January 1987

BNL 39293

Fabrication of an 8:1 ellipsoidal mirror for a synchrotron x-ray microprobe

BNL--39293

DE87 006457

Keith W. Jones, Peter Z. Takacs, Jerome B. Hastings
Brookhaven National Laboratory, Upton, New York 11973

John M. Casstevens and Christopher D. Pionke
Optic-Electronic Corp., Dallas, Texas 75243

Presented at

SPIE's O-E/LASE '87 Optoelectronics and Laser Applications in Science and Engineering
and Electro-Optic Imaging Systems and Devices '87

Los Angeles, California

January 11-16, 1987

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

The submitted manuscript has been authored under contract DE-AC02-76CH00016 with the Office of Basic Energy Sciences, US Department of Energy. Accordingly, the US Government retains a nonexclusive, royalty-free license to publish to reproduce the published form of this contribution, or allow others to do so, for US Government purposes.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Fabrication of an 8:1 ellipsoidal mirror for a synchrotron x-ray microprobe

Keith W. Jones, Peter Z. Takacs, Jerome B. Hastings

Brookhaven National Laboratory, Upton, New York 11973

John M. Casstevens and Christopher D. Pionke

Optic-Electronic Corp., Dallas, Texas 75243

Abstract

The fabrication of an 8:1 demagnifying ellipsoidal mirror to be used for an x-ray microprobe at the National Synchrotron Light Source X-26 beam port. The design aim was to produce a mirror that could be used over the photon energy range from about 3 to 17 keV. The 300-mm long mirror was required to operate at a grazing angle of 5 mr. The semimajor axis was 4500 mm and the semiminor axis 14.142 mm. Surface roughness of 1 nm or less and slope errors of 1 arc second parallel to the long axis and 200 arc seconds parallel to the short direction were specified. Production of the first electroless nickel-coated aluminum mirror using a diamond-turning technique has been completed. The mirror meets the 1 arc sec surface figure specification except for areas near the ends of the mirror. The reasons for these deviations arise from subtle details of the diamond-turning process which have not been fully incorporated into the computer program that controls the diamond-turning machines. Further work in computer correction of repeatable errors of the diamond-turning machine can eliminate the waviness at the ends of the mirror. The diamond-turned mirror surface was not fully polished under this effort and therefore does not meet the roughness specification; however, surface smoothness of a fully polished cylindrical mirror manufactured using the same techniques does meet the specification. It can be concluded that it is now technically feasible to meet the required specifications for the mirror and that the x-ray microprobe based on its use can be achieved.

Introduction

The use of the synchrotron light source for x-ray fluorescence (XRF) of trace elements is now well established at a number of different synchrotron centers.¹⁻⁶ The high flux of the x rays from such sources means that the sensitivity of the XRF determinations is very high and, in addition, that the size of the spot probed on the sample can be limited to diameters of the order of some micrometers. The combination of these two qualities means that synchrotron radiation-induced x-ray fluorescence (SRXRF) is a unique way of measuring the elemental composition of heterogeneous samples over small regions which are, in themselves, homogeneous. Two examples of disciplines where this is useful are in the biomedical and earth sciences.⁷⁻⁹

SRXRF measurements can be made effectively with very simple apparatus. For example, at Brookhaven we are operating a microprobe which uses a stepping-motor-controlled tantalum collimator to make beam spots with sizes down to 10 micrometers in diameter from the unfocussed white x-ray spectrum produced by a bending magnet source. The minimum detection limit (MDL) which can be attained under these conditions can be under 1 part per million (mass fraction) in some sample matrices. This amounts to detection of only about 10^{10} atoms.

The performance of the microprobe apparatus rests on the number of photons delivered per unit area and time at the target. An increase in target flux means that the measurements can be made in shorter times, or with better sensitivity, or with better spatial resolution, or with monochromatic beams. The various parameters can be adjusted to meet the demands of the variety of individual experiments which are of interest with more flexibility if the flux is high.

The use of a focussing mirror in the beam line can potentially increase the photon flux by several orders of magnitude and thus qualitatively improve the operation of the microprobe. The design, fabrication, and testing of an 8:1 ellipsoidal mirror for use in SRXRF experiments is described here.

Design criteria

The ellipsoidal mirror described is intended for use at the National Synchrotron Light Source on the X-26A beam line as a major element in a x-ray microprobe facility. The mirror parameters chosen were thus strongly influenced by the research aims of the groups using the beam line as well as by physical considerations of the mirror manufacture.

749 05

Several different approaches have been taken to the provision of high photon flux in a small image spot. The present design is based on the use of an ellipsoidal mirror working at grazing angles less than 5 mrad so that coverage of the energy range up to 17 keV is allowed¹⁰⁻¹¹. The properties of the ellipsoid were chosen to give maximum demagnification within the constraints of a reasonable beam line length and what were believed to be the limits of manufacturing technology. The parameters chosen are listed in Table 1. It can be seen that some of the radii are very small and that the tolerances on surface finish and slope errors are very stringent. The mirror was thus designed at what was believed to be the limit of manufacturing technology available at the time, and it was understood that meeting these specifications would be challenging.

The object size of the NSLS x-ray source is about 500 μm $\times$ 1500 μm . Reduction of that size by a factor of eight in both dimensions gives an image of minimum dimension of 60 μm . In practice the mirror would then require the use of collimation to achieve smaller and variably-sized images that could be used for making measurements. It would also be possible³ to use a collimated image from the ellipsoidal lens as the object for a second stage of demagnification by a Kirkpatrick-Baez type focussing lens. In this case it would be possible to go to a smaller image size with little loss of flux.

Table 1. Design Parameters for the 8:1 Ellipsoidal Mirror

Object Size:	0.5 $\times$ 1.5 mm ² (4 σ)
Object Distance:	8 m
Image Distance:	1 m
Grazing Angle:	5 mr
Energy Range:	4-17 keV
Major Axis Diameter:	9000 mm
Minor Axis Diameter:	28.284 mm
Length:	30 cm
Surface Roughness:	1.0 nm
Slope Error Parallel to Long Direction:	1 arc sec
Slope Error Perpendicular to Long Direction:	200 arc sec
Material:	Al substrate, electroless Ni overcoat between .05 to .5 mm

From the scientific standpoint the interest lies in the sensitive detection of trace elements with spatial resolutions close to one micrometer. For the trace element determinations, it should be possible to use x rays from around 3 keV to about 17 keV in order to make possible fluorescence of K- or L-shells in almost all elements in the periodic table. It is possible to operate using either monoenergetic photons or the total spectrum of radiation from the synchrotron. These factors imply that the microprobe should provide maximum flux at the sample with a divergence angle which is small so that good depth of field is provided. The sensitivities that are feasible have been calculated¹²⁻¹⁴.

Manufacturing techniques

The basic approach to the production of the required mirror used a computer-numerically-controlled (CNC) diamond-turning machine to carry the part past a small flycutter mounted on the machine's air-bearing spindle. To produce the required contour requires three axes of CNC control. The mirror blank is mounted on a fixture attached to the machine's rotary table. This table is under machine control and is referred to as the "B" axis.

The diamond-turning machine is configured to allow the air-bearing spindle which carries the flycutter to be swivelled at an angle to the machine's two linear axes. The linear "X" axis is perpendicular to the machine spindle when it is in its home position, and the "Z" axis runs perpendicular to the "X" axis and, therefore, parallel to the spindle axis of rotation.

To provide clearance for a large and sufficiently stiff flycutter, the spindle is swivelled at an angle to the linear slides. The angle of the spindle and the size of the flycutter radius of rotation are chosen to give an elliptical path that closely approximates the radius of curvature at one end of the elliptical groove. As the part is advanced past the rotating cutter by the "X" axis movement, the "Z" axis is simultaneously advanced to increase the depth of the cut.

As the two linear motions move the part past the flycutter along an elliptical path, the rotary "B" axis rotates the part to change the angle of attack of the rotating flycutter. The minor radius of curvature approximated by the elliptical cutter path smoothly changes from the 8.27-mm radius required at one end of the mirror to the 9.44-mm radius needed at the opposite end.

The set-up that was used for the diamond turning is shown schematically in Figure 1. The actual arrangement is illustrated in the photograph shown in Figure 2.

Figure 1. Mirror blank is shown positioned in diamond-turning apparatus during the machining of the ellipsoidal surface.

Figure 2. Diagram showing the motion of the mirror past the diamond flycutter in the diamond-turning machine.

The original computer program used to control the diamond-turning machine used several simplifying assumptions to approximate the surface of the required ellipsoid. For example, the effect of the finite radius of the diamond tool was ignored. The results of the first attempt to fabricate the mirror are shown in the profile given in Cut 1, Figure 3. After this first attempt, it was realized that the program should be refined to correct these problems by using more sophisticated correction algorithms. The first correction accounted for the effects of the finite tool radius. The results of this correction can be seen in Cut 2, Figure 3. Other sophisticated correction algorithms were added to the computer program resulting in the contour error as shown in Cut 3, Figure 3.

Figure 3. Results of measurement of the surface contour of the mirror after three attempts to machine the ellipsoidal surface.

At this point available funding to continue the project was exceeded, and therefore, further efforts to correct the computer program were not made. However, in order to make the mirror usable, a limited amount of post-polishing was attempted in order to give a better surface finish and to remove some of the worst slope errors. The results of this approach are described in the next section.

BNL testing results.

The mirror was shipped from Optic-Electronics Corp. in Dallas to Brookhaven for measurement in the Instrumentation Division Metrology Laboratory. A picture of the final mirror being readied for its rigorous testing routine by one of the many skilled BNL Metrology technicians is shown in Figure 4.

The mirror was first examined with a Wyko NCP-1000 Digital Optical profiler with a 2.5X objective at a filter center wavelength of 652.9 nm. Measurements were made along the central trough of the mirror surface at several locations. Two types of profiles were obtained: one showing the effect of scratches along the axis (Figure 5), the other apparently free of these major defects (Figure 6).

Figure 4. Mirror blank being adjusted by technician for measurements of surface roughness and figure using the laser beam methods described in the text.

Analysis was performed with the BNL PROFILE code.¹⁵ After analysis of the profiles, it was possible to compute an "average" periodogram estimate of surface power spectral density (PSD). The average periodogram for the scans that were free of obvious surface scratches is shown in Figure 7.

The RMS roughness (σ) and RMS slope error (m) are computed from the PSD curve by integration over the appropriate bandwidth. The standard bandwidth ranges for the 2.5X objective are defined as:

Full bandwidth = 5.0 mm to 9.88 μ m
 Low frequency = 5.0 mm to 1.0 mm
 High Frequency = 1.0 mm to 9.88 μ m

Figure 5. Surface profile measured for the mirror in the region of a surface scratch.

Figure 6. Surface profile of the mirror measured in a region free of scratches.

Figure 7. Surface power spectral density obtained from analysis of the surface profiles taken in regions free of surface scratches.

Table 2. Results of Measurements on RMS Roughness and Slope Error

PSD File	RMS Roughness (nm)			RMS Slope Error (mrads)	
	σ_{Full}	σ_{Lo}	σ_{Hi}	m_{Lo}	m_{Hi}
All scans (10)	35.925	32.554	8.828	.087	(.161)
Scratchless scans (4)	5.237	5.141	1.123	.012	(.089)

The RMS roughness values can be compared to measurements made on a diamond-turned aluminum flat. In this case values of 1.23 and 2.41 nm were found for the high and low frequency values. This is much better than was attained for the ellipsoidal mirror.

The periodogram curves can be directly related to the scattered light intensity in the reflected beam, according to the basic scattering equation in the grazing incidence approximation:¹⁶

$$\frac{1}{I_R} \frac{dI_S}{d\theta_S} = \frac{1}{\pi} \cdot k^3 \theta_i \theta_S^2 \cdot W(f)$$

where θ_i and θ_S are the grazing incidence and scatter angles and $W(f)$ is the power spectral density as a function of surface spatial frequency. If we integrate over the angle subtended by the geometric size of the image, calling it the detector solid angle, ψ , then we can normalize the angular scattering curve to the intensity in the specular beam:

$$\frac{S(\theta_S)}{S_R} = \left[\frac{\psi}{\pi} \cdot k^3 \theta_i \theta_S^2 \right] W(f)$$

This is the function that would be measured if a narrow slit were scanned through the image in the focal plane. For small scattering angles about the specular beam, the $\theta_S = \theta_i$ and the term in brackets is a constant.

For the case considered here where:

$$\psi = \frac{30 \mu m}{1 m} = 30(-6) \text{ radian}$$

$$\theta_S = \theta_i = 5 \text{ mrad}$$

$$K = \frac{2\pi}{\lambda} \text{ where } \lambda = 1 \text{ A}$$

The value of the scale factor in brackets is $300 \mu m^{-3}$.

The horizontal frequency axis is converted to image plane distance by means of the grating equation factor

$$\frac{\lambda F_2}{8i} = 2 \times 10^4 \mu\text{m}^2.$$

If these scale factors are applied to the average PSN curve for all ten scans, the results shown in Figure 8 are obtained. The wings of the scattering curve outside the region of specular reflection should normally be much less than unity at distances greater than $15 \mu\text{m}$ from the center. Scaling the power spectrum shows, however, that the ratio of the scattered to the specular intensity is greater than unity out to about $65 \mu\text{m}$ from the center (indicated by the rectangular box drawn on the curve). Since the predicted scattering intensity is much greater than unity at $15 \mu\text{m}$, the smooth-surface approximation used in deriving the above scattering equation breaks down for this case, and the shape and magnitude of the true scattering curve can only be guessed at from this analysis. It appears that the size of the image formed by the mirror will be about $65 \mu\text{m}$ in radius.

An independent approach to measure overall mirror figure is to perform a point spread function scan.¹⁷ In this experiment, a $50\text{-}\mu\text{m}$ diameter pinhole source was placed 8 meters away at the F_1 position of the ellipsoidal mirror. The scanning slit was set up 1 m away at the F_2 point in the image plane. A mask was placed immediately in front of the mirror to reduce the horizontal aperture to about 4 mm. The vertical aperture is estimated to have been about 1.2 mm, as deduced from the results of the model calculations. Scans through the image in the vertical direction (the plane of incidence) were made by stepping the slit aperture in increments of $100 \mu\text{m}$. A series of scans was made by varying the distance of the scanning slit from the mirror: the slit was placed at 80 cm, 90 cm, 100 cm, and 120 cm away from the mirror center. The measured image profile for the slit at the 100-cm nominal focus position is shown in Figure 9. What is displayed is a typical point spread function of a rectangular aperture distorted by residual aberrations in the mirror. In this case the first order peak on the right hand side is larger than the corresponding peak on the left hand side. The shape of the diffraction image does not change significantly as a function of focal distance, which indicates that the overall image at visible wavelengths is extremely diffraction-limited.

Figure 8. Relative intensity of the image predicted as a function of image plane distance. The curve indicates that the size of the image will be about $160 \mu\text{m}$ in diameter for a point source at 8 m from the mirror.

Figure 9. Scan of mirror laser beam image measured by scanning a rectangular slit normal to the mirror axis at a distance of 1 m from the mirror center. The dashed curve is the result of a model calculation which can be used to deduce the RMS surface slope error.

Figure 9 shows the result of the DIFFRACT¹⁵ model calculation overlayed on the data from the $F_2 = 100$ cm scan. The wavefront aberration required to reproduce the observed asymmetry is a single period sinusoid across the projected vertical aperture with an amplitude of 0.02 waves (1 wave = $0.6328 \mu\text{m}$). The RMS surface slope error can then be estimated using the relation:

$$\langle m \rangle_{\text{RMS}} = \frac{\sqrt{2}}{2} \frac{\pi A}{W} \cdot N$$

where A is the wavefront amplitude, W is the aperture width, and N is the number of sinusoid periods. The RMS surface slope is then estimated to be about $23.4 \mu\text{rad}$, or 4.8 arc seconds.

One interesting sidelight in comparing the Wyko Profile data to the point spread function measurement is shown in Figure 10. Here the two PSD curves from the ellipsoid are plotted together. The upper curve is for the full set of files, the lower curve for the four smooth files. Also plotted on this graph is an "X" in the upper left corner which represents the PSD of the single period sinusoid deduced from the model calculations of the residual wavefront error. This is the point corresponding to a model wavefront error with amplitude 0.020 waves over a spatial period of 300 mm. The coordinates of the point on this graph are $[4018 \mu\text{m}^3, 3.3(-6) \mu\text{m}^{-1}]$. The fact that this point appears to lie on an extrapolation of the PSD curves may be coincidental, but it may also tell us something more fundamental about the validity of each measurement and of the true nature of polished electroless nickel surfaces.

Figure 10. Surface power spectral density functions obtained from the Wyko profilometer data. The full curve represents all data taken and the dashed curve includes only the data for scratch-free regions. The point obtained from the laser scan is also shown. It is consistent with the value obtained from the direct profile measurements.

Conclusions

The work on mirror production that has been described here has been a very interesting and stimulating experience in many respects. A number of conclusions can be drawn at the present time.

We were aware from the beginning that the desired mirror would challenge all aspects of the mirror maker's arts. This has indeed been the case, and the results for surface finish and figure that are shown here are evidence that the original design goals were not completely met during the course of the project.

Large progress was made on the computer programming for the computer-controlled diamond-turning apparatus. To successfully machine the radii specified in the design required a sophisticated approach to properly control the motion of the tool along the mirror surface. At the start of the work, the importance of the finite size of the tool and the way that changes in the cutting angles could affect the mirror figure were not appreciated. As a result of the first machining effort, this effect was recognized, and ways of incorporating the predicted deviations from the proper surface could be properly incorporated into the computer programming for the diamond-turning machine.

A short attempt to attain better surface conditions was made by polishing the mirror after the last diamond-turning cut. This postpolishing was shown to help conditions, but it is probably doubtful that it is desirable to use it to try to get to an acceptable condition for the surface. The alternative approach of using a lacquer coating followed by an evaporation of a platinum overlayer is now being considered as an easier and more effective approach.

We conclude that the first trial mirror manufactured here should be capable of producing images, but only with a demagnification factor which is close to one. Obviously, there are simpler mirror designs that could be used to attain this value. The information gained during the present manufacturing process should, however, make it possible to reach demagnification values closer to design values of eight in the future.

Acknowledgments

The work described here was supported by the Processes and Techniques Branch, Division of Chemical Sciences, Office of Basic Energy Sciences, US Department of Energy under Contract No. DE-AC02-76CH00016.

We acknowledge many discussions with M. R. Howells, L. Grodzins, and P. Boisseau. The driving force for the mirror development has been the analytical work in which the BNL Department of Applied Science Division of Atomic and Applied Physics is engaged. We are particularly indebted to staff members in that Division for many provocative discussions.

References

1. Hanson, A. L., Kraner, H. W., Jones, K. W., Gordon, B. M., Mills, R. E., and Chen, J. R., "Trace Element Measurements with Synchrotron Radiation," IEEE Trans. Nucl. Sci., Vol. NS-30, pp. 1339-1342. 1983.
2. Gilfrich, J. V., Skelton, E. F., Oadri, S. B., Kirkland, J. P., and Nagel, D. J., "Synchrotron Radiation X-Ray Fluorescence Analysis," Anal. Chem., Vol. 55, pp. 187-190. 1983.
3. Knochel, A., Petersen, W., and Tolkiehn, G., "X-Ray Fluorescence Analysis with Synchrotron Radiation," Nucl. Instr. and Methods, Vol. 208, pp. 659-663. 1983.
4. Bos, A. J. J., Vis, R. D., Verheul, H., Prins, M., Davies, S. T., Bowen, P. K., Makjanic, J., and Valkovic, V., "Experimental Comparison of Synchrotron Radiation with Other Modes of Excitation of X-Rays for Trace Element Analysis," Nucl. Instr. and Methods, Vol. B3, pp. 232. 1984.
5. Iida, A., Sakurai, K., Matsushita, T., and Gohshi, Y., "Energy Dispersive X-Ray Fluorescence Analysis with Synchrotron Radiation," Nucl. Instr. and Methods, Vol. 228, pp. 556-563. 1985.
6. Baryshev, V. B., Kulipanov, G. N., and Skrinisky, A. N., "Review of X-Ray Fluorescent Analysis Using Synchrotron Radiation," Nucl. Instr. and Methods, Vol. A246, pp. 739-750. 1986.
7. Jones, K. W., Gordon, B. M., Hanson, A. L., Kwiatek, W. M., and Pounds, J. G., "X-ray Fluorescence with Synchrotron Radiation," Ultramicroscopy, to be published.
8. Chen, J. R., Martys, N., Chao, E. C. T., Minkin, J. A., Thompson, C. L., Hanson, A. L., Kraner, H. W., Jones, K. W., Gordon, B. M., and Mills, R. E., "Synchrotron Radiation Determination of Elemental Concentrations in Coal," Nucl. Instr. and Methods, Vol. B3, pp. 241-245. 1984.
9. Sutton, S. R., Rivers, M. L., and Smith, J. V., "Applications of Synchrotron X-Ray Fluorescence to Extraterrestrial Materials," Nucl. Instr. and Methods, to be published.
10. Howells, M. R., and Hastings, J. B., "Design Considerations for an X-Ray Microprobe," Nucl. Instr. and Methods, Vol. 208, pp. 379-386. 1983.
11. Boisseau, P., and Grodzins, L., "Design of the NSLS Microprobe Optical System." 1983. Unpublished.
12. Gordon, B. M., "Sensitivity Calculations for Multielemental Trace Analysis by Synchrotron Radiation Induced X-Ray Fluorescence," Nucl. Instr. and Methods, Vol. 204, pp. 223-229. 1982.
13. Gordon, B. M., and Jones, K. W., "Design Criteria and Sensitivity Calculations for Multielemental Trace Analysis at the NSLS X-Ray Microprobe," Nucl. Instr. and Methods, Vol. B10/11, pp. 293-298. 1985.
14. Grodzins, L., "Electron, Proton and Photon Induced X-Ray Microprobes: Analytic Sensitivity Versus Spatial Resolution," NeuroTox., Vol. 4, pp. 23-35. 1983.
15. Church, E. L., and Takacs, P. Z., "Use of an Optical-Profiling Instrument for the Measurement of the Figure and Finish of Optical-Quality Surfaces," presented at the Third Intern. Conf. on the Properties and Metrology of Engineering Surfaces, April 1985, Middlesborough, United Kingdom, Wear, to be published.
16. Church, E. L., "The Role of Surface Topography in X-Ray Scattering," Proc. SPIE, Vol. 184, pp. 196-202. 1979.
17. Takacs, P. Z., and Colbert, J., "Evaluation Techniques for X-Ray Mirrors and Systems Using Visible Light," Nucl. Instr. and Methods, Vol. A246, pp. 223-226. 1986.