New Synchrotrons Light Up Microstructure of Earth

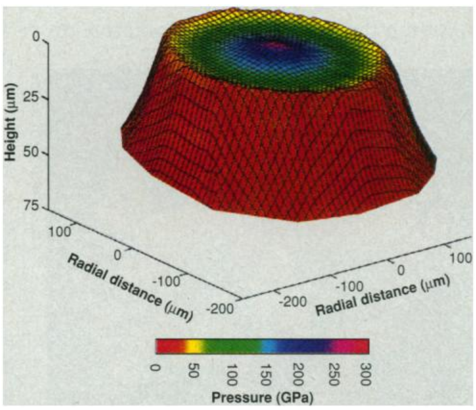
For geophysicists, "to be able to reproduce the conditions of Earth's interior, from the crust down to the core, [is] something of a Holy Grail," says Ho-kwang Mao of the Geophysical Laboratory of the Carnegie Institution of Washington. In pursuit of it, researchers like Mao take tiny samples of material typical of Earth's interior—such as the silicates and oxides of the upper mantle or the iron of the core—squeeze it to huge pressures between the tips of two diamonds, and heat it to a few thousand degrees. But mimicking the deep Earth is of no use unless you can see how your sample is behaving. So geophysicists have been adding a new stop on their grail quest: a pilgrimage to a third-generation synchrotron.

Work with second-generation machines has already shown that scattering x-rays off high-pressure samples can reveal how the crystal structure of minerals in the deep Earth changes under pressure. The new synchrotrons, with their higher intensities, are providing sharper pictures of structure and properties, and their finer beams are allowing researchers to probe the crumb-sized samples required for ultrahigh-pressure experiments. "It's a very exciting time at the moment. … The new facilities are very important," says geochemist Surendra Saxena of Uppsala University in Sweden.

Geoscientists are not the only ones noticing the possibilities. Other researchers are compressing high-temperature superconductors to try to gain some clues to how they work. Still others are putting the squeeze on the soccer-ball–shaped carbon molecules called fullerenes to see how their properties and structure change at high pressure. Work at the new machines is also illuminating the interiors of planets other than Earth by probing how hydrogen—which makes up the core of Jupiter and Saturn—behaves at enormous pressures, where it may become metallic, or even a superconductor.

Perhaps the most contentious area at the moment is the study of iron. Earth's core is the driving force behind all the processes of the planet's interior, so exactly what form iron takes in the core will have a fundamental impact on geophysical models. "Iron will change the whole thing," says Denis Andrault of the University of Paris. To study iron's properties under such extreme conditions, researchers squeeze a sample to enormous pressures between two flawless diamonds in a device called a diamond-anvil cell, then heat it with a laser. By watching how x-rays are scattered as they pass through the diamonds and the sample trapped between them, researchers can gather clues to how the atoms are arranged in the sample.

In 1993, a team led by Saxena squeezed a sample of iron to about 38 gigapascals (GPa)—hundreds of thousands of times atmospheric pressure—while heating it to between 1200 and 1500 kelvin. They detected signs that under those conditions the iron is no longer in its normal high-pressure structure, known as hexagonal close-packed (hcp), in which atoms are arranged like racked billiard balls, with the balls in each layer lined up with those above and below. Since then, several teams have been probing iron at second-generation synchrotron sources in attempts to identify the new structure. Saxena believes it to be an arrangement called polytype double-layer hcp, or dhcp—in which each layer is slightly out of step with its neighbors. But he admits that "it's still an open question what happens at high temperature. Most agree there is a phase change, but some groups believe it is a different structure."



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The big squeeze. Colors indicate the pressures acting on the tip of a diamond-anvil cell.

The third-generation machines could help settle the matter by sweeping aside some of the obstacles earlier studies faced. One stems from the fact that the laser spot that heats the sample is roughly the same size as the x-ray beam. To avoid contaminating the x-ray scattering data with sample areas at the edge of the laser spot that are not at the full temperature, researchers must narrow down the beam. Doing this at a second-generation source reduces the intensity so much that analysis becomes too slow to maintain temperature stability. "You can barely do [this technique] at second-generation sources," says Mao. Third-generation machines speed up the analysis considerably.

The new machines also allow researchers to get a more direct view of crystal structure. Traditional high-pressure studies have used a technique called energy-dispersive x-ray diffraction, in which a beam with a range of photon energies is fired at the sample. A detector positioned on the far side of the sample, at a fixed angle from the beam axis, collects a diffraction spectrum, showing how many photons are scattered to that angle at each energy. The spectrum provides key lattice parameters of the crystal, such as the size of its smallest repeating unit, but not its structure.

The new synchrotrons can produce a beam of a single energy that is intense enough to do angle-dispersive diffraction, which produces an actual diffraction pattern on a flat image plate behind the sample. From that, researchers can discern structure as well as lattice parameters. "With energy-dispersive diffraction, hcp and dhcp are hard to distinguish," says Saxena. "Angle-dispersive diffraction can resolve low-intensity peaks. It makes life so different."

The same goes for researchers doing high-pressure studies of hydrogen. To search for a metallic phase of hydrogen, researchers had to squeeze it to even higher pressures than those in the iron studies. "We needed to go to 100 GPa," says Mao. The amount of force a diamond anvil can apply has a limit, but because pressure is force divided by area, researchers can approach the required pressures by making the sample smaller—on the order of a few tens of micrometers. That is too small to study with the comparatively coarse beams of second-generation machines. "At high pressure, the sample gets small, too small for ordinary synchrotrons," says high-pressure specialist Michael Hanfland of the European Synchrotron Radiation Facility (ESRF). "The hydrogen stuff couldn't be done before at these pressures."

The 30-micrometer beams of the ESRF have changed all that. When ESRF opened for business in 1994, hydrogen studies were a high priority, and the sense of urgency grew last year with reports that shock-wave experiments on liquid hydrogen had revealed hints of a metallic phase. A team of researchers from the Geophysical Laboratory and the University of Paris reported in *Nature* last October that they had succeeded in compressing solid hydrogen to more than 100 GPa. The results included the finding that hydrogen is much softer than predicted by theory, but "we did not see metallization," Mao says.

Mao, like many high-pressure researchers who spoke with *Science*, thinks the best is yet to come. "We're still learning with the third-generation sources," he says. "It's all happening just now," says Saxena. "We feel like explorers."

–Daniel Clery