Fabrication of plano-elliptical neutron focusing supermirror by numerically controlled local wet etching with ion beam sputter deposition

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Abstract. High-performance optical devices with a figure accuracy of sub-micrometer level and a surface roughness of atomic level are required to collect and/or to focus a neutron on a sample without scattering loss. To fabricate a high-performance neutron focusing supermirror, a two-stage numerically controlled local wet etching technique combined ion beam sputter deposition was developed. By applying this technique, a plano-elliptical substrate made of synthesized quartz glass with a figure accuracy of less than 0.5 µm was fabricated, and its surface was deposited with NiC/Ti multilayers by ion beam sputtering to produce an m=4 supermirror. The focusing performance of the supermirror was evaluated at the SUIREN of JRR-3M, and a focusing gain of 6 in peak intensity was achieved compared with a nonfocused direct beam.

1. Introduction

In recent years, very large proton accelerator facilities, such as J-PARC in Japan, ISIS in the UK and SNS in the USA, have been constructed. These facilities generate various quantum particles, including neutrons, by nucleus spallation. Neutron diffraction and scattering techniques are very useful for examining the physical, chemical, mechanical and magnetic properties of a material. However, the neutron intensity is very weak compared with synchrotron orbital radiation intensity, even in these facilities. Therefore, high-performance neutron collecting or focusing optical devices are required [1]. In these quantum optical devices, a figure accuracy of sub-micrometer level and a surface roughness of atomic level are essential for collecting and/or focusing neutrons onto a sample without scattering loss. However, in conventional machining processes, such as lapping or polishing, the accuracy of machining strongly depends on the stiffness of the machining equipment used and is also affected by external disturbances, such as the vibration or thermal deformation of the workpiece and the machine caused by the contact-removal mechanism. Therefore, it is very difficult to fabricate an ultraprecise optical device with a figure accuracy of sub-micrometer level with a high reproducibility.

To resolve these issues, we have developed a numerically controlled local wet etching (NC-LWE) process, which involves forced suction of an etchant with its volatile component using a vacuum pump, to fabricate an ultraprecise optical device [2]. The forced suction nozzle makes it possible to obtain a free configuration of the nozzle direction and prevents unwanted surface roughening by the adsorption...
of the volatile component of the etchant. Using this figuring system, although the cost of introduction is very low, highly stable and highly reproducible figuring properties are achieved more easily than when using other conventional machining systems, because the LWE process is insensitive to external disturbances, such as vibration or thermal deformation, owing to its noncontact chemical removal process.

2. Experimental procedure

2.1. Two-stage NC-LWE process

A plano-elliptical mirror substrate for focusing a neutron beam was fabricated by the NC-LWE figuring technique. The shape of the elliptical mirror is represented by equation (1).

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\frac{x^2}{(1050.31)^2} + \frac{z^2}{(25.6)^2} = 1 \quad \text{(unit: mm)}
\] (1)

The material, outer size, focal length and clear aperture size of the mirror substrate were synthesized quartz glass, 100 mm × 50 mm × 15 mm, 1050 mm, and 90 mm × 40 mm, respectively. The composition and temperature of the etchant were 37 wt% HF and 40°C, respectively. The curvature in the sagittal direction of this mirror was zero, and the neutron beam was focused one-dimensionally at the same magnification. The two-stage NC-LWE process was applied to fabricate a plano-elliptical mirror substrate. In this process, coarse figuring by one-dimensional numerically controlled scanning was performed using a large rectangular nozzle head with a size of 51 mm × 12 mm, then two-dimensional numerically controlled raster scanning was performed using a circular nozzle head with a diameter of 15 mm to correct the residual figuring error [3]. Therefore, this novel figuring process makes it possible to fabricate an ultraprecise mirror substrate with a high efficiency.

2.2. Ion beam sputter deposition

The surface of the plano-elliptical substrate was deposited with NiC/Ti multilayers using ion beam sputtering (IBS) to fabricate a supermirror for increasing the critical angle of neutron reflection. The IBS system installed at the Japan Atomic Energy Agency (JAEA) had an effective deposition area of 0.2 m² (diameter of 500 mm) [4-6]. The NiC layer was obtained by Ni-C cosputtering [7]. The carbon atoms added into the nickel layer suppressed interfacial roughness by reducing the crystal grain size of the nickel layer. A NiC/Ti supermirror with \(m=4\), where \(m\) is the ratio of the effective critical angle of the supermirror to that of natural nickel, was fabricated. The thickness and total number of multilayers were 6 µm and 1201, respectively.

2.3. Evaluation of focusing performance

The focusing performance of the fabricated supermirror was evaluated at the Apparatus for Surface and Interface Investigations with Reflection of Neutrons (SUIREN) of Japan Research Reactor No. 3 Modified of Japan Atomic Energy Agency (JRR-3M).

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Figure 1. Schematic diagram of the measuring setup at the SUIREN. (Top view)
Figure 1 shows a schematic diagram of the setup for measuring the neutron beam profile at the SUIREN. The wavelength of the monochromatized neutron was 0.393 nm. The sagittal plane of the mirror was installed vertically on the goniostage to focus the neutron beam horizontally. The mean incident angle of the neutron beam was 1.40 degrees. Slit-2 was placed at the focal point of the ellipse, and the imaging plate or the $^3$He detector was placed at the other focal point. The divergence of the neutron beam in the horizontal direction was set to 0.12 degrees by the combination of slit-1 and slit-2 to irradiate the neutron on the effective area of 90 mm length. The vertical divergence of the neutron beam was 0.39 degrees. The two-dimensional images of focused and nonfocused direct neutron beams were measured using an imaging plate (FUJIFILM BAS-ND; resolution: 50 $\mu$m), and detailed profiles of the beam were measured with a $^3$He detector (Eurisys 43NH10/5X) and slit-3. The width and scanning pitch of slit-3 were 0.25 mm and 0.1 mm, respectively.

3. Results and discussion

Figure 2 shows the figure errors of the fabricated mirror in the meridional direction measured with a laser autofocus measuring machine (Mitaka Kohki NH-3SP; z resolution: 1 nm). The two-stage NC-LWE process achieved a figure error of less than 0.5 $\mu$m with a total processing time of 318 min. However, the figure error degraded to about 1 $\mu$m after the deposition of the NiC/Ti multilayers. It seems that the compressive stress of the deposited thin film deformed the substrate, which may be prevented by depositing films of the same thickness on both sides of the mirror substrate.

Figure 2. Figure errors of the fabricated plano-elliptical mirror.

Figure 3. Neutron beam images detected by the imaging plate.
Figure 3 shows the two-dimensional images of the neutron beam detected using the imaging plate. Cold neutrons were uniformly focused at the effective mirror width of 40 mm. The neutrons detected beyond the mirror width were generated by the vertical divergence of the neutron beam. Figure 4 shows the detailed profiles of both focused and nonfocused direct neutron beams measured by the scanning of slit-3 placed in front of the $^3$He detector. The full width at half maximum (FWHM) of the focused beam was 0.35 mm, and a focusing gain of 6 in peak intensity was achieved compared with the nonfocused direct beam.

![Figure 4. Neutron beam profiles measured by scanning of the slit-3.](image)

4. Conclusions

A neutron focusing supermirror was fabricated by applying an NC-LWE technique combined ion beam sputter deposition. A plano-elliptical mirror substrate made of synthesized quartz glass with a clear aperture size of 90 mm × 40 mm was fabricated by applying a two-stage NC-LWE process. A large rectangular nozzle head and a small circular nozzle head were used in the first coarse figuring and second fine finishing stages, respectively, to reduce the fabrication time. The figure accuracy of the plano-elliptical substrate of less than 0.5 µm was achieved after NC-LWE figuring with a total fabrication time of 318 min. A supermirror with $m=4$ was produced by depositing of NiC/Ti multilayers on the substrate. The uniform one-dimensional focusing of cold neutrons and a focusing gain of 6 were achieved using the fabricated elliptical supermirror.

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