

CASTING LIGHT ON MATERIAL STRUCTURES

X-rays and light that once represented lost energy are proving themselves an unparalleled research tool

BY DIETRICK E. THOMSEN

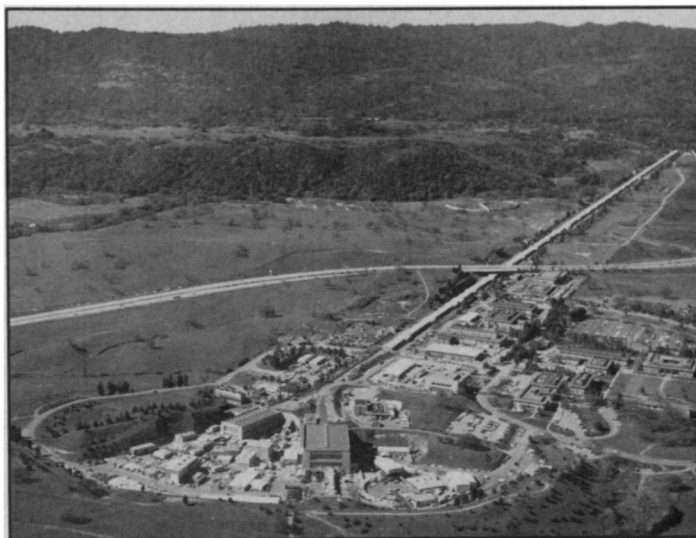
Synchrotron radiation is generated whenever electrically charged particles moving at speeds near that of light are forced into bent paths by magnetic fields. It is electromagnetic radiation, which can be visible light, ultraviolet or X-rays, depending on the energy of the particles and the strength of the magnetic field. The radiation is named for the class of accelerators in which it first became a serious problem, electron synchrotrons.

Because electrons have less than 1/1800th as much mass as protons, they reach relativistic velocities at much lower energies than do protons. At high energies synchrotron radiation robs so much energy from electrons moving in circular paths that when what is now the world's most energetic electron accelerator—the 20-billion-electron-volt machine of the Stanford Linear Accelerator Center—was planned, the builders decided to make it a straight line two miles long. It may be considered somewhat ironic that that linear accelerator now supplies accelerated electrons to a facility that uses synchrotron radiation for research in several different scientific fields.

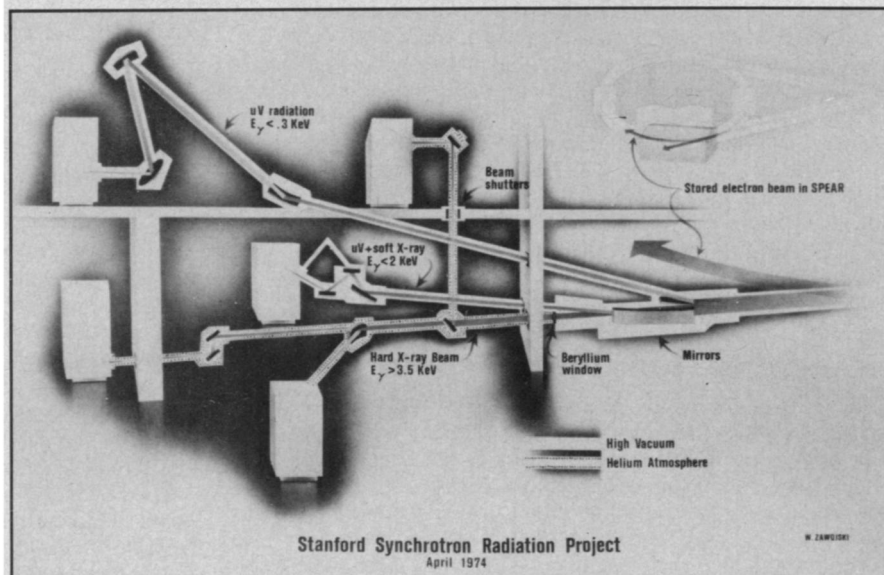
Synchrotron radiation used to be considered a dead loss by accelerator operators. In recent years it has suddenly become a very important new research field. In the words of Herman Winick, deputy director of the Stanford Synchrotron Radiation Laboratory, "There is an explosive growth of interest in its applications."

At the moment there are about 10 storage rings and 11 synchrotrons around the world at which synchrotron radiation experiments are done. The SSRL gets its synchrotron radiation from the SPEAR storage ring. A smaller storage ring at the University of Wisconsin, the Deutsches Elektronen-Synchrotron at Hamburg, and more than one ring at Novosibirsk in the USSR are among those in the world now supplying synchrotron radiation for experiments.

The attitude of the U.S. scientific establishment and its government funders has undergone a total "turnaround from three or four years ago," Winick says. At that time Winick was working at the now defunct Cambridge Electron Accelerator in



The two-mile linear accelerator feeds electrons to the SPEAR storage ring (lower right of top picture), where they generate synchrotron radiation used by the SSRL (building at right of ring). Mirrors permit several experiments to share one radiation beam (bottom).



Stanford Univ.

Cambridge, Mass. An attempt to get \$1 million a year to keep the CEA going as a facility for synchrotron radiation was unsuccessful. Over the next three years, the National Science Foundation plans to give about \$7 million to the SSRL alone. At the same time, plans have been announced for enlargement of the facility in Wisconsin and for another national synchrotron radiation facility at Brookhaven National Laboratory, where an accelerator will be built to be solely a source of synchrotron radiation.

Other sources of X-rays, of which the best are 60-kilovolt rotating anode tubes, do not provide the broad spectrum or high power of synchrotron radiation. As an example of the difference, Winick cites a

group of scientists from the Bell Telephone Laboratories who came to SSRL to do a spectrum that they had previously done by other methods. With synchrotron radiation it took them 20 minutes to do a spectrum that had previously taken two weeks. Another advantage of synchrotron radiation is that it comes in bursts that correspond to the bunches of electrons circulating in the storage ring. This gives experiments a built-in time resolution and makes possible the study of chemical and biological processes over time. A study of contracting muscle fibers is one possible experiment. Other biological possibilities include studies of membrane action, which are a particular interest of Sebastian Doniach, who has just completed his

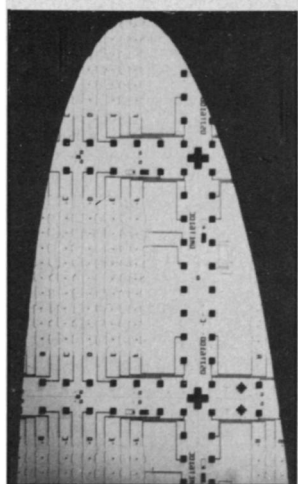
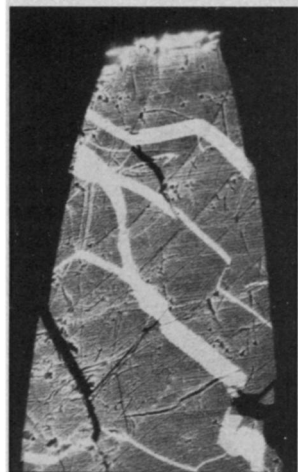
tenure as director of SSRL and will return after a leave as a consulting director. In the spring, Arthur I. Bienenstock will become director.

The broad spectrum and high power of synchrotron radiation make possible the development of a technique called EXAFS (Extended X-ray Absorption Fine Structure) in which the energy of the X-rays striking an experimental sample is gradually varied over the spectrum available to see exactly what energies it absorbs and what that tells about its structure. The variation is accomplished by inserting a monochromator into the radiation. The monochromators available can select energies with differences as fine as one volt in 10,000. They are expensive, and some of them have been built by private firms whose scientists wished to use the synchrotron radiation.

EXAFS can be used to study the geometry of complicated chemical compounds and processes. An example is the oxidation of hemoglobin. There are four iron atoms in a hemoglobin molecule. It is to these that oxygen atoms bind. Finding out what happens as the oxygen is bound to the iron atoms may help elucidate how and why it happens. With EXAFS the geometry of the hemoglobin molecule in the neighborhood around the iron atoms can be studied, and any changes it undergoes during oxygenation can be found out. This is done by irradiating the hemoglobin with X-rays of gradually changing energy until the point is reached at which electrons in the K shell of the iron atom begin to absorb the X-rays. This absorption produces a photoelectric effect: The energy gained allows electrons to jump out of the atom. It is a principle of quantum mechanics that a moving electron is the equivalent of a wave. As this electron wave proceeds out of the iron atom, it encounters the neighboring atoms and is partially reflected by them. The backscattered wave interferes with the outgoing wave, and the interference changes the probability that the iron atom will absorb energy from the incoming X-rays. To put it briefly, the environment of the iron atom changes the iron atom's ability to absorb X-rays, and by studying the changes in X-ray absorption, scientists can figure out the geometry of the environment.

The four iron atoms in hemoglobin lie in a plane. One of the questions was whether the plane is altered by oxygenation: Do the iron atoms move out of the plane when oxygen binds to them? The answer, as determined by EXAFS, is no. This, says Winick, is a very important result that has forced changes in the theoretical understanding of hemoglobin.

In a similar way, EXAFS can be used to study a large number of catalytic and enzymatic reactions. In vitamin B17 a cobalt atom sits in a plane with four nitrogen atoms. Biophysicists and biochemists are interested in finding out how the cobalt moves around as vitamin B17 reacts with



William Parrish/IBM Research Lab, San Jose

X-ray diffraction pattern produced by reflection from a nearly perfect gadolinium garnet wafer perpendicular to the X-ray beam (top). Enlargement of a single spot (elongated because of grazing incident reflection) from the image of a gadolinium garnet wafer on which an epitaxial film was grown and a metallurgical circuit pattern deposited (middle). Enlargement of a single spot from lithium fluoride crystal showing imperfections (bottom).

other chemicals. A very important enzyme is nitrogenase, which is responsible for the uptake of nitrogen by the roots of plants. Nitrogenase has a molecular weight of about 2,000. In all that mass there are two molybdenum atoms. EXAFS can zero in on the molybdenum atoms to try to find out what their behavior may contribute to the nitrogen-fixing reaction.

Another wide field of application for EXAFS and synchrotron ultraviolet is the physics and chemistry of surfaces. The technique can be used to find surface imperfections in solids and to study what happens as layers of other substances are adsorbed on a surface or react chemically with it. EXAFS can concentrate its attention on the surface better than can other tech-

niques. Using the example of oxygen layers on a catalyst, Doniach points out that with seven layers of oxygen on a surface, the X-ray penetration can be varied to within an atomic layer or two.

Knowledge of surface reactions is essential to the understanding and manufacture of most modern electronic circuit components. Indeed, a direct practical use of synchrotron radiation might be in the manufacture of integrated circuits. An apparatus for manufacturing them was actually built in Japan, and there has been some thought of it in the United States, but it seems that the method is not economically superior to more conventional ones.

Another direct use of synchrotron X-rays may ultimately be in medical diagnostics. Since the wavelength of synchrotron X-rays can be controlled, a given picture could be made with less total dosage than is now usual. Alternately, if the total dosage is kept the same, the fact that it all comes at one wavelength instead of over a fairly wide band would make it possible to bring out differences in contrast too subtle for present techniques.

About 100 scientists from all over have participated in the experiments done at SSRL up to now. Although new experimental areas are being added, the synchrotron radiation work is hindered somewhat by being parasitical on the particle physics work that is the primary purpose of the SPEAR storage ring. That particle physics work earned a Nobel prize for Burton Richter. The SSRL people are happy that he won it, but they wish he had won it at higher energy.

The SPEAR storage ring can handle electrons with energies up to about 4 billion electron-volts. The interesting particle physics (the charmed particles and their relatives) happened at about 1.6 billion electron-volts. This means that the storage ring is not run at its highest energies as often as it might be, and it is the higher energies that produce synchrotron radiation at the energies that experimenters at SSRL prefer. To do the study of molybdenum in nitrogenase, mentioned above, X-rays around 20 kilovolts energy are needed. To get a decent amount of flux at 20 kilovolts requires running the storage ring at 3.5 billion electron-volts, and it doesn't often run there. If SPEAR belonged entirely to SSRL, they would run it at higher energies more of the time and they would insert "wigglers," magnets intended to force sharp turns in the electron path and enhance the spectrum of synchrotron radiation.

In a few years those things may come to pass. The particle physicists are now building a new storage ring, the Positron-Electron Project (PEP), which will handle electrons up to an energy four or five times the maximum in SPEAR. At that time the interest of the particle physicists is expected to shift to the higher energies, and the plan is to turn SPEAR over completely to SSRL. □