and powerful technique, but spa-

## The Brightness Advantage

The newer synchrotron radiation sources have been designed for high brightness, sometimes called brilliance. Brightness is a measure of the number of photons per second per solid angle per source area per unit bandwidth; thus, higher brightness can be achieved by increasing the number of photons, reducing the source size, or reducing the solid angle of emission.

The highest brightness is achieved with the use of "undulators," periodic magnetic structures that force the electrons in the synchrotron storage ring to oscillate about their central trajectory. The cumulative effect of many such undulations is to produce a beam of radiation in a very narrow angular cone about the forward direction, as shown in the figure. (As indicated, the angular width of the cone,  $\theta_{cen}$ , depends on N, the number of undulator periods, and  $\gamma$ , the electron energy in terms of its rest mass.) This, combined with the inherent smallness (30  $\mu$ m  $\times$  200  $\mu$ m) of the electron source in the ring, is the brightness advantage. <sup>12</sup>

But what does brightness really buy you? Brightness is a conserved quantity through a perfect optical system, which means that high brightness makes it possible to put a lot of light into a small spot. This has three practical consequences:

Small spot on the sample. Techniques (such as spectroscopy and diffraction) can now be performed with fine spatial resolution, giving rise to a variety of x-ray microscopes.

Narrow slits. With small spot size, it is possible to put a lot of light through the very narrow slits of a monochromator or spectrometer, thereby trading brightness for very high spectral-resolving power.

Tiny pinholes. It is likewise possible to get a lot of light through tiny pinholes, thereby generating perfect spherical waves of high amplitude. This is the property of spatial coherence that can be exploited for interferometry, speckle, and dynamic scattering experiments.

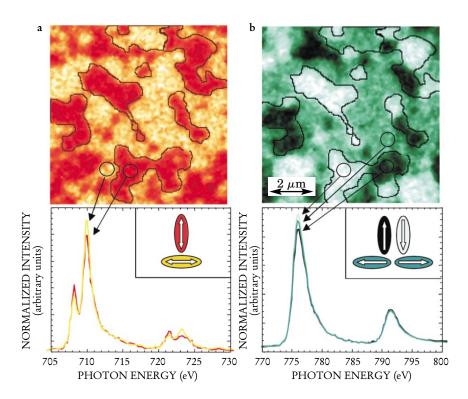


FIGURE 2. IMAGES AND LOCAL SPECTRA from a ferromagnetic cobalt layer on an antiferromagnetic LaFeO, substrate. (a) Antiferromagnetic contrast is obtained at the iron L-edge using linear dichroism, which uses the difference in absorption of linearly polarized x rays when the electric vector of the light is parallel or perpendicular to the antiferromagnetic axis. (b) Ferromagnetic contrast is obtained at the Co L-edge using circular dichroism, which uses the difference in absorption between left and right circularly-polarized x rays. The inset arrows indicate the in-plane orientation of the antiferromagnetic axis and ferromagnetic spins. Note that the ferromagnetic Co domains align with the substrate antiferromagnetic domains, but can split into subdomains of opposite magnetization. Spatial resolution of the images is 20 nm. (Figure courtesy of S. Anders.)

tion and good contrast.

In figure 1, numerous organelles, granules of various sizes, and tubular structures such as mitochondria can be readily seen in the cytoplasm. Even the contents of the nucleus, which is approximately 5  $\mu$ m thick in these cells, can be observed without the need for physically sectioning the cell. Nuclear structures such as the nucleoli, which are densely packed with ribonucleic acid, appear as dense bodies, and the nuclear envelope, a double-layered membrane encircling the entire nucleus, appears as a distinct line around the nucleus. The striking contrast of the cellular ultrastructure is solely the result of the use of the water window; these cells had not been exposed to any chemical fixatives or contrast enhancement agents. Because of the high differential contrast provided by x-ray imaging, the location of specific proteins and nucleic acids can be readily identified by using gold-tagged labels that are easily distinguished from biological constituents.

Other ongoing research includes an effort to obtain three-dimensional information by collecting images at different tilt angles and then using tomographic reconstructions;<sup>2</sup> this approach takes advantage of the large depth of focus of the Fresnel zone plates. The effects of radiation damage on soft matter have been of concern in the past; while these effects still need to be considered, they do not appear to be an obstacle to the emergence of x-ray microscopy as an important tool for determining structure–function relationships of proteins and cells.

# Photoelectron microscopy

Fresnel zone plates represent only one of the ways to achieve fine spatial resolution. Another way is to use electron optics rather than photon optics. In photoelectron emission microscopy (PEEM), the basic idea is to illuminate an area on the sample and then pass the ejected photoelectrons through an electron microscope column, thus producing an enlarged image of the illuminated spot. Because the detected particles are electrons instead of photons, the experiment must be done in vacuum; this

precludes the investigation of wet samples. Nevertheless, there are plenty of dry systems of interest.

Fritjhof Nolting and collaborators3 have recently shed light on the poorly understood phenomenon of exchange bias, which is the directional coupling between adjacent ferromagnetic and antiferromagnetic spins across an interface in a layered material. This is something of an old chestnut in condensed matter physics, but it has recently assumed considerable importance because of its relevance to the artificial magnetic layered structures used in the manufacture of magnetic read heads and magnetic memory cells (see the article by E. Dan Dahlberg and Jian-Gang Zhu in PHYSICS TODAY, April 1995, page 34). These magnetic structures use an antiferromagnetic material as a substrate to pin the orientation of the first ferromagnetic layer, but the pinning mechanism is not well understood. Nolting and his coworkers, by a very artful switch between two contrast mechanisms—linear dichroism for antiferromagnetic contrast and circular dichroism for ferromagnetic contrast—have provided an incisive new technique to study the problem.

The system chosen for study was a thin ferromagnetic Co film on an antiferromagnetic substrate of LaFeO<sub>3</sub>. Figure 2 shows the PEEM micrographs and spectra. The domain orientation in the antiferromagnetic substrate (vertical or horizontal) was determined, using x rays at the Fe L-edge, by means of linear dichroism—taking the difference in the absorption of linearly polarized x rays having electric field vector parallel to or perpendicular to the antiferromagnetic axis. The orientation of the ferromagnetic Co domains was determined, using x rays at the Co L-edge, by circular dichroism, which is based on the difference in absorption between left and right circularly polarized photons. In figure 2, the ferromagnetic domains of the Co overlayer are indeed aligned with the domains of the antiferromagnetic substrate, with the ferromagnetic magnetization pointing in either direction. The high-resolution PEEM technique can be used to zoom in on specific areas and measure the magnetization reversal there.

These measurements can be taken as a function of temperature; eventually, using the pulsed nature of synchrotron radiation, they will be taken as a function of time. The present spatial resolution of the PEEM measurements is 20 nm, but it is expected that aberration-corrected instruments being developed in the US and Germany will improve resolution to 2 nm.

### Spectral resolution and electronic structure

One of the most tantalizing problems in condensed matter physics is the origin of high-temperature superconductivity. Philip Anderson of Princeton University has declared his belief that angle-resolved photoemission spectroscopy (ARPES) will provide the "smoking gun" to support a theory based on spin excitations at the Fermi surface (the boundary in k space separating the occupied from the unoccupied electronic states), and that ARPES "is, for this problem, the experiment that will play the role that tunneling played for BCS" (see PHYSICS TODAY, June 1991, page 54). What ARPES essentially does is to measure both the energies E and momenta  $\mathbf{k}$  of photoelectrons, directly determining the  $E(\mathbf{k})$  dispersion relations. Great strides have indeed been made: ARPES experiments have shown that the supercurrent-carrying electrons pair in a highly anisotropic d-wave state4 (see PHYSICS TODAY, January 1996, page 19 and March 2000, page 17) and they have revealed the existence of a "pseudogap" between the conducting electrons and the Fermi surface (see PHYSICS TODAY, June 1996, page 17).

Increasing attention is now being paid to stripes,<sup>5</sup> a phase with regular magnetic and charge ordering that has been observed in many high-temperature superconductors (see Physics Today, June 1998, page 19). Recently, some ARPES experiments by Zhi-xun Shen and coworkers<sup>6</sup> have made a connection between stripes and the Fermi surface. The experiments were done on a model cuprate compound, (La<sub>1.28</sub> Nd<sub>0.6</sub>Sr<sub>0.12</sub>)CuO<sub>4</sub> (called Nd–LSCO). This remarkable

compound has the one-eighth strontium doping (Sr<sub>0.12</sub>) at which superconductivity is actually suppressed. Replacing some lanthanum in LSCO with neodymium stabilizes the stripe phase; with this doping, the phase consists of rows of charge carriers separated by triple rows of antiferromagnetically ordered spins whose ordering flips phase across the charge stripes (see figure 3). Shen and coworkers extracted a Fermi surface for this system

by integrating the photoemission spectra over an energy range near the Fermi level to obtain maps of the electron momentum distribution function. As shown in figure 3, the Fermi surface they determined is in the shape of a cross. A likely interpretation of the data, consistent with the stripe picture, is that the cross is a superposition of bar-shaped Fermi surfaces from two orthogonal domains, depicted in the diagrams in the upper left and lower right of figure 3. The boundaries of the bars are given  $|k_x| \leq \pi/4a$  and  $|k_y| \leq \pi/4a$  (where a is the lattice constant), consistent with the fourfold periodicity and the oneeighth doping. The shape of the Fermi surface implies that the electron system is highly one-dimensional; the data are inconsistent with the more rounded Fermi surface calculated for two-dimensional copper-oxygen planes.

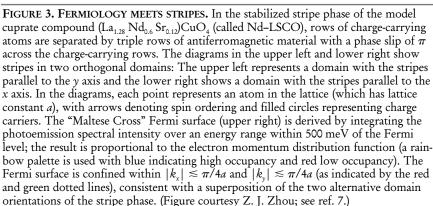
## Approaching the Fermi level

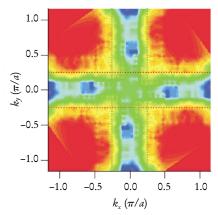
Increases in spectral resolving power are permitting photoemission spectroscopists to probe behavior ever closer to the Fermi level  $E_{{\scriptscriptstyle F}}$ . Energy–momentum relations  $E({\bf k})$ can now be found with great precision. For example, the long-anticipated kink in the  $E(\mathbf{k})$  curve close to  $E_F$  due to the electron-phonon interaction has now been observed. Attention has also turned to the lifetime width of peaks in the photoemission spectra. The energy dependence of the lifetime width in the vicinity of  $E_F$  is a strong indicator of whether or not a material behaves like a Fermi liquid. And it is the excitations around the Fermi energy that are of prime importance for determining the origin of hightemperature superconductivity.

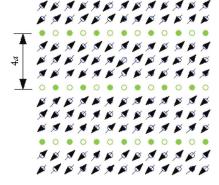
The measured photoelectron energy spectrum at a given angle of emission is proportional to the spectral function, given by

$$A\left(\mathbf{k},\omega\right) \propto \frac{\mathrm{Im}\Sigma\left(\mathbf{k},\omega\right)}{\left[\omega-E_{k}-\mathrm{Re}\Sigma\left(\mathbf{k},\omega\right)\right]^{2}+\left[\mathrm{Im}\Sigma\left(\mathbf{k},\omega\right)\right]^{2}}$$









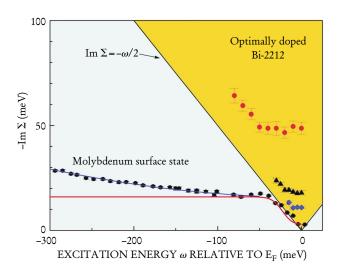


FIGURE 4. EXCITATION LIFETIME WIDTHS in two materials near the Fermi level, as measured by angle-resolved photoemission spectroscopy (ARPES). The lifetime of an electronic excited state is inversely proportional to the magnitude of the imaginary part of the single-particle self energy  $|\text{Im }\Sigma|$ , which is observed with ARPES by measuring the width of the photoemission peak; the lifetime width is  $-2 \text{ Im } \Sigma$ . A molybdenum surface state, Mo(110), was used as a test-bed to establish the validity of the method; the results (black circles) are well matched by theory (blue curve). The blue curve is the sum of contributions from the electron-phonon interaction (red curve) and from Fermi-liquid theory (a parabola, not shown). In contrast, measurements of the superconducting material Bi-2212 suggest that the system exhibits quantum critical behavior. Lifetime widths for Bi-2212 are shown for three temperatures: 300 K (red circles), 90 K (black triangles), and 48 K (blue diamonds). The widths lie in a region (shaded orange), where Fermi liquid theory does not apply. (Figure courtesy of T. Valla.)

where Re  $\Sigma(\mathbf{k},\omega)$  and Im  $\Sigma(\mathbf{k},\omega)$  are the real and imaginary parts of the single-particle self energy and  $E_k$  is the single-particle (Hartree) energy. The imaginary part of the self energy is inversely proportional to the lifetime of an electronic excitation in the material, and can readily be determined using ARPES. Modern state-of-the-art photoemission spectrometers simultaneously display two dimensions of information: photoelectron energy and angle. Peter Johnson and his collaborators, working at the NSLS, have shown that the cleanest cut through this space for the determination of Im  $\Sigma$  is the momentum distribution curve (MDC), which is a plot of photoemission intensity as a function of momentum **k** for a constant excitation energy  $\omega$ . The MDCs are immune from distorting effects of the Fermi function and the inelastic background to the spectra.

Figure 4 shows a comparison of lifetime widths measured by Johnson's group on optimally doped  $Bi_2Sr_2CaCu_2O_{8+\delta}$  (called  $Bi\text{-}2212)^8$  with those measured on a test-bed system, a molybdenum surface state. The test-bed system was used because the measurement of photoemission linewidths is fraught with experimental artifacts, and a well-understood system such as an established surface state on the Mo(110) surface provides a good test of the method. In fact, the test-bed measurements shown in figure 4 match the theory well: The  $\omega$  dependence of Im  $\Sigma$  is well described by a contribution due to the electron–phonon interaction plus an  $\omega^2$  term characteris-

tic of a Landau Fermi liquid. By contrast, the measurements of Im  $\Sigma$  for Bi-2212 are clearly inconsistent with Fermi liquid behavior. To be valid, Fermi liquid theory requires that  $2 \mid \text{Im } \Sigma \mid < \omega$ , but the Bi-2212 data lie above the line in the figure marked  $|\text{Im } \Sigma | = \omega/2$ . The  $\omega$  dependence of the data and the temperature dependence of the self-energy suggest that the system exhibits quantum critical behavior. This interpretation, like the previous discussion concerning the stripe phase, remains controversial. Nevertheless, the incisiveness of ARPES does appear likely to provide Anderson's smoking gun. The key will be further improvements in energy resolution, for which high-brightness synchrotron radiation will be essential.

# Coherence and interferometry

Following Moore's Law, the density of circuit elements on microchips has doubled roughly every 18 months, resulting in smaller, faster, and cheaper computers. However, optical lithography, the present technology of microchip manufacture, cannot continue indefinitely on this course. The materials from which one could conceivably make optical lenses, CaF<sub>2</sub>, MgF<sub>2</sub>, and LiF, will not transmit light at wavelengths less than 100 nm, which limits the size of features that could be fabricated in this way. One alternative being considered is to switch from refractive optics to reflective optics—that is, mirrors. The availability of molybdenum—silicon mirror coatings that have reflectances as high as 70% provides special impetus for reflective technol-

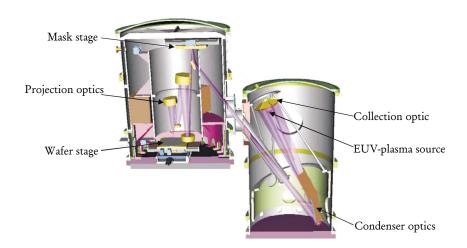


FIGURE 5. AN OPTICAL SYSTEM for the manufacture of microchips using extreme ultraviolet (EUV) projection lithography at 13 nm. Multilayer coated mirrors (yellow) in the stepper create a reduced image of the mask onto the wafer. A plasma source provides the EUV radiation. Synchrotron-generated soft x rays are used to measure mirror characteristics. (Figure courtesy of R. Stulen, Sandia National Laboratory.)

ogy. The optimum wavelength, determined as a compromise between the conflicting requirements of fine lateral spatial resolution and large depth of focus into the chip structure, is about 13 nm. This wavelength is popularly referred to as extreme ultraviolet (EUV); the corresponding photon energy is 100 eV, which qualifies it as a soft x ray.

A consortium of microelectronics companies (Intel, Motorola, and Advanced Micro Devices) has joined forces with a consortium of national laboratories (Lawrence Livermore, Sandia, and LBNL) to build a prototype EUV stepper, an optical camera of the sort that will produce small computer chips. A schematic of such a stepper is shown in figure 5. It comprises a four-mirror optic that produces a reduced image of the design mask on the wafer. To attain the required high reflectivity at 13 nm wavelength, the mirrors in the device must be curved and coated with interfering multilayers. In actual chip manufacture, the light source will not be synchrotron radiation but rather an EUV-emitting plasma. The role of synchrotron radiation for this technology is to measure the optics.

An old adage says, "If you can't measure it, you can't make it." At LBNL, Jeff Bokor is leading a team that has been entrusted with the task of interferometric characterization of the high-precision mirrors required for the stepper. As part of this effort, the team has developed an atwavelength phase-shifting point-diffraction interferometer (PS/PDI).9 The PS/PDI first passes the beam from an ALS undulator through a pinhole to generate a high-amplitude, perfectly spherical wave. A diffraction grating then splits the light into two parts: a test beam, which passes through the optic being measured, generating an aberrated wavefront characteristic of the imperfections in the test optic; and a reference beam, which passes unobstructed through the test optic and then is forced through a second pinhole to generate a perfectly spherical reference wavefront that interferes with the test beam. The resulting interferogram reveals the departures from figure accuracy. The design goal was for a test accuracy of 0.10 nm over the surface of the mirror, and the accuracy actually achieved was 0.05 nm. This length is the Bohr radius of a hydrogen atom! A four-mirror optic manufactured at Sandia has already been tested with very satisfactory results. This work demonstrates that coated optics can be produced to the desired tolerance, thereby enhancing the prospects of EUV lithography as the next technology of choice for the manufacture of ever denser microchips.

# Great promise for the future

The preceding is an attempt to convey some of the richness of the science that can be done with synchrotron radiation in the soft x-ray and VUV regions of the spectrum and to show some of the promise of the newer high-brightness facilities. However, there is much more activity in the field than could be covered in this article. A particularly promising area of current research is soft x-ray emission spectroscopy, 10 a photon-in/photon-out technique in which the incident beam photon energy is chosen to knock out core electrons of, say, carbon atoms. Valence electrons can then decay into the resulting hole by releasing soft x-ray photons, whose spectrum replicates the local valence density of states. The technique has the power of photoelectron emission as well as atomic specificity. Since the detected particle is a photon rather than an electron, the measurements do not have to be done in ultrahigh vacuum, as photoelectron emission measurements do. Because a high vacuum is not needed, soft x-ray emission spectroscopy can be used to

study buried interfaces and the wet samples that are of intense interest in catalysis, biology, and environmental science. The absorption cross sections are very small, however, and a high-brightness source is required to achieve the full potential of this technique. Another promising area is high-resolution spectroscopy in the gas phase. Synchrotron-based experiments are being done in atomic and molecular physics and in chemical dynamics that are not possible with laser sources.11

The potential of high coherence, one of the three brightness advantages, remains relatively unexploited. Fledgling attempts at speckle and dynamic scattering have only just begun in the soft x-ray range. The length scales that can be probed in this way are, of course, greater than those accessible using hard x rays, but the larger soft x-ray cross section (which scales as the square of the wavelength) is a real advantage. Thus, we can confidently expect a surge of effort in dynamic scattering studies of systems with the appropriate length scale in magnetism, soft matter, and biology. It is obvious that we have only scratched the surface of "Science with Soft X Rays".

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