Cornell researchers set record for smallest hard X-ray beam

Donald Bilderback, Stephen Hoffman, and Daniel Thiel

Cornell staff members Donald Bilderback, Stephen Hoffman, and Daniel Thiel have recently developed tapered glass capillaries that compress x-rays down to a size less than one-thousandth the diameter of a human hair—the smallest beam diameter ever achieved for hard (high-energy) x-rays. X-rays confined on such a small scale have yielded the highest resolution image ever obtained with hard x-rays, with an observed spatial resolution of 50 nanometers.

These beams can be used for the characterization of materials at an unprecedented resolution. Glass capillaries offer significant advantages in x-ray microdiffraction studies because they can concentrate wide-band polychromatic as well as monochromatic x-rays. At this time, tapered capillaries are the only x-ray optical component producing nanometer spatial resolution with high-energy x-rays. Other components such as phase plate pinholes, Fresnel zone plates, and Kirkpatrick-Baez mirrors are producing focused, nearly monochromatic x-ray beams on a length scale 10 to 100 times larger.

The lead-filled glass capillary takes the x-ray beam generated at CHESS and compresses it because the glass is tapered to a fine point. The glass tube, which can be made to a variety of diameters, acts as a funnel for the beam of x-rays, successively bouncing x-rays off the smooth inside wall by total reflection as they pass down the length of the tube. The beam leaving the capillary is smallest in size right at the tip and has a divergence of a few milliarcmin.

Virtually all of the traditional methods of studying the nature of a sample with x-rays such as x-ray dif-

(Above) From left: Stephen Hoffman, Daniel Thiel, and Donald Bilderback with their glass capillary at Wilson Lab.

(Photo: Peter Movius/Cornell University Photography)
fraction (monochromatic and Laue, wide- and small-angle scattering, etc.), elemental fluorescence mapping, near and extended edge absorption spectroscopy, tomography, radiographic imaging, etc., are usable with tapered capillaries. Recent experiments in imaging and Laue diffraction serve to illustrate how capillaries can be applied to the emerging field of x-ray microscopy.

A record-setting spatial resolution of 50 nm was observed in an imaging experiment on the CHESS-B2 bending magnet station (E = 10 keV, E = 10 keV, E = 8.5 keV) with a beam of 100 nm thick gold stripe pattern prepared on a silicon substrate. Figure 1 shows the conceptual outline of the imaging experiment. A flat total-reflection mirror upstream of the capillary (not shown) set an upper energy cutoff of 8 keV. The lower end of the beryllium mirror was approximated to an energy of 8 keV and was determined by the length of the air path and the thickness of the beryllium windows on the beamline. The contrast in the image (Figure 2) was obtained from x-rays being absorbed by an extra 4% as the beam was scanned over the 100 nm thick gold portion of the sample. This demonstration experiment was done under very special conditions. The beam was intensified in flux (x-rays/sec/m²) by a factor of 100 over the incident beam. We have observed gains as high as 950 in other experiments.

An additional experiment was also performed to show that microfocus diffraction could be achieved with 1 nm beams and larger volume gold crystals. The 0.1 mm thick gold was chosen for the monochromatic rotation method for simplicity. The exposure time is 100 to 1000 times longer than for the monochromatic rotation method and the excitation of the crystal is reduced during exposure.

Figure 3 shows a Laue x-ray diffraction image taken with a 0.1 mm diameter beam, about 1/20th the diameter (and 1/40th the area) of the 100 micron diameter collimator typically used for small protein single crystals. Obviously, smaller diameter beams may be employed, but the lower bound in size will probably be determined by the x-ray radiation resistance of the sample and the x-ray size of the sample. In a separate study, radiation insensitive single crystal fibers of silicon and gold were examined. They were able to observe Laue diffraction from a 0.1 mm thick gold single crystal with a 100 nm diameter beam. In contrast to the perfect round spot shapes observed with a perfect 0.1 mm thick silicon wafer with the same capillary, the gold Laue spots were radially streaked, consistent with a mosaic spread of a few degrees. The streaking was observed with larger diameter beams up to 100 microns in size. We conclude that the dimensions of the mosaic crystal domains were less than the beam size of 300 nm.

In the future, we hope to fabricate optimally-fractioned tubes which should come close to achieving the higher gains that are theoretically predicted. It is also possible, in principle, to make smaller beams, down to perhaps 200 nm, at which size the skin depth of x-rays penetrating into the glass turns out to be the fundamentally limiting parameter.

The capillary technology described here has received an R&D 100 Award from Popular Science Magazine for one of the year's most significant products in 1999. The individuals involved in this effort are Donald Biler, Howard Gold, and Daniel Thiel of Cornell University; Aaron Levine of Hofstra University, Jericho; and Edward Stern of the University of Washington. The lithography work was performed at the National Nanofabrication Facility at Cornell University. The work was supported by the National Science
Foundation and the National Institutes of Health.

Call for Support of Microscience X-ray Station at CHESS.

The microscopy group at Cornell is interested in new applications of microbeams to science. For instance, we plan to look at fibers of a few microns in diameter in the near future using wide angle X-ray scattering to determine the structural differences between the skin and core of composite fibers. We also plan to map the strain, microstructure, and composition of tiny polycrystalline crystals using high spatial resolution with microdiffraction and microtomography measurements. Many biological experiments are also in the conceptual stage.

We are in the process of raising money to support equipment cost in CHSS beamlines for these kinds of investigations. A limited amount of work is being supported from the CHESS and MacCHESS organizations, but we need to generate additional support in the Material Science community. We are asking for help in building a specialized beamline over a several-year period of time, including a person to help construct and oversee collaborative experiments (miniature microtomes, translation stages, fluorescence detectors, tiny 2-D CCD detectors, etc.). We need to raise about $200,000 to $400,000 to adequately fund these efforts. Additionally, we would like to attract visiting scientists to come and join us in these efforts for some period of time. If you are interested in contributing in time, effort, or even funding for these activities, please contact Don Biberbach by phone at 607-255-0915 or via Internet at cberry@cornell.edu.

(Figure 5) Laser diffraction from a monocrystal taken with a 0.8-inch diameter X-ray beam on Kodak DEF film with a spectrum extending from 2 to 12 keV. The room temperature sample was translated 18 microns every 24 seconds during the 300-second total exposure to minimize the radiation damage to the sample. The crystal diffraction at 2.2 keV resolution. The divergence of the x-ray beam was 0.60 microns measured on the x-ray film. This important shows the possibility of using tetramer crystals in the study of biological materials that do not form the larger (of order 100 microns) size needed for conventional x-ray crystallography.

* Presently at Northwestern University.

6. A 8.4% neutron beam at 15 keV fabricated has been used at the ESRF Grenoble from a Rigaku detector built by A. Sugita and

Y. Asano, private communication from C. Reid, 1986.
7. I. B. 2800111-0411, Center for X-ray Optics - 1990-4-11980.

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