Precision Machining of Electroless Nickel Mandrel and Fabrication of Replicated Mirrors for a Soft X-Ray Microscope*

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A Wolter type I aspheric mirror which is a key optical element used in a soft X-ray microscope requires super smooth surface and highly accurate figure. This paper deals with fabrication of the Wolter type I microscope mirror by an epoxy replication method. The required figure accuracy of the mirror optimized with surface roughness of 2 nm rms and photon energy of 539 eV was estimated for a prototype soft X-ray microscope. A master mandrel was prepared by single-point diamond turning and polishing, and a precise aspheric gold mirror with axial symmetry was successfully obtained from the master mandrel through replication processes. The fabricated mirror showed surface roughness of 1.12 nm Ra and figure error of 34 nm rms, and several mirrors could be obtained from only one master mandrel without destroying its surface roughness and shape.

Key Words:  Single-Point Diamond Turning, Aspheric Mirror, Replication, Figure Error, Surface Roughness

1. Introduction

Soft X-ray microscopes can be used to investigate hydrated specimens, particularly living cells, with high resolution(1). A Wolter type I mirror(2) as shown in Fig. 1 has been used in X-ray microscope systems. This aspheric mirror which has inner reflecting surface requires super smooth surface and highly accurate figure simultaneously. In addition, the diameter of the aspheric mirror is generally not so large (less than several centimeters) in X-ray applications. The fabrication of the aspheric mirror is very difficult because of its small geometrical dimension and tight tolerances.

There are two methods for fabricating the mirror. One is direct machining of inner reflecting surface using single-point diamond turning (SPDT) and polishing processes(3). This method is not easy to fabricate a mirror with less than 40 mm in diameter(4). The polishing of inner reflecting surfaces is also difficult to obtain the desired surface roughness and shape. The other is a replication method using a master mandrel and copy processes(5). The master mandrel prepared to fabricate a desired X-ray mirror has a counter surface, and its machining is relatively easy compared to the direct inner machining.

The replication method is suitable for fabricating a mirror with a high aspect ratio that has a small diameter and a long length. This advantage can offer possibility to make a flexible mirror design bringing high throughput. In addition, it is possible to fabricate many replicated mirrors using only one master mandrel, and it is surely a cost effective method. However, for example, in a glass replication method(5) the original roughness of the master mandrel is changed after making one replica because of destroying of its surface by heating it up about 973 K to melt an outer mirror substrate (Pyrex glass), and occasionally the replicated Pyrex mirrors need to additional polishing to reduce the surface roughness. The quality and the number of the mirror fabricated by replication technique surely depend

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on the replication method or its processes.

The contrast and the resolution of an image obtained from an X-ray microscope critically depends on the performance of optic used. Thus, the optic is a key element in an X-ray microscope. In this paper, we deal with the fabrication of a Wolter type I microscope mirror with super smooth and highly accurate surface and with axial symmetry using an epoxy replication method. One design example of the mirror is given, and its optical performance and figure tolerance are discussed briefly. A master mandrel is prepared by SPDT and polishing processes, and the designed mirror is then fabricated as following the epoxy replication processes. Surface roughness and figure error of the replicated gold mirror are examined.

2. Mirror Design and Required Tolerances

The shape and size of a Wolter type I mirror can be determined by four independent parameters: magnification, central grazing incidence angle, object-to-image distance, and length of hyperboloid\(^6\). We consider an objective mirror of Wolter type I used in a soft X-ray microscope with laboratory-scale size based on the laser-produced X-ray source. This was designed under a magnification of 32 times, a central grazing incidence angle of 28.8 mrad, an object-to-image distance of 1640 mm, and a length of the hyperboloid of 13.08 mm. The designed mirror has a total length of 29.06 mm and a minimum diameter of 8.71 mm, as shown in Fig. 2. This mirror was optimized with an X-ray photon energy of 539 eV and a 2 nm root-mean-square (rms) surface roughness. In other words, the surface roughness of the designed mirror should be at least 2 nm rms in order to achieve the desired optimal performance.

Image quality can be thought of as resolution or how close two objects can approach each other while still being distinguished from one another\(^7\). The theoretical performance of the designed mirror is given in Fig. 3 in terms of encircled energy which is energy percentage plotted as a function of image radius. The radius containing 80% of the energy on a detector is related to the resolution. The achievable resolution within a field height can be estimated by the radius (or diameter) and the magnification of the optic: that is approximately 2× radius/magnification. For a field height of 6.3 μm, for example, 80% of the energy falls within a radius of 0.8 μm. Thus, the estimated resolution is 50 nm within field of view of 6.3 μm in radius from the optical axis. As we expect, at on-axis the optical system shows a diffraction-limit image quality, and the most dominant contribution to image blurring at off-axis field height results from coma aberration. The theoretical performance of the designed-ideal mirror is considerably different to the field heights as shown in Fig. 3. We assume that alignment of optical components in an X-ray microscope is perfect.

The surface roughness of the X-ray mirror can affect image quality related to the contrast and should be also considered with link to X-ray reflectivity. Resolution of an X-ray image strongly relies on the figure accuracy of the mirror. In a theoretical point of view, we can simply estimate the required figure errors for the designed objective mirror using ray tracing technique. Figure errors of the mirror can be expressed with sinusoidal forms in axial and radial directions\(^6\). We consider only axial figure error because of its sensitivity in image degradation.

The ideal Wolter type I mirror, i.e., the perfect mirror, can resolve a fine structure about 50 nm within field of view of 6.3 μm in radius in spite of a low magnification for an ideal detector as mentioned previously, however surface deviation from the ideal figure rapidly destroys image quality. Figure 4 shows the relation between image resolution and amplitude of figure error in the axial direction for the designed mirror at zero and 6.3 μm in field height. The image quality, resolution, critically depends on the figure error when it is introduced to the mirror. The estimation of the required figure error is very useful in order for ex-
pecting the mirror performance. In order to obtain a 50 nm resolution image for a 6.3 μm field height, the amplitude of figure error in the axial direction is necessary for less than approximately 3 nm as shown in Fig. 4. When the amplitude of figure error makes an approach to zero in Fig. 4, the resolutions for on-axis and 6.3 μm in field height, i.e., diffraction-limit and 50 nm, are the same results obtained from Fig. 3. Actually, the axial figure error is approximately ten times more sensitive in image quality than that of the radial direction(6). Figure 4 was obtained from simulation results of ray tracing for an ideal detector.

Image resolution is determined by not only a mirror but also a detector used(7). When a CCD (charge-coupled device) detector with 13 μm × 13 μm pixel size, which is a common specification nowadays, is adopted in a soft X-ray microscope, the obtainable resolution of an X-ray image taken with the microscope system is restricted as approximately 400 nm, of which the resolution can be estimated by ray tracing simulation under the fixed 32 X magnification of the mirror, because of the limitation of pixel size even if the used mirror has no figure error, i.e., ideal mirror. In other words, the image resolution of the soft X-ray microscope is mainly determined by the CCD detector for fine structures of below 400 nm size. However, for objects of over 400 nm size the resolution is largely governed by the mirror, i.e., figure error of the mirror. A mirror point of view, a 400 nm resolution can be a reference to set the required figure error of the mirror. In order to obtain a 400 nm image resolution using the soft X-ray microscope, the axial amplitude of figure error of the mirror should be smaller than 27 nm as shown in Fig. 4. In a high magnification, the restriction of the resolution by the pixel size of the CCD detector can be released. In this case, the image resolution is governed by the figure error of the mirror mainly.

### 3. Precision Machining of an Electroless Nickel Mandrel

To fabricate the Wolter type I microscope mirror using an epoxy replication method, we prepared sample mandrels by two steps. First, we machined A7075 aluminum alloy samples to a thickness below 75 μm from the ideal surface contour, and 110 μm electroless nickel was then deposited on the samples. The plated electroless nickel contained about 10 weight percent phosphorous and showed amorphous structure. Machined surface and tool wear in SPDT depend on the structure of electroless nickel which is functions of amount of phosphorous and temperature of heat treatment(8).

The diamond turning lathe used in this experiment has two perpendicular slide tables (Z and X axes), an air bearing spindle on the Z-slide table, and a rotary table (B-axis) with a tool post on the X-slide table. The X and Z-slide tables are controlled within 1 nm positional step, and the B-table can be rotated with an angular step of 0.000 1°. However, in this experiment the B-table was fixed. Single crystal diamond tools with zero rake angle and 5° clearance angle were used for machining of the electroless-nickel-plated mandrels.

In order to obtain a smooth surface of a mandrel, the machining vibration by resonance(9) should be escaped and a slow feed rate is indispensable. Surface roughness can be achieved about from 2 nm to 3 nm rms under optimum cutting conditions: a cutting speed of 0.34 m/sec, a feed rate of 1 μm/rev, and a depth of cut of 1 μm for a 3 mm nose radius of a single crystal diamond tool. One mandrel shows an average of 2.45 nm Ra (or 2.93 nm rms) with a standard deviation of 0.48 nm (or 0.34 nm). The ellipsoid part gives slightly smoother surface than that of the hyperboloid. The mandrel used for fabrication of the Wolter type I mirror is given in Fig. 5. Figure 6 shows a surface topography, measured with an optical profiler. The surface roughness is 1.94 nm Ra (2.41 nm rms and 15.85 nm Ry). The surface was also obtained with the optimum cutting
The surface roughness of a diamond-turned mandrel was not sufficient for fabricating an X-ray mirror because of its rough surface. A rough surface results in strong X-ray scattering, and X-ray reflectivity shows exponential decay of the square of rms surface roughness. Thus, an X-ray mirror has to be smooth as at least the X-ray wavelength used. Since the Wolter type I mirror optimized with 2 nm rms in surface roughness showed good optical performance, we reduced the surface roughness of the master mandrel by hand-polishing to meet the required surface roughness of the aspheric mirror. The polishing was carried out in the vertical direction to tool marks. A Nomarski differential interference micrograph of a polished electroless nickel mandrel is shown in Fig. 8. The tool marks on the surface of the mandrel were disappeared. Surface roughness of the polished surface was improved as approximately 1 nm rms. The main contribution to surface smoothness is based on the removal of a high spatial frequency of 2.5 µm. However, the dominant waviness (a 120 µm in spatial wavelength) still remained.

In our polishing approach (hand-polishing), the removal rate of electroless nickel is very low, and high spatial frequencies in surface irregularity are removed. This fact can play an important role in figuring a master mandrel because polishing may frequently destroy the figure accuracy and figure control by polishing is not easy for an aspheric shape. SPDT can easily access to accurately generate an aspheric shape. The figure errors of a mandrel before (after SPDT) and after polishing were compared to each other and were almost the same as shown in Fig. 9. In our polishing approach point of view, the figure error of the mandrel is mainly determined by the step of SPDT. Thus, figuring of a mandrel by SPDT is very important.

Frankly speaking, our polishing approach also has latent possibility to destroy figure accuracy if sub-nanometer surface roughness is required to a mandrel. However, there is a trade-off between figure accuracy and surface roughness for the mandrel in the polishing step. The result of ray-tracing for figure error and the required surface roughness of the mirror were considered. Because
the surface roughness of 2 nm rms could be achieved by the polishing, figure accuracy had priority, and the polishing was carried out not to destroy the figure accuracy of the diamond-turned mandrel.

4. Fabrication of a Wolter Type I Mirror

As following epoxy replication processes\(^{[10]}\) we coated 300 nm-thick gold layer on the master mandrel and made a metal mirror substrate (steel or brass) by turning on a conventional lathe with a numerical controller, and then the coated master mandrel was inserted into the mirror substrate. The space between two objects, approximately 35 µm, was filled with epoxy resin. We separated the two objects by a cooling process after curing of the epoxy resin. The thin gold layer on the epoxy resin plays a very important role as a reflecting surface of soft X-rays and a parting material between the master mandrel and the mirror substrate. We successfully fabricated an aspheric mirror with axial symmetry using a master mandrel by the replication procedures. Figure 10 shows the structure of the replicated gold mirror.

It is difficult to measure the surface roughness and figure of the replicated mirror which has inner reflecting surface of a small size (diameter). A semicircular part of the mirror was fabricated to measure the surface roughness and figure through the same replication procedures with the same master mandrel to protect the deformation and the contamination of the mirror while parting the mirror. Figure 11 shows two-dimensional surface map of the replicated gold mirror. The surface roughness is 1.12 nm Ra (1.40 nm rms and 16.21 nm Ry) in the 360 × 270 µm\(^2\) area. The master mandrel, on the counterpart of the measured area of the replicated gold mirror, shows very similar surface roughness: 1.06 nm Ra, 1.32 nm rms, and 11.34 nm Ry. From the analysis of spatial frequency for surface profiles of the replicated gold mirror, it is found that a 120 µm spatial wavelength is also dominant as that of the master mandrel. This can be evidence that the epoxy replication method has excellence in copy ability.

Defects, two different kinds, can be found on the replicated gold mirror. One originates from the spikes of the master mandrel by defective polishing. In this sense, the finishing of the master mandrel is very important. These spikes are beyond the measurement ability of an optical microscope (Nomarski microscope). Thus, these spikes are not appeared on the Nomarski micrograph shown in Fig. 8. The other is brought about by large gold particles generated during the coating process. Some large gold particles occasionally remain on the master mandrel even after the separation process. Generally, the defects by large gold particles are smaller in height and width than those by the defective polishing. Figure 12 shows surface topographies for a replicated gold mirror and its counterpart of the master mandrel. The surface of the replicated gold mirror shown in Fig. 12(a) is displayed inversely in order to easily compare to that of the master mirror. Defects marked with “A” and “B” shown in Fig. 12(a) result from the defective polishing and the large gold particle, respectively. The counterpart of “A” exists on the surface of the master mandrel, while “B” is not found out on the master mandrel.

The shape accuracy of the replicated gold mirror is related to deformation (including contraction) and thermal hysteresis of the epoxy resin as well as figure error of the original master mandrel. We measured the shapes of the master mandrel and the replicated gold mirror using a contact type surface profiler. Figure 13 shows the figure errors of the replicated gold mirror and the master mandrel. The figure errors of the replicated mirror and the master mandrel are 144 nm P-V (peak-to-valley) (34 nm
(a) Replicated gold mirror

(b) Master mandrel

Fig. 12 Surface topography of a replicated gold mirror with defects of two different kinds (A and B) and its counterpart of the master mandrel

Fig. 13 Comparison of figure errors for the replicated gold mirror and the master mandrel

Fig. 14 One electroless nickel mandrel and Wolter type I aspheric gold mirrors fabricated by the epoxy replication method

drel which was already used in making a mirror still keeps the original surface roughness and shape, and again it can be used in fabricating another mirror through the same replication process. Figure 14 shows the replicated gold mirrors made of brass and steel substrates and the master mandrel.

5. Conclusions

We considered a Wolter type I mirror used in a soft X-ray microscope. The X-ray mirror requires very tight tolerances, such as super smooth surface and highly accurate figure, and has small diameter, thus it is not easy to fabricate the mirror. We successfully fabricated the aspheric mirror by the epoxy replication method. The results obtained in this study are as follows:

1. An electroless nickel plated mandrel was machined by SPDT, and finished the surface by polishing. Figure error of a master mandrel was mainly determined by the SPDT in which the surface roughness was not enough to fabricate a soft X-ray microscope mirror.

2. The Wolter type I mirror fabricated by the epoxy replication method showed that the surface roughness and the figure error were 1.12 nm Ra (1.40 nm rms) and 34 nm rms (±72 nm P-V), respectively.

3. Several gold mirrors were fabricated by using only one master mandrel without destroying its surface roughness and figure.

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