

A new computerized capillary puller for hard x-ray applications

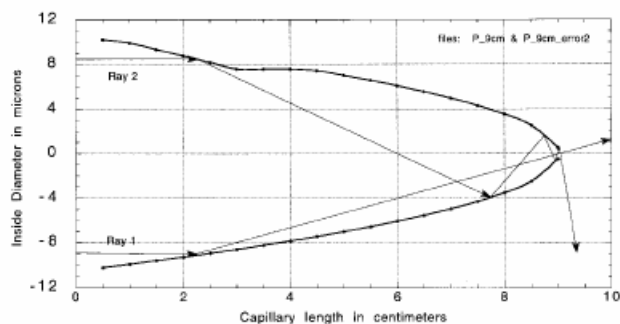
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In the 1994 CHESS Newsletter, we reported on our first experiments using leaded glass monocapillaries to make hard x-ray beams with a size ranging from 50 nanometers up to 5 microns [1]. Rather than continue to work with capillaries that are far from optimal, we decided to try improving the optics by employing design tools and constructing a computerized glass puller that can make better engineered and reproducible parts. We have also been trying to fabricate capillaries from borosilicate material that is lead-free, an important issue for some x-ray experiments using x-ray fluorescence, spectroscopy methods, or Laue diffraction. Below we discuss the results we have achieved in making very straight, nearly parabolic shaped capillaries as well as the problems we are experiencing to obtain borosilicate glass tubing with an inner surface smooth enough to show a high x-ray intensity gain.

Design Progress

X-rays passing through the capillary must remain below the critical angle for efficient operation as they are compressed to smaller and smaller cross section before exiting the tiny tip. Capillary tube design starts with specifying an upper x-ray energy at which the optic will function (e.g. 12 keV), the desired exit beam size, and the maximum exit beam divergence. Given these parameters and the properties of the x-ray source, a suitable design can be made with a two-dimensional ray tracing program RATB [2]. This program can take glass parameters and compute the reflectivity at hard x-ray wavelengths, but as of yet has no soft x-ray capability.

For synchrotron applications, we have found that parabolic and elliptical tapered capillaries show high gain when ray traced. We recently have employed the same tools to determine the tolerance on slope errors, i.e. the local deviation from the design figure that will be present in any real optic. Figure 1 shows that for a ray originating from an on-axis source point (Ray 1) and a parabolic taper, only one reflection from the inner



wall is needed to steer the beam through the tip opening. If a 50 microradian error in the slope is artificially added in the same region where this beam is reflected, then 5 bounces (Ray 2) are required, but the beam does still successfully exit the capillary. However, increasing this error to 100 microradians steepens the angle of the beam relative to the glass until the critical angle is exceeded and so this situation shows a net reflection efficiency of 0.1%. We estimate that good performance will be obtained with slope errors of up to 1/10th of the 50 microradian slope error found for Figure 1 so that an acceptable tolerance for the drawing process in the glass puller is around ± 5 microradians.

Of course the important question is: Once we design a capillary optic on paper, will we then be able to successfully fabricate it?

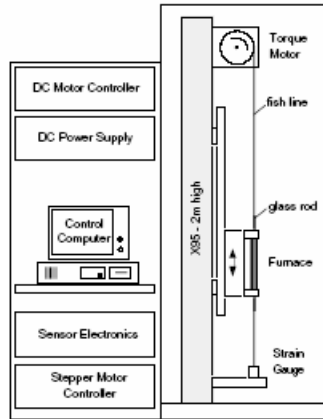
Building a Computerized Puller

The first method we used to obtain glass capillaries was the "luck of the draw" method. In this method, we would hang a glass tube in a cylindrical furnace with a 50 gram weight at the pulling end. By raising the temperature of the furnace above the softening point of the glass, gravity acting on the weight would begin to elongate the capillary until eventually a needlelike optic would form as the two parts separated. Such capillaries were somewhat irregular in shape, often had crooked tips and were not very reproducible in manufacture. Only a small fraction of the pulled capil-

(Figure 1) Computed ray tracing of a parabolic shaped capillary from an on-axis x-ray source intercepting the capillary from a distance of 15 m. The inside diameter of the capillary design is 22 microns and its length is 9 cm. The net throughput is the calculated reflectivity of a single bounce from a smooth surface or 99.9%. If a purposeful slope error of 50 microradian is introduced, then 5 bounces are needed to exit the tube. The throughput for this ray is 0.1% and a beam of higher divergence is created than for the single bounce situation. The exit divergence of rays 1 and 2 are 0.14 mrad and 3.5 mrad respectively.

laries were found to be useful optical components.

We decided, therefore, to build a drawing station to manufacture capillaries of much higher quality. The new setup has already produced 3 to 28 cm long tapers, but the equipment is actually capable of pulling up to 1 m long tapers. Figures 2 and 3 respectively show an outline of the vertical glass puller and a view of the capillary during drawing. The glass pulling process is controlled by a computer using a LabWindows interface. The controlling parameters for a given glass type are pulling force, furnace temperature, and furnace position. We also measure the velocity of the glass leaving the pulling region to assist in the control process. All the relevant parameters are sampled on a 1 second time period and logged during the draw that generally lasts 15 minutes. A strain gauge tied to the bottom of the desired part keeps the lower section of the glass in fixed position and simultaneously measures the applied tension.



(Figure 2) Schematic drawing of the 2.1 m high glass drawing tower. The rail supporting the drawing equipment is attached to a large electronic equipment rack holding power supplies, electronic chassis, etc. A torque controlled motor is mounted at the top of the rail. A strain gauge is mounted at the bottom. The glass tube is supported in between on fish line while a moveable furnace softens the glass tube during the pulling process.

lary whose figure is being measured and recorded into a local computer file.

Definition and Measurement of X-ray Gain

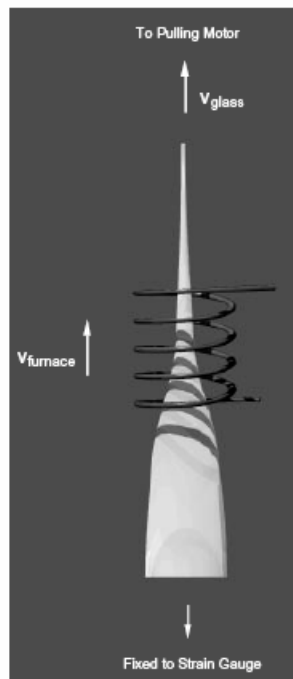
The performance of capillary optics is determined by three parameters: exit beam size, beam divergence, and intensity gain. The gain is defined as $G=T(A_1/A_0)$, with T the fraction of the flux leaving the capillary compared to that entering the capillary (as mea-

Control of the diameter vs. length profile is accomplished by varying the velocity of the furnace relative to the velocity of the glass pulled upwards by the motor. The furnace moves from bottom to top shaping the glass locally as it moves steadily upward. Near the base of the capillary, the furnace is moving rapidly and little glass is pulled from the heated zone. Thus the initial capillary diameter is reduced only by a small amount. Towards the end of the drawing process, the furnace has reached the tip region and is moving more slowly. The additional glass pulled from the heated zone reduces the diameter still further and thus shapes the fine tip of the capillary.

When we implemented these general ideas, we were able to achieve a figure close to our design value (Figure 4) after just a few trials. In subsequent tests, we have found that the capillary shapes of successive pulls are virtually identical, showing that this glass puller now can make reproducible parts.

Metrology

The figure and straightness of the pulled capillaries were measured using an optical microscope (Figure 5) equipped with a translation stage with 0.5 micron resolution (but runout in the bearings of a few microns). Our new capillaries are straight to within a few microns, the experimental error, over a taper length of up to 28 cm. By scaling the outside glass diameter to the inside diameter, one can infer the inner slope to a few microradians over a length of a few centimeters. Figure 5 shows a capil-



(Figure 3) A resistive heating coil of generally 3 to 6 cm length surrounding the glass tube provides the heat for the drawing process at 800 to 900 °C. Computerized equipment controls the furnace velocity as well as the glass extension and thus the velocity of the glass leaving the furnace.

sured through a short straight segment). A_1/A_0 is the ratio of the cross sectional area at the upstream end (A_1) and the downstream end (A_0) of the tapered capillary. Thus the gain factor describes the net increase of flux/area.

The capillaries were tested at station D1. X-rays from a bending magnet were monochromatized by a double-crystal monochromator set to 12 keV. The capillary was mounted on a gimbal mount providing a horizontal and vertical adjustment of its optical axis to the beam center with an accuracy better than 8.0×10^{-6} rad. Ionization chambers located at the upstream and downstream ends of the capillary recorded the incident and exit intensities.

The exit diameters of the borosilicate capillaries ranged from 0.5 to 5 microns. The initial inside diameter for this glass was 28 microns. Although the figure of the optics is very close to optimal, we did not measure high gain factors. The maximum gain observed was 31, the average value was much smaller. Since transmission of the capillary was very low, we changed the experimental setup and repeated our tests with a white x-ray beam. The statistics in our measurements improved significantly but no change in gain was observed. With white beam the glass showed some evidence for radiation damage, i.e. discoloration from F-centers. To confirm our results we tried several newly fabricated tapered capillaries of leaded glass, the same material for which gain factors of up to 960 had been previously measured using 12.3 keV x-rays. With the very intense white beam at CHESS the upstream end of the leaded glass melted and sealed the capillary. Therefore a gain could not be determined. In the future, improved shielding of the upstream end of the capillary should allow us to carry out white beam experiments without melting the glass.

Problems with Rough Glass Surfaces

After pulling borosilicate glass capillaries so close to the optimal figure, it was very disappointing to observe a gain of only 5 to 30 in x-ray flux when the design and ray tracing programs predict a gain of about 500 for a similar capillary condensing a beam from 28 to 1 micron diameter.

For high performance glass capillaries there are three ingredients required: a diameter vs. length profile that approaches an ideal taper; a straight guide

tube; and glass that is smooth on an atomic scale. Since the first two criteria were satisfied, this inferred that the glass must not have a smooth surface. Sectioning the glass along its length has revealed a very rough inner reflecting surface, that is, the glass shows many 'particle-like' defects. Using an optical microscope the size of these features were determined to be 0.5 to 2 microns in diameter, located only a few microns apart.

Future Directions and Goals

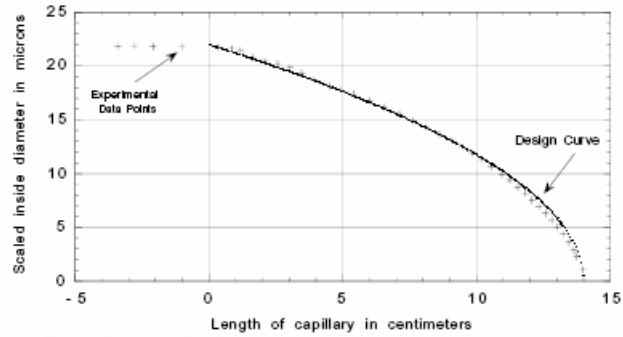
The obvious next step is to troubleshoot the current glass production process or try to obtain glass of higher quality from different manufacturers. We are confident that when good glass is available, we will be able to make capillary x-ray concentrators with high gain in flux.

Once the glass optics can be made routinely, we would like to see enough activity develop to support a micro-science beamline at CHESS.

Acknowledgments

We thank Vic Pollock for his help with the electronic instrumentation, Basil Blank for his help in design and machining of specialty parts, and Dan Thiel for many helpful discussions on capillary optics. We also thank Ernie Fontes and Jim Patel for helping us to obtain the borosilicate samples and to conduct some of the first experiments in the application of these tapered capillaries (see page 37).

1. Nanometer Spatial Resolution Achieved in Hard X-ray Imaging and Laue Diffraction Applications, D. H. Bilderback, S. A. Hoffman, and D. J. Thiel, Science Vol. 263 (1994) 201-203.
2. Time-resolved XAFS of a molecular excited state and glass-capillary concentration of x-rays, D. J. Thiel, Ph. D. Thesis 1992, Cornell University.



(above, Figure 4) Observed capillary diameter (+) vs. length of the capillary as measured with an optical microscope. The outer diameter (OD) was recorded and scaled as a measure for the inner diameter (ID) profile. The dashed line represents the parabolic design value. The profiles deviate only slightly showing that the pulling equipment produced a capillary close to the design value.

(below, Figure 5) Metrology setup with microscope featuring high optical magnification and a large translation stage with digital readouts used to measure the figure of the capillary after pulling. The boundaries of the capillary, i.e. the OD and if the glass is transparent also the ID, can be tracked optically to yield its size and straightness.

