An automated test system for measuring x-ray transmission through glass polycapillaries

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To support the development of the recently invented capillary-based x-ray optics technology, it is necessary to have quick and repeatable precision measurements of x-ray transmission. A novel fully automated measurement system has been built for this purpose. Measurements are reproducible within half a percent.

I. INTRODUCTION

In the early 1980's, M. A. Kumakhov proposed a new kind of optics based on the total external reflection of x rays from smooth surfaces. Since then the technology to control x rays using systematic arrangements of glass polycapillaries has been developing rapidly. Depending on the specific arrangement, beams can be focused or divergent beams made quasiparallel. Such capillary-based x-ray optics have significant potential for use in medical applications, astronomy, material analysis, crystallography, and lithography.

Different applications have different needs which affect the manufacturing of the capillaries. For example, in high energy applications, it is important that the diameters of the polycapillary channels be as small as possible; in low energy applications, however, the most important factor is that the composition of the glass have the lowest possible absorption. As new applications with differing requirements for polycapillaries are identified, new techniques of manufacturing must be developed and the results of those techniques evaluated. Accordingly, it is essential to develop a method to quickly and accurately measure the transmission of x rays through glass capillaries. Manual measurement techniques, like those used before the development of the automated system, are time consuming, require substantial investment in the training of personnel, and exhibit results that can vary greatly with the skill and experience of those performing the measurements.

The automated system can also facilitate scientific investigation. An example is a continuing analysis of radiation exposure effects. When glass is exposed to intense radiation, it is vulnerable to a number of physical and chemical changes. These changes may affect glass reflectivity, which depends on the characteristics of the glass, including its surface smoothness, composition, and density. Under irradiation, the density of the glass increases, which can result in surface cracking. Desorption of oxygen, alkali metal segregation, and the formation of oxygen bubbles may also occur. All of these radiation-induced effects can decrease the surface reflectivity of the glass. Since transmission through a fiber depends on the reflectivity to the nth power, where n is the number of reflections, even a slight decrease in reflectivity can be highly damaging to fiber transmission. The automated test setup helps the investigation of radiation effects by simplifying the measurement of large numbers of capillaries before and after irradiation.

II. EXPERIMENTAL TECHNIQUE

A. Overview

The setup, shown schematically in Fig. 1, consists of a small portable x-ray source, collimating pinhole, grooved fiber holding plate, and x-ray detector. The source, pinhole, and plate can each be moved independently in the x-y plane (the plane perpendicular to the fiber axis) for a total of six degrees of control. The detector is fixed. The problem is to simultaneously align the fiber axis with the detector, the pinhole, and the center axis of the x-ray beam emitted from the source. Small misalignments cause apparent variations in the measured fiber transmission. The problem differs from laser fiber optic alignment primarily in the lack of source coherence and collimation, lower photon count rate and greater requirement for shielding.

B. Procedure

Before alignment, the fiber, source, pinhole, and detector do not lie along the same axis and thus no signal is detectable through the fiber. The fiber holding plate is lowered, and the small pinhole is replaced with one of larger diameter. The source is moved vertically and horizontally until the signal is maximized. The smaller pinhole is remounted, signal is again maximized and the plate raised to its previous position. Since the detector window is much larger than the fiber diameter, there is no need for fine adjustment in positioning the detector. This process has been termed “prealignment” and is accomplished with the “scan” program described in Sec. II C. The program allows the user to specify an accurate range of motion along an axis and an energy region of interest (ROI) on the multichannel analyzer and to obtain on the computer screen either a spectrum, as shown in Fig. 2, or a plot of counts in the region of interest versus the position along the fiber.
axis, as shown in Fig. 3. Semiautomated prealignment of the system is required after preliminary installation of system components or major changes to the system, such as replacing the grooved plate to accommodate different fiber lengths.

Rough pre-alignment is maintained when fibers are inserted and removed from the system. Fine adjustment is performed under the control of the "auto" software, also described in Sec. II C. First, the computer scans the pinhole vertically to obtain a plot of the integrated counts in the ROI versus the position of the pinhole. The computer returns the pinhole to the position corresponding to the center of a Gaussian fit to this curve. (The scan distance and step size, the live time for which the multichannel analyzer acquires at each step, and the ROI is specified by the user prior to auto-alignment by means of a pop-up menu as shown in Fig. 4.) Next, the pinhole is scanned horizontally. Again, the best position of the pinhole is found and the pinhole set at that position. At this point, the pinhole and the fiber are in a straight line.

The position of the source is scanned in the same way as the pinhole. Since transmission efficiency versus source position gives significant information about the roughness of the capillaries, the preset live time at each step is increased for the source horizontal scan to keep statistical fluctuations of the counts under 1%. At each step of the source horizontal scan, a full spectrum is recorded for later extraction of detailed information. The best source position is determined and the source set at that position. At this point, the source, pinhole, fiber and detector lie on a straight line. A complete spectrum is taken; the fiber-holding platform is lowered out of the path of the beam, and another spectrum is taken. The source to detector distance is kept constant. The ratio of the two spectra determines the transmission as a function of energy.

C. Software

The software consists of two programs in C, one for rough alignment ("scan") and one for automated fine alignment and transmission measurement ("auto"). The "scan" program gives the user basic positioning control over each of six different axes. Spectra may be acquired, displayed and saved. The major purpose of the program, however, is to acquire counts at specified intervals over a range of positions for a given axis. Counts versus position are displayed on a line graph and may also be saved.

The "auto" program displays five graphs as shown in Fig. 5. Four of the graphs display scans, both vertical and horizontal, for the pinhole and source, as described in Sec. III. A scan consists of the plotted data points and a superimposed Gaussian best fit. The Gaussian is determined by means of the popular Levenberg–Marquardt algorithm for nonlinear least-squares fitting. The fifth graph displays spectra. The appropriate graphs are displayed and updated in
FIG. 3. Counts vs position of the actuator plot from the "SCAN" program.

real time as each capillary is aligned. The user may abort the alignment process at any stage. Since the program is capable of aligning several capillaries in succession, the user has the option of reviewing the graphs displayed for any of the capillaries aligned.

A separate menu allows the user to set up the parameters for the automated process, including the number of capillaries to be aligned, the distance separating them, the axes associated with the pinhole, source and capillary plate, and the MCA preset live times. The minimum count rate, maximum dead time, and level of source stability are set by the user to trigger an automatic abort where appropriate. Other automatic abort conditions (e.g., hardware limits, badly centered peaks in scans or spectra, failure to find a Gaussian fit in 15 iterations, etc.) are built into the software. The range and interval for scans over all axes may be set independently. In addition, each capillary to be aligned may have its own independent set of parameters so that different types and sizes

FIG. 4. Pop-up menu of "AUTO" program.
of capillary may be tested together. Individual parameters for single capillaries may be saved, as well as sets of parameters for groups of capillaries. Different group parameters may then be easily used to serve different purposes. A "rough scan" parameter set may have optimal settings for speed; another parameter set may be optimized for precision.

III. EXPERIMENTAL SETUP

A. Mechanical

We use a microfocus x-ray source manufactured in Russia by Svetlana Manufacturing with a copper anode and a source spot size of about 100 μm. The source is mounted on a Newport 416 series vertical stage, which in turn is mounted on a Newport 443 series horizontal stage. The stages are fitted with 1 in. 850B-1 series and 2 in. 850B-2 series Newport actuators, respectively. The pinhole holder is slotted to make it easy to change the pinhole size. The pinhole holder is mounted to a Newport 461 series XY stage, which is fitted with two Newport 0.5 in. 850B-05 actuators. The small pinhole (see Sec. II B) is slightly smaller than the fiber diameter. The capillaries are held straight by a precisely machined grooved plate. The plate can be moved horizontally and vertically with Newport 443 series linear positioning stages fitted with 2 in. Newport 850R-2 series actuators. All six actuators are connected to a Newport PMC400 actuator motion controller. The detector is installed next to the plate. The base mounts of the source, pinhole, plate, and detector are all mounted on a Melles Griot rail which rests on a Newport Optical table. Rough manual positioning is achieved with manual micrometers and vertical adjustment racks on the source, pinhole, capillary holding plate, and detector. Iron filings are used to stop x rays from passing around the outside of the fiber. X-ray leaks can be detected by measuring transmission through the fiber after placing water on the tip of the capillary. Since the water fills the interior of the capillary, the x-ray signal drops to background if there are no x rays passing around the capillary.

B. Detection electronics

An LND proportional counter is used for measuring transmission, and a second counter is used for monitoring source fluctuation. The resolution of the proportional counters is about 1.3 keV at 8 keV. The signal from each counter is amplified with a charge sensitive preamplifier and a Tennelec TC241 linear amplifier. Both amplified detector outputs are routed through a Tennelec TC306 router/multiplexer into a Tennelec PCA-Multiport multichannel analyzer. Source stability is monitored automatically during alignment. If the fluctuation in the monitor signal becomes greater than a preset value, a source instability error message is displayed, and the alignment is halted for 5 min to allow source fluctuation to diminish. Alignment is then automatically restarted from the beginning.

C. Computer control

Both "SCAN" and "AUTO" programs communicate with a Newport PMC400 motion controller and a Tennelec PCA-Multiport multichannel analyzer through an IEEE 488 (GPIB) interface bus. The PMC400 is a moderately priced intelligent motion controller. The PMC400 is designed to drive Newport's dc servo-based linear actuators, which have a resolution and a bi-directional repeatability of <1 μm. The PCA-Multiport has an 8 K internal ADC and is compatible with a wide variety of detectors and amplifiers.
Because GPIB facilitates the synchronization of multiple instruments, it was a natural choice for coordinating motion control and data acquisition. The IEEE 488 interface board used is the National Instruments AT-GPIB, which supports data transfer rates of over 1 MByte/s. The computer used is a 50 MHz 486DX2, made by Standard Computer Corporation (no longer in business). The programs were developed with the use of the National Instruments LabWindows technical programming environment. One important feature of LabWindows is that it permits the development of a complex "point and click" graphical interface with relative ease. The source code was compiled into executable code with the Microsoft C/C++ 7.0 compiler. The executable code runs as a DOS application under Windows 3.1 in 386 enhanced mode.

It should be noted that the PMC400 motion controller was designed for an earlier (and slower) generation of computers and GPIB interface boards. As a result of the high speed of our computer or the IEEE 488 interface card or both, some experimentation was necessary to ensure reliable performance of the PMC400 motion controller. The computer is run with "turbo" off to disable all internal and external cache capability, thus effectively slowing down the computer. In addition, wait states have been inserted into the code at strategic points. However, some functions of the PMC400 have proven too unreliable to be of use with our equipment.

IV. RESULTS

The data displayed in Figs. 2, 3 and 5 were obtained with a 12.5 cm long borosilicate polycapillary fiber 0.45 mm in diameter with 17 μm channel size. Plots of intensity obtained with the scan program as the source is scanned horizontally and vertically with no fiber in place are shown in Fig. 6. The pinhole and detector were fixed and the fiber-holding platform lowered out of the path of the beam. For a point source and pinhole, the expected width of the scan is

\[ w = \frac{x_s D}{x_d} = \frac{30 \text{ cm}}{17 \text{ cm}} \left(\frac{4.2 \text{ mm}}{2}\right) = 7.4 \text{ mm}, \]

where \( x_s \) is the source to pinhole distance which was 30 cm, \( x_d \) is the detector to pinhole distance, 17 cm and \( D \) is the diameter of the detector aperture 4.2 mm. The measured widths of the scans are 7.5 mm, in reasonable agreement.

A typical output screen from the auto program is shown in Fig. 5. The position marked by the dot was chosen by the software as the position of the center of the Gaussian fit to the intensity during the alignment of the pinhole. The auto program allows the user to select an energy region of interest. Typically, the unresolved Cu K peak, as shown in Fig. 2, is chosen as the energy at which the auto program computes the fiber transmission. Reliability of the automated system was tested by measuring transmission through a fiber, removing and replacing the fiber, and repeating the measurement. A set of transmission values resulting from this measurement procedure is shown in Table I. The measurements were repeated using the intensity maxima as the center position for alignment, in order to test the effect of the Gaussian fitting. The Gaussian fitting produced better repeatability, and had the added advantage of triggering an abort condition if the data points were too widely scattered to be meaningful. The tabulated transmission values have a one sigma mean variation of half a percent. For these measurements, the total number of counts was about 45000, so that Poisson statistics account for about 0.2% of the variation.

<table>
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<tr>
<th>Capillary type</th>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 3</th>
<th>Run 4</th>
<th>Run 5</th>
<th>Run 6</th>
<th>Run 7</th>
<th>Run 8</th>
<th>Run 9</th>
<th>Run 10</th>
<th>Avg.</th>
<th>s.d.</th>
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<td>98.1</td>
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<tr>
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<td>99</td>
<td>98.7</td>
<td>99.6</td>
<td>98.9</td>
<td>97.7</td>
<td>96.3</td>
<td>98.3</td>
<td>98.58</td>
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<td>32 μm poly Max</td>
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<td>61.4</td>
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<td>64.9</td>
<td>64.3</td>
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<td>64.4</td>
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<tr>
<td>Gauss</td>
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<td>66.4</td>
<td>66.4</td>
<td>66.4</td>
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<tr>
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<td>57.7</td>
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V. DISCUSSION

The automated procedure for measuring the x-ray transmission of polycapillaries is reliable and reasonably quick. An indefinite number of capillaries (the fiber-holding plate is currently machined for ten) can be measured in 1 run without operator intervention. Since most of the parameters governing the behavior of the process can be easily modified by the user from a pop-up menu, the software is very versatile. The system can be reliably operated with minimal training; during multiple measurements the user is free for other tasks. The results are reproducible to within half a percent.

ACKNOWLEDGMENT

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