Development of highly-mechanically polished metal-substrate for neutron supermirrors

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Abstract.

A small-angle neutron scattering instrument using an ellipsoidal focusing mirror has been developed at Hokkaido University and a prototype machine (mfSANS) has been installed at the JRR-3 research reactor at Japan Atomic Energy Agency (JAER). They are based on an ellipsoidal focusing mirror on a borosilicate glass substrate. It turned out that borosilicate glass was very brittle and difficult to machine or polish it to the required surface-finishing process. In order to improve this situation, we decided to develop a method to create a metal substrate based ellipsoidal focusing mirror. As the first step, test pieces of flat and ellipsoidal surface neutron supermirrors using metal substrates were fabricated. They were first roughly shaped by a conventional numerically controlled (NC) cutting machine and amorphous NiP was plated. They were then machined using an ultrahigh precision cutting (UPC) method and finished by NC polishing techniques. Surface roughness of $R_a \approx 0.78$ nm and surface figure error of sub-micrometer were realized so far.

1. Introduction

A neutron supermirror is an extremely powerful and useful optical device used to handle neutron beams. Polished-glass, float-glass or polished silicon single crystal plates are usually used as neutron supermirror substrates. In only a few trials have neutron supermirrors been fabricated with metal substrate, although metal substrates can be very useful [1]. This is because surface roughness of less than about 0.3 nm is required for the coatings for high-m supermirrors and small d-spacing monochromators, which is not so easy to realize with most metallic materials due to the grain boundaries in their polycrystalline structure. Because NiP is a metal amorphous which has no such boundaries, we predict that we can polish the surface as smooth as a glass surface. Namba et al. fabricated a Wolter-type convex mirror for X-ray telescopes with sub-nanometer roughness using metal material [2]. The material used was aluminum alloy with electroless NiP plating on the surface and was fabricated by turning using a diamond tool. For Wolter-type mirrors, turning is applicable since the shape is axisymmetric. However, by using a shaper cutting process, aspherical profiles such
as ellipsoidal surfaces can be made. This research was conducted to develop a new fabrication method for aspheric mirror substrates using a metal material, particularly for use in aspheric neutron supermirrors such as used in a mirror focusing small-angle neutron scattering (SANS) instruments or in a focusing reflectometer.

The SANS technique [3] is a powerful tool for the study of various structures in the range of nanometer to micrometer scale. Typical conventional pinhole SANS instruments requires rather long instrument length of about 20-30 m. However, by using focusing optics, the beam diameter on the detector can be reduced to the order of a few millimeters and can therefore reduce the instrument length while maintaining angular resolution. Furthermore, it can be realized without reducing the sample size, therefore not sacrificing intensity. For time of flight (TOF) SANS instruments, shorter instruments result in a wider wavelength band, especially to a longer wavelength range. It is therefore possible to measure a low momentum transfer (Q) region while maintaining intensity.

The mini-focusing small-angle neutron scattering instrument (mfSANS) is a SANS instrument that employed an ellipsoidal neutron focusing mirror with supermirror coating. The basic concept of mfSANS is to make a SANS instrument short, about one-tenth of the conventional SANS instruments, while covering the Q range by application of a focusing optical system. The mfSANS instrument was first developed at Hokkaido University using the Hokkaido University Neutron Source (HUNS). Later, a prototype has been installed at the research reactor (JRR-3) at the Japan Atomic Energy Agency (JAEA). Figure 1 shows a schematic layout of mfSANS.

![Figure 1. Schematic layout of mfSANS: The neutron beam passes through an incident aperture, which is set at one of the focal points. It is then reflected by a focusing mirror which focuses it around the other focal point downstream. It is possible to cover the conventional Q range of a typical SANS instrument together with a larger Q range.](image)

| Mirror specifications |
|-----------------------|
| Shape                 | Ellipsoid |
| Radius                | Major: 1250 mm Minor: 20 mm |
| Size                  | 3 segments each 300 mm × 60 mm × 9 mm (Full length × 900 mm) |
| Machining method      | Grinding + Polishing |
| Machining time        | 6 months |

The mfSANS instrument yielded reasonable results, especially in the wide-Q range of up to about 5 nm\(^{-1}\) [4]. However, the background level at the small-Q region was rather high due to the diffuse scattering from the mirror. Beam profile deformation caused by misalignment of the mirror segments was also observed. Both problems are related directly to the surface condition of the ellipsoidal mirror. The ellipsoidal mirror installed in the prototype of mfSANS was made of borosilicate glass. Due to the size limitations posed by the coating machine, the mirror was divided into three segments. Each segment was 300 mm long. The surface figure error was 5.0 μm Peak-to-Valley (P–V) for the minor axis direction and 7.5 μm P–V for the major axis direction with 39.7 μm difference in level between the mirror segments. The surface roughness was about 1.0 nm Root-Mean-Square (RMS). This was reasonable for grinding conducted using a #800 grinder. It was planned to use a #1200 grinder, which was cancelled due to machine failure. The machining process was 6 months for grinding and polishing the whole mirror, all three segments in this case, including the time for tool adjustments, such as truing and exchanging polishing tools.
By using a NiP plated metal substrate and applying ultra-high precision cutting (UPC) and polishing methods, there is a hope of making an aspherical mirror while drastically reducing the machining time and maintaining very good surface finish of sub-nm level. By using a metal substrate, it also enables the production of a mechanical fixture directly to the substrate to improve the alignment between the mirror segments. The main objective of this paper is to present the validity of the machining method described above for creating aspherical mirror substrates for neutron mirrors. We fabricated test pieces of two types: a flat and an ellipsoidal test pieces.

2. Fabrication of test pieces
All test pieces were machined from bulk blocks of metal. The UPC method was used, whereby cutting was conducted from only one direction. Surface roughness was characterized by using a white light interferometer, and neutron reflectivity, as described later in this paper.

2.1. Flat test pieces
Stainless steel was used as the substrate for flat test pieces. They were then roughly finished using a conventional numerically controlled machining process with subsequent electroless NiP plating with 100 μm thickness. The dimensions of the test pieces were 50 mm × 50 mm × 13 mm. The plated NiP surface was then roughly finished by turning with a single crystal diamond bite with subsequent machining by UPC. The machine used for the turning and UPC processes was the NIC-300 from NAGASE INTEGREX. As the final process, the surface was finished using a numerically controlled mechanical polishing instrument with colloidal silica as the slurry. The machining time was 13 hours for UPC and 24 hours for mechanical polishing.

2.2. Ellipsoidal mirror test pieces
The base material was reviewed as aluminum alloy for an ellipsoidal mirror test piece. The dimensions of the substrate were 100 mm × 30 mm × 10 mm, as shown in Figure 3(a). The base substrate was roughly shaped using a machining center with a ball-end mill. After creating a rough ellipsoidal surface by milling, the surface was finished by the UPC method. The same machine and machining method was applied as the flat test pieces for the UPC process. Both machining processes required calculation of the tool offset. The machining time was 3 hours for rough shaping and 13 hours for UPC.

3. Surface roughness and surface figure error
The surface roughness was measured using a white light interferometer (new view 7200; Zygo). The measured area was 140 μm × 100 μm, with spatial resolution of 100 nm × 100 nm. The surface profile was measured using a Point Autofocus Probe Surface Texture Measuring Instrument (PF-60; Mitaka Kohki. Co., Ltd.). The laser spot diameter was 1 μm; the pitch between each point was 10 μm. Figure 2 shows the results of surface roughness and the surface profile measured by the interferometer after UPC and mechanical polishing. For the sample finished by UPC, the roughness was 1.3 nm [rms] in the cutting direction and 3.8 nm [rms] for the plane. Horizontal cutting marks were observed. For the sample finished by polishing, the sample roughness after 24 hours of mechanical polishing was 0.78 nm for the plane [rms]. The cutting marks were no longer visible. Figure 3 shows the picture of the ellipsoidal test piece and the surface figure error after the UPC process. The surface figure error was 0.7 μm P–V for the major axis and 3.0 μm P–V for the minor axis.

4. Reflectivity measured by neutrons
In order to determine whether the surface roughness of the flat test piece is adequate, we conducted a neutron reflectometry experiment at the CN3 beam port at the Kyoto University Research Reactor Institute (KUR-CN3) using flat test pieces with different finishing conditions. The samples were flat test pieces with the NiP plated surface finished by (a) UPC and (b) polishing. Sample (a) was set so that the neutron beam is parallel to the cut marks.
Figure 2. (a) Diagram of the flat test piece. (b) Sample surface profile after UPC. Roughness was 1.3 nm [RMS] in the cutting direction and 4.14 nm [RMS] for the plane. Horizontal cutting marks are visible. (c) Surface profile of the sample after polishing. The surface roughness after 24 hours of mechanical polishing was 0.78 nm [RMS] for the plane. Cutting marks are no longer visible.

Figure 3. (a) Picture of the ellipsoidal test piece. (b) Surface figure error of the major axis was 0.7 μm P–V. (c) Surface figure error of the minor axis was 3.0 μm P–V.

4.1. Geometry of neutron reflectometry experiment

Figure 4 shows a schematic drawing of the geometry of the reflectometer at KUR-CN3 reactor-based neutron source. Consequently, the energy spectrum of the neutron beam was measured by application of a beam chopper combined with the time of flight (TOF) method and a time sensitive 2D position sensitive detector. The wavelength resolution was 10% for 0.2 nm neutrons. The sample was set on a goniometer used to adjust the incident angle of the beam, which was collimated using a set of vertical slits. The reflectivity and beam profile were measured using the 2D position sensitive detector.
4.2. Results of neutron reflectometry experiment

4.3. Reflectivity was measured only for flat test pieces. The measurement time was 2 hours. The reflectivity and intensity of diffuse scattering is shown in Figure 5. Diffuse scattering is visible on the figure showing the horizontal projection of the beam profile. Diffuse scattering is visible from $5.0 \times 10^{-2}$ for (A); the test piece machined only with UPC, but is not visible for (B); the test piece after polishing, within the level of background.

Figure 5. Neutron reflectivity: (a) 2D map of the incident and reflected beam. (b) Vertical projection of the beam profile normalized by peak height.
5. Discussion

The surface figure error in the major axis direction is already better than the previous ellipsoidal mirror, which was in the order of several micrometers. The surface roughness we achieved for the flat test pieces could be improved by optimizing the polishing conditions such as the polishing pressure, type of slurry, and polishing speed. The surface figure error could also be improved using a diamond bite with better shape accuracy. Based on these results, the fabrication method we applied is considered feasible for creating a substrate with better surface figure error and surface roughness compared to the former ellipsoidal mirror used in mFSANS. The intensity of diffuse scattering from the flat test piece was reduced by finishing the surface by polishing, but it was too small to be evaluated quantitatively using our current reflectometer. This resulted from the low signal to background ratio, which was partially attributable to the small incident angle and the beam chopper for monochromization.

6. Conclusion

We fabricated flat test pieces with electroless NiP coating on metal blocks. They were then machined by UPC and polished and the surface roughness we achieved this time was as low as 0.78 nm [rms]. Diffuse scattering was measured by neutron reflectometry. The sample finished by polishing exhibited lower diffuse scattering than the one finished by UPC, but it was not evaluated quantitatively because of its low signal–background ratio of the reflectometer.

We also fabricated ellipsoidal test pieces using the same method. The surface figure error was quite good, 0.7 μm P–V for the major axis and 3.0 μm P–V for the minor axis.

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