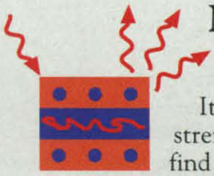
X-rays Find New Ways to Shine

The brightness and beam quality of the new third-generation synchrotrons are helping researchers to probe matter more quickly and effectively, and to try out some new tricks



Materials researchers have long wanted to unravel the structure of spider silk. Its unique combination of strength and elasticity would find innumerable applications if it could be reproduced in industry. So far, the detailed molecular structure responsible for these properties has eluded researchers. But now one team is bringing a new sledgehammer to bear on this intractable nut: a stadium-sized machine called the European Synchrotron Radiation Facility (ESRF), in Grenoble, France. With the ESRF's brilliant, needlelike beams of x-rays, they hope to dissect single fibers of spider silk.

ESRF is one of a new generation of synchrotron radiation sources with high-quality beams of high-energy, or hard, x-rays that are revolutionizing many areas of research—from materials to geophysics to molecular biology. Previous generations of synchrotrons, for example, produced beams about 100 micrometers across, far too coarse to reveal the structure of a 5- to 8-micrometer strand of spider silk. But a team led by Christian Riekel has taken the ESRF's beam—already finer and brighter than those of older synchrotrons—and focused it down to a width of 1 or 2 micrometers. Confident that this will reveal the spider's secret, the group has already learned, says Riekel, "that you can get enough signal—a diffraction pattern—from a single fiber, and this is big progress. ... A few years ago we needed at least a few hundred fibers."

ESRF was the first of these third-generation synchrotrons producing hard x-rays to come online, in 1992, and it was opened to the x-ray community in 1994. In the United States, the Advanced Photon Source (APS) at Argonne National Laboratory near Chicago generated its first x-ray beams in March 1995 and opened to users in 1996. And Japan has just begun commissioning the third and most powerful of the sources, Spring-8. The new sources improve on their predecessors not only through raw power, but also by tailoring the x-ray beam to users' precise requirements with devices called undulators and wigglers. These "insertion devices" can tune the beam's frequency, make it coherent, polarize it, and focus it to exquisite sharpness.

All of this enables these machines not only to outdo their predecessors in determining material structures by the tried-and-tested technique of x-ray diffraction, but also to exploit new techniques for probing materials. Going by names such as holography, phase-contrast imaging, tomography, scanning microscopy, microdiffraction, and photon correlation spectroscopy, among others, these techniques can resolve minute defects and structural interfaces in materials. Take phase-contrast imaging, which can detect tiny cracks in metals or interfaces between soft matter by tracking shifts in the phase of the x-rays passing through the object. The technique "is possible with second-generation sources," says Friso van der Veen of the University of Amsterdam in the Netherlands, "but it has undergone an acceleration with the introduction of ESRF, because the beam has such a strong brilliance."

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Ring of light. The Advanced Photon Source at Argonne National Laboratory will give thousands of researchers from diverse fields access to x-rays of unprecedented quality.

The principle of a synchrotron source is simple: When fast-moving charged particles, such as electrons, are forced into a circular path by magnets, they shed energy in the form of photons. With enough particles and sufficient speed, the result is powerful x-ray beams. Earlier synchrotrons relied simply on bending magnets to get the particles to emit photons, but third-generation sources use a different approach. In straight sections of the electron storage ring, they position insertion devices. One type, called an undulator, consists of rows of magnets of alternating polarity that force the electrons to follow a slalom path. At every turn the electrons emit photons, and the photons from all the turns add up to produce an intense, sharply focused beam just 50 micrometers wide. "With undulators, we obtain beams that are narrower than laser beams," says Pascal Elleaume, who is responsible for undulator development at ESRF. The undulator radiation is also coherent: Photons in the beam that have the same wavelength travel in step. Other insertion devices called wigglers force electrons into a giant slalom—fewer and wider turns—to produce beams that are less well collimated but have a continuous spectrum of energies, akin to white light.

These beams can then be brought to an even sharper focus with the special x-ray optical devices that synchrotron researchers are developing. X-rays cannot be refracted by a lens, but mirrors can deflect x-rays at very low grazing angles, bringing them to a focus. Researchers are also trying out other types of optics. For example, ESRF's Anatoli Snigirev has developed a lens consisting of series of holes drilled in pieces of aluminum or beryllium. To an x-ray, the holes have a greater refractive index than the metal, so the holes act as an array of lenses. Another focusing system uses glass capillary tubes with a reflecting inner surface. And a collaboration of researchers from APS, ESRF, and Brookhaven National Laboratory has built a thin-film wave guide consisting of a layer of polyimide 1556 angstroms thick, sandwiched between totally reflecting layers of silicon and silicon dioxide. "With capillary tubes, you can get a beam down to a micron, but wave guides can get the beam down to the angstrom level, 100 to 1000 angstroms," says Sunil Sinha, who is responsible for inhouse experiments at APS.

Such small beam sizes are transforming diffraction, the standard technique for investigating crystal structure, in which a beam is fired at a crystal and the pattern of deflected photons provides details of its interior. Because the very fine beams provided by the new sources are, in most cases, smaller than the crystal, researchers can investigate different parts of a crystal separately. Says Riekel: "You can look at inhomogeneities inside the crystal, or inside a large sample, such as a polymer fiber"—or a strand of spider silk. And Simon Mochrie of the Massachusetts Institute of Technology explains that because x-rays have wavelengths much shorter than light, ranging from a fraction of 1 angstrom to a few angstroms, they can resolve much more detail. "Polymer molecules are about 100 to 1000 angstroms wide, and that is a convenient size you can reach with x-rays."

Because the beams are brilliant as well as finely focused, researchers can gather data much more quickly than they could at previous sources. While speed is always welcome, it has opened up new opportunities. For example, Walter Lowe at Howard University in Washington, D.C., heads a team whose first experiment on an APS beamline used a very intense beam to record diffraction patterns of a semiconductor film at a rate of 30 frames per second. Besides probing the material, the beam also heats it. "The beam does some annealing in the film and we look at thermal changes," says Lowe. While this first study simply demonstrated what was possible, Lowe's team plans to look at the dynamics of many different materials. "We look at everything from biology to semiconductor materials," he says.

Researchers at ESRF are doing similar work, says Director-General Yves Petroff: "We can now look—at a time scale of a few nanoseconds, with a repetition rate of 100 picoseconds—at what happens in a molecule" as it reacts with another molecule or changes shape. "We can make a 'movie' of what happens. ... This is something entirely new and has never been done before."

The coherence of the undulator beams is also allowing researchers to exploit new types of imaging. One, phase-contrast imaging, exploits two coherent beams that have slightly different phases. Superimposing the beams yields an "interference" pattern—the intensity of the combined beam will decrease where the phase difference becomes greater. This phenomenon allows researchers to image body tissues and minute cracks in metals, which do not block x-rays but simply shift their phase slightly. When x-rays that have passed through the region of interest are combined with x-rays that have bypassed it, phase shifts will show up as changes in intensity, forming an image.

In the past, researchers made such phase-contrast images by separating a beam into two components and sending one through the object while directing the other around it. More recently, researchers have simplified this process with a technique called "inline holography," in which a small object is placed in the middle of a large coherent beam. The parts of the beam that pass through the object then combine with those that pass around it.

Researchers are now enhancing phase-contrast imaging with other techniques. In x-ray microscopy, for example, a coherent beam is made to diverge by passing it through a thin-film wave guide. It then casts an enlarged image of the sample on a detector placed some distance away. "We have achieved a microscope that operates ... with a resolution of 1200 angstroms," says Petroff. And by taking multiple images of a sample, such as biological tissue or fibers, from different orientations and combining them in a computer, researchers can reconstruct three-dimensional images of the sample's interior, a technique called phase-contrast x-ray–computed tomography.

Another technique made possible by undulators' coherent x-ray beams is photon correlation spectroscopy, otherwise known as "speckle spectroscopy." It can garner information from disordered materials, such as colloids, polymers, alloys, and synthetic multilayers, which have structures that are too chaotic for diffraction studies. A coherent beam scattered from such materials produces a set of speckles characteristic of the sample. The speckle pattern does not provide any insight into the material's structure, but researchers can follow how the intensity of specific speckles change over time, gleaning information on phenomena such as phase transitions and sedimentation. "People have been doing this for many years with lasers," says ESRF's Gerhard Grübel. "But now you can do it in the x-ray regime, and that means that your spatial resolution has become much finer" because of the much shorter wavelengths of x-rays. "You can now probe the disorder and dynamics of systems on an atomic-length scale," he says.

Although researchers at the new sources, in particular at ESRF, are already turning out new results at a prodigious rate, they are also exploring and perfecting new approaches, such as combining two techniques in a single experiment. At the APS, for example, Sinha and his colleagues have plans to combine phase-contrast imaging and scattering: "You would like to use phase-contrast imaging to look at a crack propagating in a material. And around the crack you would use scattering measurements to measure the strain distributions."

In some cases, new techniques are emerging from this bustle of innovation faster than the demand for them. For example, Riekel cites a new technique in which the fluorescence of atoms induced by x-rays reveals the presence of trace compounds at a sensitivity 100 times greater than existing methods. Says Riekel, "We still have to look for applications. It is so sensitive that you have to go out and find the clients for this."

–Alexander Hellemans

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THIRD-GENERATION HARD X-RAY SYNCHROTRON SOURCES

Source	Location	Opened	Maximum Electron Energy (GeV)	Planned Beamlines
European Synchrotron Radiation Facility	Grenoble, France	1994	6.0	52
Advanced Photon Source	Argonne, United States	1996	7.0	70
Spring-8	Nishi-Harima, Japan	1997	8.0	61