High-precision figured thin supermirror substrates for multiple neutron focusing device

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Abstract. An aspherical supermirror is one of the most useful neutron-focusing optics. We aim to develop multiple aspherical supermirror devices using high-precision figured aspherical focusing supermirrors to focus neutron beams with high intensities, because multiple mirrors collect a very large beam divergence. Thin mirrors with a millimeter thickness are required to minimize the absorption loss of incident neutron beams since the thickness of a mirror shadows the reflective area of the other mirrors. However, it is difficult to fabricate thin mirror substrates with a form accuracy of sub-micrometer level by conventional machining. Conventional machining deforms a substrate by machining force and spring back after machining causes figure error. Furthermore the deposition of supermirrors deforms the mirror substrate by film stress. Thus, we developed a new process of fabricating a precise millimeter-thick elliptical supermirror. This process consists of noncontact figuring by the numerically controlled local wet etching technique and the ion beam sputter deposition of NiC/Ti multilayers on both sides of the mirror substrate to compensate for film stress. In this paper, we report on the fabrication results and focusing performance of elliptical supermirrors with a thickness of 1.5 mm.

1. Introduction

Recently, various measurements using neutrons have been of considerable practical interest because of their ability to identify hydrogen atoms and water molecules precisely, sensitivity to magnetism and penetrating power. High-intensity proton accelerator facilities, such as J-PARC in Japan, ISIS-TS2 in UK and SNS in USA, are constructed for neutron research. However, neutron intensity is very low compared with that of X-ray in synchrotron orbital radiations, even in these latest neutron sources. Therefore, high-performance optical devices for focusing neutron beams are required to realize practical measurements. Reflective aspherical supermirror optics would be advantageous for focusing neutron beams with a wide wavelength range since it has no chromatic aberration. Neutrons can be focused within a small area of less than 1 mm² using a high-performance elliptical supermirror with a high form accuracy and a low roughness at both the substrate surface and the multilayer interface. Therefore, reflective optics using an elliptical supermirror improves S/N ratio, reduces measurement time and can be applied to a wide range of neutron experiments, such as the diffraction-based measurement of small biomolecules and position-sensitive prompt gamma analysis, at neutron reactors and pulsed neutron sources.
We developed a numerically controlled local wet etching (NC-LWE) technique[1] to figure aspherical mirror substrates and an ion beam sputter deposition technique [2] to deposit a NiC/Ti multilayer for the realization of usable aspherical supermirror optics. Previously, we fabricated a plano-elliptical supermirror with a clear aperture size of 90 x 40 mm\(^2\) by applying these techniques, and obtained a focusing gain of 6 and a reflectivity of 0.64-0.7 in the near-critical-angle region for \(m=4\) [3,4]. The m-value indicates the ratio of the effective critical angle of the supermirror to that of natural nickel.

To increase the focusing gain and reflectivity, a decrease in surface roughness and the enlargement and multiplexing of supermirrors are essential. Previously, we succeeded in developing a fabrication process for an aspherical mirror substrate with a large clear aperture size [5]. In the case of the multiplexing of supermirrors, mirror substrates must be thin to reduce the absorption loss of incident neutron beams. Previously, a Kirkpatrick-Baez mirror consisting of orthogonal stacks of reflectors for an X-ray telescope was reported [6]. However, the figure accuracy of the mirror has not been referred to because the weight of the mirror is more important than its shape in space X-ray optics. On the other hand, the figure accuracy of the mirror is most significant for the beam width in neutron focusing optics. It is difficult to fabricate thin mirror substrates with a form accuracy of sub-micrometric level and a surface roughness of sub-nanometric level because conventional precision grinding deforms the workpiece by machining force and spring back after grinding causes figure error. In addition, conventional polishing for reducing surface roughness increases figure error. Furthermore, the deposition of supermirrors deforms the mirror substrate by film stress. As a fabrication technique for SiC mirrors with a millimeter thickness, ELID grinding with a feedback compensation method has been reported. In this method, the tool path is determined from numerical calculation results of the profile deformation analyzed by FEM [7].

On the other hand, the NC-LWE technique is very suitable for fabricating an ultraprecise millimeter-thick mirror substrate, because such a technique enables us to fabricate an aspherical shape without processing pressure to the workpiece and wear of machine tools because of its noncontact removal mechanism utilizing a chemical reaction. Moreover, a high reproducibility is also achieved owing to the insensitivity of such a technique to external disturbances, such as vibration or thermal deformation. Thus, the NC-LWE technique is very feasible for fabricating an ultraprecise millimeter-thick mirror substrate with a high reproducibility. We developed a new process of fabricating a precise millimeter-thick elliptical supermirror. This process consists of noncontact figuring by the NC-LWE technique, the minimization of surface roughness without degrading form accuracy by low-pressure polishing with a polishing pressure less than about 7 kPa (1 psi) and the ion beam sputter deposition of NiC/Ti multilayers on both sides of mirror substrate surfaces to compensate for film stress.

In this work, we fabricated and evaluated a plano-elliptical neutron focusing supermirror with a thickness of 1.5 mm.

2. Multiple neutron focusing device

An aspherical supermirror is one of the most useful optics for focusing neutrons with a wide wavelength range. Multiple aspherical supermirror devices using high-precision figured aspherical focusing supermirrors have received considerable attention with the aim of focusing neutron beams with high intensities, because multiple mirrors collect a very large beam divergence. Thin mirrors with a millimeter thickness are required to minimize the absorption loss of incident neutron beams since the thickness of a mirror shadows the reflective area of the other mirrors.

2.1. Optical design of multiple mirror system

To increase the focusing gain, it is effective to multiplex the supermirror. We designed a multiple mirror neutron focusing system to be applied to reflective small angle scattering measurements. Figure
1 shows the optics design of the multiple mirror neutron focusing system. This focusing system consists of 4 mirrors with elliptical shapes represented by

$$\frac{x^2}{a^2} + \frac{y^2}{b^2} = 1 \text{(unit: mm)}$$

The a and b values of each mirror are shown in Table 1. The thickness of each mirror substrate is 1.5 mm and the clear aperture size is 100 x 50 mm$^2$. The magnification of the mirrors and the focal length and incident angle of the outmost mirror center are 1:1, 1050 mm and 1.4°, respectively.

2.2. High-precision figured thin elliptical supermirror

Thin mirrors with a millimeter thickness are required to minimize the absorption loss of incident neutron beams since the thickness of a mirror shadows the reflective area of the other mirrors. However, it is difficult to fabricate thin mirror substrates with a form accuracy of sub-micrometer level by conventional machining. Conventional machining deforms the substrate by machining force and spring back after machining causes figure error. Furthermore, the deposition of supermirrors deforms the mirror substrate by film stress.

We developed a new process of fabricating a precise millimeter-thick elliptical supermirror. This process consists of noncontact figuring by the NC-LWE technique and the ion beam sputter deposition of NiC/Ti multilayers on both sides of mirror substrate surfaces to compensate for film stress.

In this work, we fabricated the mirror #4 shown in Table 1 by the NC-LWE technique. The material and outer size are synthesized quartz glass and 150 x 150 x 1.5 mm$^3$, respectively. In the NC-LWE technique, figuring is performed by controlling the dwelling time of the nozzle head. Because of its noncontact chemical removal mechanism, the NC-LWE technique enables us to fabricate a millimeter-thick mirror substrate without generating subsurface damage and deformation. As the processing parameters in NC-LWE figuring, we used 37 wt% HF aqueous solution with a temperature of 42°C, a circular nozzle with a diameter of 15 mm and a feed pith of 0.5 mm.

In NC-LWE figuring, raster-type numerically controlled scanning is applied to correct the residual figure error. After raster scanning, shallow periodic grooves, which are similar to the tool marks formed in conventional lathe turning, are inevitably generated on the figured surface by the overlapping of the scanned etching path. We have reported a method of decreasing the height of the tool marks by a two-stage NC-LWE process [3]. However, in NC-LWE figuring with raster scanning, the tool marks cannot be completely eliminated. Low-spatial frequency roughness (LSFR: $\lambda_s > 1$ mm), such as that caused by tool marks, degrades the focusing beam profile. To eliminate LSFR, we applied a first polishing process with a hard polishing pad.

Additionally, the mid-spatial frequency roughness (MSFR: 1 mm > $\lambda_s > 1$ μm) and high-spatial frequency roughness (HSFR: 1 μm > $\lambda_s > 1$ nm) components of the processed surface tend to increase after NC-LWE figuring. The MSFR and HSFR components are one of the causes of the decrease in the reflectivity of the supermirror. Subsurface damage is considered as one of the causes of the increases in MSFR and HSFR because the etching rate of the damaged layer is different from that of an undamaged layer. Thus, it is essential to minimize the surface roughness to obtain a higher
reflectivity and thus realize the practical application of the neutron focusing mirror. Therefore, a second polishing process using a soft polishing pad was applied to improve the MSFR and HSFR of the figured mirror substrate.

The deposition of supermirrors deforms the mirror substrate by film stress. In particular, the film stress with a GPa level generated by ion beam sputter deposition deforms the mirror substrate with a millimeter level thickness. We performed the ion beam sputter deposition of NiC/Ti multilayers on both sides of mirror substrate surfaces to compensate for film stress. A NiC/Ti supermirror with \( m=3 \) was fabricated. The layer sequence of the deposited supermirror was designed using the algorithm proposed by Hayter and Mook. The thickness, total number of multilayers and size of deposition were 3 \( \mu m \), 400 and 110 x 60 mm\(^2\), respectively.

2.3. Evaluation of focusing performance and reflectivity

We evaluated the focusing performance of the fabricated mirror at pulsed neutron instrument with disc chopper (CHOP) in Japan Research Reactor No. 3 Modified (JRR-3M) of Japan Atomic Energy Agency. Figure 2 shows a schematic of the setup for measuring the neutron beam profile. The wavelength of the neutron beam ranged from 3 to 10 \( \AA \). The width of the slit in this experiment was 0.25 mm. The magnification of the optical design was 1. The beam profiles were measured by using an imaging plate.

The reflectivity of the fabricated supermirror was evaluated at the Neutron Multilayer Interferometer Reflectometer 2 (MINE2) of JRR-3M. The wavelength of the monochromatized neutron beam was 8.8 \( \AA \). The meridional direction of the mirror under test was set vertically, and the neutron beam defined by two slits was irradiated in the sagittal direction, in which the test mirror has no curvature.

![Figure 2. Schematic of the setup for measuring the neutron beam profile at CHOP.](image)

![Figure 3. The surface roughness of the mirror substrate. (64 x 48 \( \mu m^2 \))](image)

3. Results and discussion
The residual figure error after NC-LWE was about 0.2 μm. The shape of the mirror substrate was measured with a laser autofocus measuring machine (Mitaka Kohki NH-3SP, z resolution: 1 nm). This result indicates that the noncontact removal by controlling the dwelling time of the etchant nozzle deterministically figures the aspherical shape with a form accuracy of sub-micrometric level.

Figure 3 (a) and (b) show the surface roughnesses of the figured mirror substrate after NC-LWE correction and second polishing, respectively. The surface roughness was measured using a scanning white-light interferometer (Zygo NewView 200CHR). Many pits were observed on the surface after NC-LWE correction, as shown in Figure 3 (a), and the rms surface roughness was 4.6 nm. To remove these undesirable surface structures, a soft polishing pad was used in the second polishing process with a removal depth of about 40 nm. Figure 3 (b) indicates that the surface morphology was improved and an rms roughness of less than 0.15 nm was obtained by applying the second polishing process. It also indicates that the second polishing process is very effective for improving the overall surface roughness of the mirror substrate in the MSFR regions.

In general, the application of a polishing process to improve the surface roughness degrades the form accuracy because it is difficult to maintain a constant removal rate over the area of the clear aperture of the mirror. The residual figure error after the second polishing process was about 0.2 μm p-v. The low-pressure polishing process has the ability to improve the surface roughness satisfactorily while maintaining a sub-micrometer form accuracy. These results indicate that low-pressure polishing is very effective for improving the surface roughness without degrading the shape of the mirror substrate. We deposited a NiC/Ti supermirror with $m=3$ on both sides of the figured mirror substrate by ion beam sputter deposition. Figure 4 shows the residual figure error observed after deposition. If one side of the mirror substrate is deposited, film stress deforms the mirror substrate with a deformation amount of 50-60 μm. However, we succeeded in inhibiting deformation less than 1 μm p-v by depositing NiC supermirror on both sides of the figured mirror substrate. Figure 5 shows the surface roughness observed after deposition. The rms roughness of the supermirror was 0.2 nm, thus maintaining the surface roughness of the mirror substrate.

| Table 1. Optical parameters of neutron focusing mirrors |
|-------------------------------------------------------|
| Mirror #1   | $\theta=1.40$ (degree), $a=1050.31$, $b=25.66$ (mm) |
| Mirror #2   | $\theta=1.19$ (degree), $a=1050.23$, $b=21.83$ (mm) |
| Mirror #3   | $\theta=1.00$ (degree), $a=1050.16$, $b=18.34$ (mm) |
| Mirror #4   | $\theta=0.83$ (degree), $a=1050.11$, $b=15.17$ (mm) |
| Focal length| 1050 mm                                              |

Figure 4. Figure errors of the fabricated mirror in meridional direction. Figure 5. The surface roughness of the supermirror. (64 x 48 μm²)
These results suggest that the application of the combined fabrication process consisting of NC-LWE figuring, low-pressure polishing and ion beam sputter deposition is a highly effective method for fabricating a high-precision millimeter-thick aspherical supermirror with a form accuracy of sub-micrometric level and a surface roughness of sub-nanometric level. Figure 6 shows the beam profiles of the focused and non-focused beams. The full width at half maximum (FWHM) of the focused beam was 0.21 mm. The focusing gain of 12 in peak intensity was achieved compared with that in the case of the nonfocused directly irradiated beam, and the peak intensity was more than 100-fold higher than background intensity. These results indicate that our thin elliptical supermirror has a high figure accuracy for focusing neutron beams with sub-millimeter focusing widths.

The result of the $\theta$2$\theta$ scan is shown in Figure 7. For the performance of neutron reflection, the critical angle for $m=3$ ($q_z=0.65$ nm$^{-1}$) was achieved. This result shows that the designed d-space distribution of the NiC/Ti multilayer was precisely formed on the elliptically figured substrate. The reflectivity in the near-critical-angle region was more than 0.8. This result suggests that the fabricated supermirror has an interface roughness of sub-nanometer level.

The above results indicate that the fabricated millimeter-thick aspherical supermirror has the ability to focus neutron beams with sub-millimeter widths and high reflectivities. In our future study, we will attempt to fabricate and evaluate a multiple mirror system using a millimeter-thick aspherical supermirror.

4. Conclusions

We fabricated a plano-elliptical neutron focusing supermirror with a thickness of 1.5 mm by using a combined fabrication process consisting of NC-LWE figuring, low-pressure polishing and ion beam sputter deposition. We succeeded in obtaining a figure error of 0.82 $\mu$m p-v with a surface roughness of less than 0.15 nm rms, a FWHM of 0.21 mm and a focusing gain of 12 in peak intensity with a reflectivity of more than 0.8 in the near-critical-angle region.

Acknowledgements

This work was partially supported by a grant for “Development of High-Precision Aspherical Supermirror for Neutron Focusing”, “Development of System and Technology for Advanced Measurement and Analysis (SENTAN)” from Japan Science and Technology Agency (JST), the “Industrial Technology Research Grant Program in 2005” from the New Energy and Industrial Technology Development Organization (NEDO) of Japan, a Grant-in-Aid for the Global COE Program “Atomically Controlled Fabrication Technology” from the Ministry of Education, Culture,
Sports, Science and Technology, Japan, and Grant-in-Aid for JSPS Fellows. The authors would like to thank Masahiro Hino, PhD for assistance with experiments at MINE2 and Kazuya Aizawa, PhD and Shinichi Takata, PhD for assistance with experiments at CHOP.

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