How to Do EXAFS in Your Own Laboratory

New x-ray systems let researchers do experiments that once were impractical without traveling to a synchrotron radiation center

There is encouraging news for proponents of "small is beautiful" in scientific research. Investigators are showing that it is possible to carry out Extended X-ray Absorption Fine Structure (EXAFS) experiments in a few hours or less using commercially available equipment that can be assembled into an EXAFS spectroscopy system in their own laboratories. Up to now, those unwilling to wait the days or weeks necessary to accumulate enough data from conventional x-ray equipment for a high-quality spectrum have had to travel to one of the few synchrotron radiation centers in the world capable of producing high fluxes of x-rays. For American researchers, this has usually meant a trip to California and the Stanford Synchrotron Radiation Laboratory. At Stanford, would-be EXAFSers have had to queue up for their turn, record as much data as they could as quickly as possible, and return home for an extended period of analysis.

The new home-grown EXAFS systems by no means put synchrotron radiation centers out of business. For most forefront-of-the-field studies, especially those involving very dilute samples with concomitant weak signals, there will be no substitute for the high-intensity x-rays from synchrotron radiation. And the even higher intensities projected for the new facilities coming into operation soon at Stanford and at Brookhaven National Laboratory in a couple of years will open the way to even more uses for this wondrous light source.

But EXAFS, which allows researchers to determine the local atomic structure of a host of materials—crystalline or amorphous; solid, liquid, or gas—has become so popular with the advent of synchrotron radiation facilities, first in the mid-1960's but primarily in the 1970's, that the potential number of practitioners has already outstripped the supply of x-rays. Moreover, many researchers are willing to forego the advantages of synchrotron radiation for the convenience of working in their own laboratories, where experiments can be rerun or modified at will. One veteran user of synchrotron radiation, Edward Stern of the University of Washington, is setting up a home EXAFS system, although by no means giving up the former. The problem of doing proprietary research at federally supported research facilities also continues to poke its head up from time to time. The ability to do EXAFS in the laboratory is thus of interest to industry, and a number of companies are said to be setting up EXAFS systems.

Researchers in groups headed by the late Nigel Shevchik of the State University of New York at Stony Brook and by Gordon Knapp at Argonne National Laboratory were among the first to appreciate the possibilities for a home EXAFS system. (Gabrielle Cohen has taken over Shevchik's EXAFS activities at Stony Brook.) Both groups have assembled working systems and are beginning to use them in their research programs.

The key feature in the new home systems is the rediscovery of the old technique of using a curved crystal to simultaneously focus the x-ray beam and monochromatize it (disperse the beam into its constituent wavelengths). The use of a curved crystal in place of the much more commonly found flat crystal enhances the intensity of the x-ray beam by a factor of about 100. This feature alone makes EXAFS experiments practical, says Joe Georgopoulis of Argonne.

A second helpful component of the systems is a 10 to 20 kilowatt rotating anode x-ray source. What rotates is the metal target that produces x-rays when struck by an electron beam. Because the same part of the metal is not constantly bombarded, it does not get as hot as a fixed target and a higher-intensity electron beam can be used to generate more x-rays without melting the target. An unsolved problem, however, is that the curved crystal also passes x-rays with wavelengths equal to multiples of those being dispersed—higher harmonics. At present this is dealt with by operating the sources at a lower power, which means a lower x-ray intensity.

One difficulty with doing EXAFS with x-rays from a tube is that the x-ray spectrum is not smooth but rather it is a mixture of a continuous bremsstrahlung spectrum and a discrete set of characteristic lines that vary with the metal target. The problem in EXAFS, which is a spectroscopic technique and therefore works best when the source spectrum is smooth, is how to accommodate the rapid changes in source intensity when scanning near a characteristic line. Both groups rely on electronics to accomplish this. Knapp says, for example, that the Argonne system works sufficiently well that any discontinuities or glitches resulting from this procedure are at most comparable to the 0.1 percent noise level in the EXAFS spectrum.

What does it cost to assemble such a system? Cohen estimates that to duplicate the Stony Brook setup would take from $165,000 to $195,000 by the time a rotating anode source, a minicomputer, a multichannel analyzer and other electronics, and other assorted hardware were paid for. But the cost, which is comparable to that of much of today's computerized instrumentation, is worth it. At Stony Brook, says Cohen, spectra on concentrated materials can be accumulated in a half hour, while dilute samples take proportionately longer. The energy resolution is not as good as that possible with synchrotron radiation, which limits the ability to study the positions of atoms other than those nearest to the absorbing species.

The Argonne group is already using its system to study the damage to the crystal structure of superconductors caused by high-energy neutrons, a problem related to magnetic confinement fusion reactors. This project would be hard to do at Stanford because of the need to irradiate samples immediately before taking data. Similarly, Stern's group at Washington is setting up to study gases absorbed on solid substrates. Because the gas can take several hours—even days—to reach equilibrium, it would be a difficult project at Stanford where time is at a premium. Meanwhile, the Stony Brook group wants, with the aid of a special high-sensitivity detector invented by Shevchik and Daniel Fischer, to study oxidation and corrosion of metal and alloy surfaces. Surfaces are tricky to look at with EXAFS because the "noise" caused by atoms beneath the top layers can overwhelm the surface signal.

All in all, home EXAFS systems seem to have a bright future. If further improvements are forthcoming and if they should catch on, funding officers in federal agencies may find themselves having to make some hard decisions when confronted with proposals for equipment for both synchrotron radiation and home EXAFS.—ARTHUR L. ROBINSON

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