Remotely controlled mirror of variable geometry for small-angle x-ray diffraction with synchrotron radiation*

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A total-reflecting mirror of 120-cm length was designed and built to focus synchrotron radiation emanating from the electron-positron storage ring at the Stanford Linear Accelerator Center (SPEAR). The reflecting surface is of unpolished float glass. The bending and tilt mechanism allows very fine control of the curvature and selectability of the critical angle for wavelengths ranging from 0.5 to 3.0 Å. Elliptical curvature is used to minimize aberrations. The mirror is placed asymmetrically onto the ellipse so as to achieve a tenfold demagnification of the source. The bending mechanism reduces nonelastic deformation (flow) and minimizes strains and stresses in the glass despite its length. Special design features assure stability of the focused image. The mirror reduces the intensity of shorter wavelength harmonics by a factor of approximately 100.

INTRODUCTION

The total-reflecting mirror described in this paper is used in conjunction with a logarithmic-spiral, crystal monochromator developed recently by Webb.[1] The system is used to focus x rays emitted from a storage-ring beam to a $0.5 \times 0.5\text{-mm}^2$ area and to select any desired wavelength within the range of 0.5 to 3 Å. It has now been in use for almost a year at the Stanford Linear Accelerator Center to study biologically important structures by x-ray diffraction.[2]

The mirror focuses the x-ray beam in one direction (in our case, vertically) and deflects it onto the silicon single crystal,[1] which in turn focuses in a direction perpendicular to the first one (in our case, horizontally). The advantage of using a mirror rather than another crystal in front of the monochromator is that it reduces harmonics and subharmonics. In the synchrotron beam, these are highly intense. The (111) planes of a silicon crystal will diffract λ and $\frac{1}{3}\lambda$ simultaneously as has been demonstrated clearly by some of our colleagues,[3] who used our system both with and without the mirror. The specific characteristics of the x-ray beam emanating from the synchrotron storage ring and its bearing on the basics of our focusing system have been described in a separate paper.[2]

DESIGN CRITERIA

The essential design criteria for our total-reflecting mirror are:

(1) The flux from the synchrotron source is to be utilized as fully as is practicable because high intensity of the focused beam is crucial for our experiments.
(2) The x-ray beam should be demagnified as far as is feasible in order to attain high resolution in small-angle diffraction with photographic recording and to keep exposure times within reasonable limits when small specimens are to be used.
(3) The mirror should cause a minimum of aberrations.
(4) The curvature of a mirror of the length needed must not depend on bending moments applied to the ends, if nonelastic deformation (flow) is to be avoided. The method of bending applied by Webb[1] to the monochromator crystal cannot be used here because of the mirror's own weight.
(5) The mirror-to-focus distance must be variable.
(6) Radiation damage of the mirror material (encountered in pilot experiments) must be minimized.
(7) Complete remote control is imperative because of the hazardous nature of the synchrotron beam.
(8) The mirror and bending device has to stay clear of the part of the synchrotron beam that is used by other research groups; only about 3.5 cm of the total 15-cm width is used by us.

The primary x-ray beam at our disposal is currently 7 mm high and 3.5 cm wide, but variations occur as has been discussed recently.[2] To intercept the entire height at the angle of total reflection (14 arcmin) requires a length of 200 cm. The longest mirror at our disposal is 120 cm, providing an entrance aperture of $120 \times \sin(14') \sim 5$ mm. Thus, 2 mm of the beam height is still not utilized.

Obviously, the mirror is to be placed so as to deflect vertically as otherwise it would be troublesome to handle the length required to fulfill condition (1) even approximately.

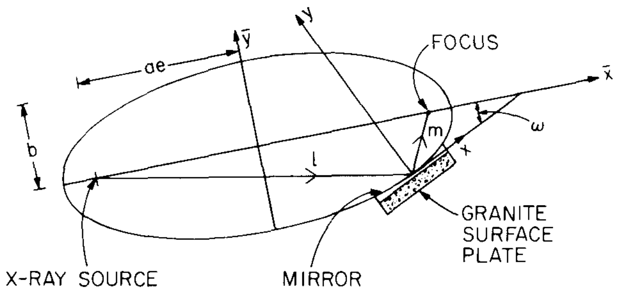

FIG. 1. Diagram of the elliptical focusing geometry. Lettering is explained in the Appendix.

CURVATURE OF THE MIRROR

To fulfill the condition of true point-to-point focusing, the mirror is curved to an ellipse in such a way that the x-ray source is located at one focus and its image at the other (Fig. 1). The demagnification of the source is determined by the ratio of the mirror-to-image distance (m in Fig. 1, here 160 cm) to the mirror-to-source distance (l in Fig. 1, here 1670 cm), which is roughly one to ten in our application.

The curvature was calculated in terms of the distance between any given point on the mirror surface and a plane tangent to the mirror surface at the center of the mirror (Figs. 1 and 2). This plane represents the surface of a flat granite plate which will be described later. The essential details of the calculations are given in the Appendix. Figure 2 shows three profiles of the mirror thus obtained, each for a different mirror-to-image distance, which is equivalent to the specimen-to-film distance in our application. It is seen that the ends of the 120-cm-long mirror will have to be lowered by about 0.01 cm if the mirror-to-image distance is changed from 200 to 300 cm. A change of the mirror-to-source distance from 1370 to 1970 cm requires a correction of only 0.001 cm at the ends. This feature is explained by the large eccentricity of the ellipse and the fact that the mirror is very close to one end of the major axis.

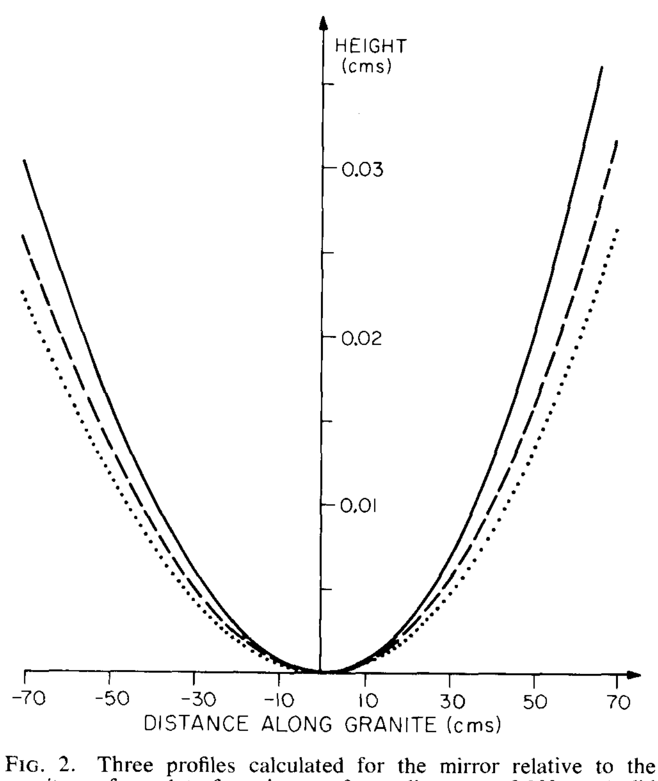

FIG. 2. Three profiles calculated for the mirror relative to the granite surface plate for mirror-to-focus distances of 200 cm (solid line), 250 cm (broken line), and 300 cm (dotted line).

SELECTION OF MIRROR MATERIAL

Various materials were tested for suitability as reflectors by exposure for extended periods of time to the highly intense synchrotron radiation. The critical angle of total reflection for nickel, platinum, and gold is approximately twice that for glass,[4] thus reducing the required length of the mirror to one-half. Unfortunately, coatings of these metals on glass did not withstand the radiation for long, but were either stripped or badly roughened. The most suitable material tested was float glass. It can be obtained at moderate cost, and selected pieces can be found with a surface smoothness comparable to that of the best polished surfaces.[5]

The sheet of float glass used is 0.5 cm thick, 30 cm wide, 120 cm long, and unpolished. It was selected from a batch of 30 pieces kindly donated to us by the Libby Owens Ford Company. The company halted the glass-making process until the pieces were cut out from the center of a continuous ribbon before passing through a varnishing process. The glass was handled by the edges with utmost care and placed in crates especially designed so as to avoid any contact with the reflecting surfaces.

In the preparation of float glass the melt is "floated" over molten tin in an inert atmosphere and cooled. The surface may show long-term undulations so as to necessitate special selection. Our test was performed by means of an optical flat, while the sheet of float glass rested on a granite surface plate. The piece selected showed no granularity within each interference ring ("orange peel"). Some high and low spots producing 50 or more fringes were observed approximately 40 cm apart. This sort of ripple might defocus the beam by ±0.01 cm (see later). Prior to final installation at SPEAR, the glass was washed with soapy water, ethanol, distilled water, and then dried under a jet of nitrogen gas.

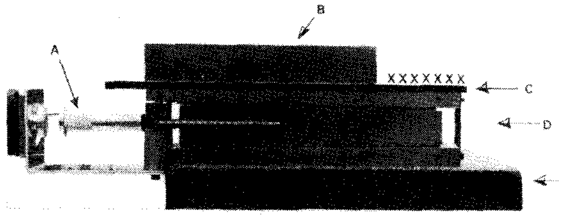

FIG. 3. End view of the focusing-mirror assembly. The glass mirror C is supported across its width by eight triple-wedge shims D resting on a granite surface plate E. The mirror is held against each shim by a 2.3-kg lead block B. A micrometer screw A driven by a stepping motor changes the height of each shim. XXX denotes the area illuminated by the x-ray beam.

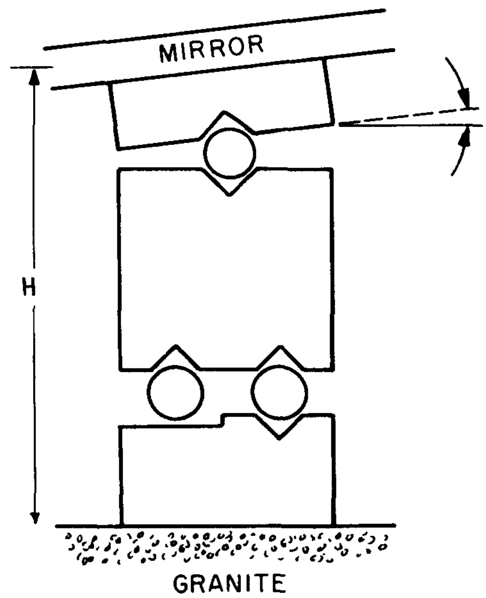

FIG. 4. Schematic of the adjustable triple-wedge shim. The height H changes by 0.010 cm per 2.54-cm travel of the central wedge. An adjustment of 0.0025 cm on the micrometer changes the height by 10^{-5} cm. The upper wedge is supported by a single ball race and adjusts itself to the curvature of the mirror as shown to the right.

BENDING MECHANISM AND SUPPORT

The prerequisites for production of a sharp and stable image are: (a) very fine control of the curvature, and (b) rigidity of the support.

The sheet of float glass described in the preceding section rests on eight triple-wedge shims of adjustable height and is held down on these by eight lead weights, one above each shim. Figure 3 shows an end view of the system. One shim is shown schematically in Fig. 4. It consists of three wedges, each one manufactured of 440 C stainless steel which was hardened and precision ground so as to assure a flatness and parallelism of the assembly to within ±0.0005 cm. It is seen that the bottom wedge has two ball races, whereas the top wedge has only one. Thus, it adjusts itself to the curvature of the mirror. The height H (Fig. 4) of each shim changes by 0.010 cm per 2.54 cm travel of the central wedge. An adjustment of 0.0025 cm on the micrometer screw changes the height by 10^{-5} cm. Since the float glass is not fastened to the shims but only rests on these under the weight of the eight lead blocks, minimal strains or stresses occur in it when it is bent.

The height of the top surface of any one shim, once set to its appropriate value, must be held constant relative to the others within a very small margin, as can be understood from the preceding section and Fig. 2; the heights of adjacent shims differ by less than 0.02 cm in most cases. Thus, the supporting plate must be extremely rigid and stable. We used a granite surface plate of the size 20 cm × 30 cm × 122 cm, having a flatness of ±0.00127 cm.[6]

The positions of the surface plate E, the triple-wedge shims D, the float glass C, and the lead weights B relative to each other are shown in Fig. 3. It is seen that each lead block B is shorter than the width of the float glass so as to avoid interference with the x-ray beam which illuminates an approximately 3.5-cm width of the mirror. The remaining 11.5-cm width of the 15-cm-wide (and 7-mm-high) synchrotron photon beam used by other research groups passes unaltered to the right of the mirror.

The approximately 225-kg weight of the granite block is supported by a welded steel structure. Lever arms, wheels, and rails incorporated in the structure provide x, y, and z translation of the surface plate and a continuously variable tilt.

Because of radiation hazards, direct access to the mirror is prohibited when the storage ring is in operation. Each of the eight micrometers (A in Fig. 3) is driven by a remotely controlled stepping motor while its scale is read via a television camera. The sideways translation, height adjustment, and tilt of the surface plate are likewise controlled remotely via stepping motors, while scale readings are performed via a television camera.

ALIGNMENT AND PERFORMANCE

Each shim height (H in Fig. 4) was preadjusted to correspond to the calculated mirror curvature (Fig. 2 and Appendix) using a dial indicator of 0.0025-cm sensitivity. The flat and smooth granite surface served as reference for each measurement.

All subsequent alignments were done via the remotely controlled stepping motors while viewing the x-ray image produced on a CsI (T1) scintillation crystal[7] through 61-cm-long fiber optics.

A series of photographs taken in the course of alignment is shown in Fig. 5. Each horizontal row represents a set of pictures of the focus as one wedge is raised 200 steps at a time (corresponding to 0.125 mm). The eight rows correspond to the eight adjustable

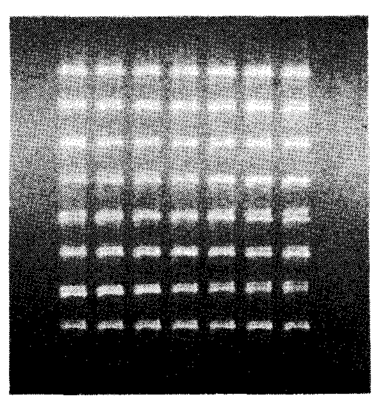

FIG. 5. A series of photographs of the x-ray beam taken at the focus. The beam was deflected away from the mirror by means of a flat silicon crystal to render it accessible to the camera. The eight rows correspond to the eight adjustable shims, beginning with the upstream shim at the top row. Each horizontally succeeding picture was taken after one shim had been raised by 0.125 mm (200 steps on the stepping motor). Each line in the fine structure arises from a section of the mirror between two shims which reflects independently of the other sections. Therefore, the height adjustment of an internal shim affects both neighboring sections, and a shim on the end affects only one end section.

shims. Thus, there are eight independent foci. The origin of each one was traced to a different part of the mirror either by systematically varying the height of one shim at the time or by shielding off a corresponding part of the mirror. The final focusing was essentially a process of superimposing the separate foci by adjusting the shims, starting at the center of the mirror and working gradually toward the ends until the image was 0.05 cm high and free from texture.

Refocusing is necessary when the critical angle of reflection (tilt of the mirror and granite plate) is changed so as to correspond to a significantly different wavelength. This was experienced when changing from 1.38 Å (K absorption edge of copper) to 1.74 Å (K absorption edge of iron). These two wavelengths are needed for experiments involving anomalous scattering.

The contribution to the overall intensity from each part of the mirror was measured by selectively aperturing down different portions of the mirror. At present about two-thirds of the mirror seem to be focusing with reasonable efficiency (60%–70% reflectivity).

The contribution of shorter wavelength harmonics to the monochromatized beam was measured with the use of a scintillation counter and a series of aluminum foils as absorbers. A plot of the intensity on a log scale versus foil thickness, and extrapolation back to zero thickness showed that the harmonics contributed less than 0.1%.

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APPENDIX

The heights of several points on the mirror surface were calculated relative to a plane tangent to the ellipse and tilted at the critical angle (14′) to the horizontal, primary beam. The equation of the ellipse as referred to this tangent is

$$(x \cos\omega + \bar{x} - y \sin\omega)^2/a^2 + (x \sin\omega + y \cos\omega - \bar{y})^2/b^2 = 1,$$

where $\bar{x}$ and $\bar{y}$ are the normal axes of the ellipse, parallel to the major and minor axes of lengths a and b, respectively; see also Fig. 1.

This equation has a solution,

$$y = \left| \frac{-|B| + (B^2 - 4AC)^{1/2}}{2A} \right|,$$

where

$$A = 1 - e^2 \sin^2\omega,$$
$$B = [-2 \sin\omega(1 - e^2)(x \cos\omega + \bar{x}) + 2 \cos\omega(x \sin\omega - \bar{y})],$$
$$C = (x \cos\omega + \bar{x})^2(1 - e^2) + (x \sin\omega - \bar{y})^2 - a^2(1 - e^2),$$

and

e = eccentricity of the ellipse,
$\bar{x} = l \cos\theta - ae$,
$\bar{y} = l \sin\theta$,
$\omega = 14'$,
l is the source-to-mirror distance = 1670 cm,
m is the mirror-to-focus distance, typically 160 cm,

and

θ is the angle between the x-ray beam and the major axis at the x-ray source.

Since the ellipse is highly eccentric, $a \approx ae \approx (l + m)/2 = 765$ cm. Some calculated curves are shown in Fig. 2, and discussed in the text.

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[6] Granite is one of the best stabilized materials at hand. Granite surface plates are used in virtually any instrument shop, are readily available in a variety of grades, and provide a flat, smooth, and stable surface at moderate cost.

[7] The image produced in a thin crystal slice of CsI(Tl) is superior in resolution to that observed on a fluorescent screen. CsI(Tl) emits a longer wavelength than NaI(Tl), which is more favorable to the phototropic eye response. Also, it is nonhygroscopic.