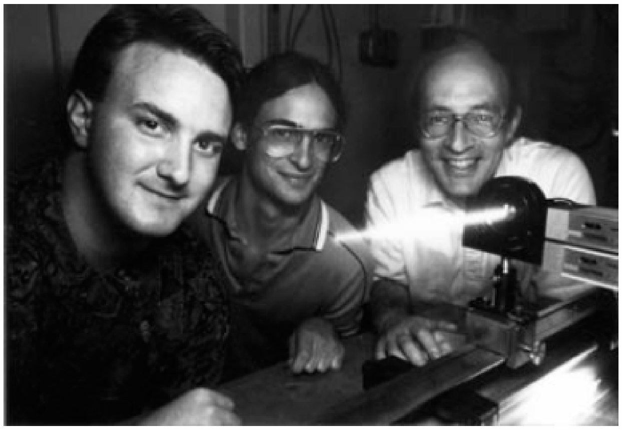
Cornell researchers set record for smallest hard X-ray beam

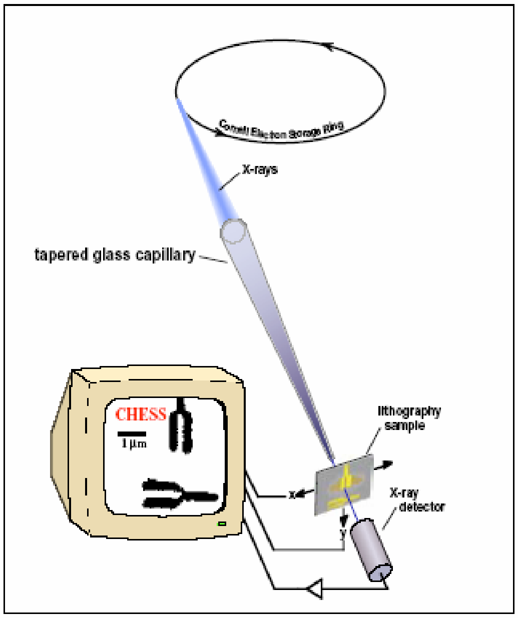
Donald Bilderback, Stephen Hoffman, and Daniel Thiel*

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[1] of 50 nanometers.

These beams can be used for the characterization of materials at an unprecedented resolution. Glass capillaries[2,3,4] 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 zone plates[5], Bragg-Fresnel lenses[6], and Kirkpatrick-Baez mirrors[7] are producing focused, nearly monochromatic x-ray beams on a length scale 10 to 100 times larger.



(Above) From left, Stephen Hoffman, Daniel Thiel and Donald Bilderback, with their glass capillary at Wilson Lab. (Photo: Peter Morenus/Cornell University Photography)



(Left, Figure 1) a). Conceptual view of the imaging experiment. Wide bandwidth synchrotron radiation from a CHESS bending magnet is first squeezed into a small size by a tapered capillary, then directed onto a lithographically prepared test sample consisting of a 100 nm thick gold pattern deposited on a 200 nm thick silicon nitride substrate. The absorption of x-rays in the thin gold layer produces the contrast seen as the specimen is scanned in the x-y plane. (Drawing by Chris Staffa)

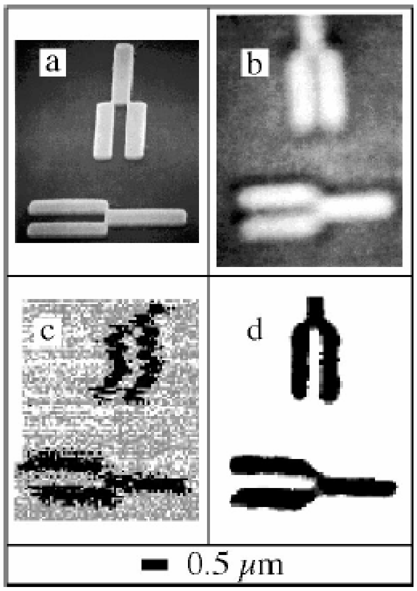
The leaded glass capillary takes the x-ray beam generated at CHESS and compresses it because the glass is tapered to a fine point. The glass tubes, which can be made to a variety of diameters, act as a funnel for the beam of x-rays, successively bouncing x-rays off the smooth inside wall by total reflection 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 milliradians.

Virtually all of the traditional methods of studying the nature of a sample with x-rays such as x-ray diffraction (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 microscience.

A record-setting spatial resolution of 50 nm was observed in an imaging experiment on the CHESS B2 bending magnet station (E_{crit}=10keV, E=5.3GeV, I=80mA) with a test sample placed a few microns away from the tip. The 4 cm long monocapillary was drawn by Dan Thiel. The sample consisted of a 100 nm thick gold stripe pattern prepared on a silicon nitride wafer 200 nm thick. 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 bend magnet spectrum was approximately 5 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 far from optimal conditions. The beam was intensified in flux (x-rays/sec/mm^2) by only a factor of 50 over the incident beam. We have observed gains as high as 960 in other pipettes[8].

A series of Laue diffraction experiments were also performed to show that microbeam diffraction could be achieved with these tiny beams and vanishingly small amounts of crystalline material. The wide-bandwidth Laue method was chosen over the monochromatic rotation method for its simplicity. The exposure time is 100 to 1000 times faster than for the monochromatic rotation method and no oscillation of the crystal is needed during exposure. Figure 3 shows a lysozyme diffraction image taken with a 5.6 micron diameter beam, about 1/20th the diameter (and 1/400th the area) of the 100 micron diameter collimators 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/or the unit cell size.



(Figure 2) a) The line widths of the features are 300 nm wide in as seen in scanning electron micrograph. b) Optical image obtained with a visible-light microscope with a numerical aperture of 0.9. The image is fuzzy because the structure is below the resolving power of the light microscope. c) Unprocessed x-ray absorption image. The image was formed from a two-dimensional scan with piezoelectric crystals consisting of 50 nm by 50 nm pixels. d) X-ray image after processing. The data in each row were horizontally shifted to compensate for the effects of spatial drift (thermal, air currents, electronic, etc.). A median processor was then applied, which averages all pixel intensities located in a circle of radius of 2 pixels.

In a separate study, radiation insensitive single crystal films of silicon and gold were examined. We were able to observe Laue diffraction from a 50 nm thick gold single crystal with a 300 nm diameter beam. In contrast to the perfect round spot shapes observed with a perfect 2 micron thick silicon wafer with the same capillary, the gold Laue spots were radially streaked, consistent with a mosaic spread of a few degrees. The same streaking was observed with larger diameter beams up to 45 microns in size. We conclude that the dimensions of the mosaic crystal domains were less than our beam size of 300 nm.

In the future, we hope to fabricate optimally-figured tubes which should come closer to achieving the higher gains that are theoretically predicted. It is also possible, in principle, to make smaller beams, down to perhaps 2 to 10 nm size, at which size the skin depth of x-rays penetrating into the glass turns out to be the fundamentally limiting parameter.

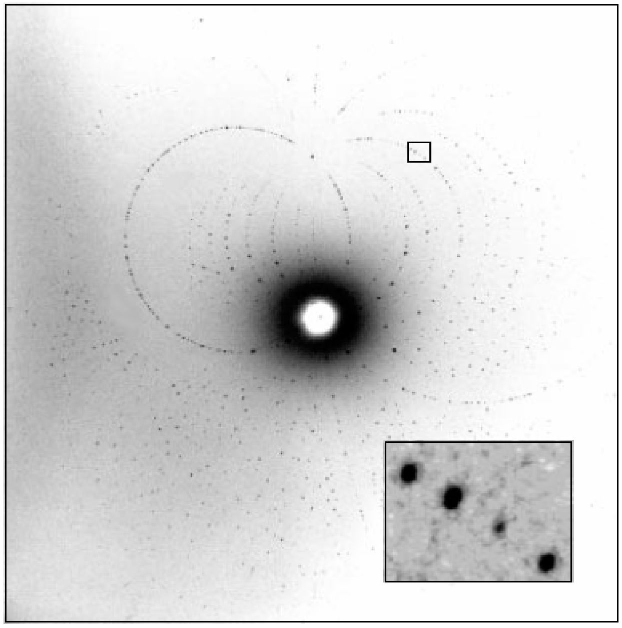
The capillary technology described here has received an R&D 100 Award from *R&D Magazine* for one of the year's most significant products in 1993. The individuals involved in this effort are Donald Bilderback; Stephen Hoffman, and Daniel Thiel of Cornell University; Aaron Lewis of Hebrew University, Jerusalem; 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 microscience 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 microfluorescence measurements. Many biological experiments are also in the conceptual stage.

We are in the process of raising funding for specific equipment to outfit a CHESS beamline 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 seeking funding to build a specialized beamline over a several year period of time, including a person to help construct and oversee collaborative experiments (miniature monochromators, 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 Bilderback by phone at 607-255-0916 or via Internet at dhb2@cornell.edu.



(Figure 3) Laue diffraction from a lysozyme crystal taken with a 5.6 micron diameter x-ray beam on Kodak DEF film with a spectrum extending from 5 to 25 keV. The room temperature sample was translated 3 microns every 24 seconds during the 300 second total exposure to minimize the radiation damage to the sample. The crystal diffracted to 2.2 Å resolution. The divergence of the microbeam was 2.6 mrad as determined by the spot size measured on the x-ray film. This experiment shows the possibility of using tapered capillaries in the study of biological materials that do not form the large (of order 100 micron) size needed for conventional x-ray crystallography.

* Presently at Northwestern University

1. D. H. Bilderback, S. A. Hoffman, D. J. Thiel, *Science* 263, 201 (1994).
2. E. A. Stern, Z. Kalman, A. Lewis, K. Lieberman, *Appl. Opt.* 27, 5136 (1988).
3. P. Engström *et al*, *Nucl. Instrum. Meth.* A302, 547 (1991).
4. D. J. Thiel, D. H. Bilderback, A. Lewis, E. A. Stern, *Nucl. Instrum. Meth.* A317, 597 (1992).
5. W. B. Yun *et al*, Proc. SPIE 1740, 117 (1993).
6. A 0.8 micron beam at 15 keV (submitted) has been made at the ESRF, Grenoble from a Bragg Fresnel lens designed by A. Snigirv and V. Aristov, private communication from C. Riekel, 1993.
7. LBL-28001 UC-411, *Center For X-ray Optics - 1989*,4-15(1990).
8. Applications of Single Tapered Glass Capillaries: Submicron X-ray Imaging and Laue Diffraction, S. A. Hoffman, D. J. Thiel, and D. H. Bilderback, *Optical Engineering* 33, 303 (1994).

NOTE: A version of this article will be published in *Synchrotron Radiation News*, May/June 1994.