Fabrication of 3D micro/nanostructures using proximity effect between mask and resist

Fumiki Kato*
Utako Takagi**
Masaki Koshiishi***
Ikuyuki Mitsuishi***
Yuichiro Ezoe**
Kazuhisa Mitsuda***
Susumu Sugiyama*

ABSTRUCT

This is the first time introduction of an X-ray total reflection mirror chip fabricated by using synchrotron radiation (SR) lithography. In this paper, the mirror chip with smooth side wall was introduced. The mirror chip was fabricated by deep X-ray lithography (DXRL) process technique and nickel electroforming process technique. The small size mirror chip was successfully obtained which overall dimension was $7.5\times7.5\times0.2~\mu\text{m}^3$ with 50 μ m-width slit. The side wall rms roughness of mirror chip that is the mirror surface was measured by AFM and its result was 12 nm at $5\times5~\mu\text{m}^2$ area.

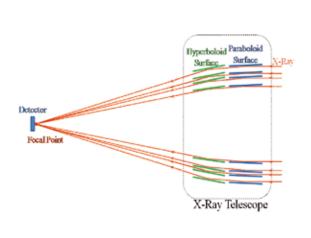
^{*} Faculty of Micro System Technology, Ritsumeikan University 1-1-1, Nojihigashi, Kusatsu, Shiga 525-8577

^{**} Department of Physics, Tokyo Metropolitan University, Minami-Osawa 1-1, Hachioji, Tokyo 192-0397

^{***} Department of High Energy Astronophysics, Institute of Space and Astronautical Science (ISAS)/ The Japan Aerospace Exploration Agency (JAXA) 3-1-1, Yoshinodai, Sagamihara, Kanagawa 229-8510

1. INTRODUCTION

To develop light-weight grazing incidence X-ray optics sufficient with scientific needs, such as a large effective area, a wide field of view and high resolution, under the limited circumstances of satellite resources is required for advancing astronomy [1]. Small MEMS chip of X-ray total reflection mirror can provide one solution. The MEMS mirror chip that is introduced in this paper can shrink dimension of X-ray optics drastically. It is known that the total reflection is occurred when X-ray beam strikes a medium boundary at an angle larger than the critical angle with respect to the normal to the surface. An X-ray optics system can be manufactured by combining the grazing mirrors using total reflection phenomenon, as shown in figure 1. The X-ray telescope consists of two sets of mirrors. One has paraboloid surface mirrors and another has a hyperboloid one. The X-ray reflected in the mirrors is focused on the surface of X-ray detectors. Conventional X-ray telescope configurations use many nested ring-shaped mirror shells which consist of polished mirrors, replicated mirrors or foil mirrors, to provide a large collection area for X-ray collection and imaging. In case of these traditional methods, weight of system becomes heavy and it costs high both of manufacturing a telescope and launching a satellite. The weight of the mirror per same opening area is as shown in figure 2. If a MEMS mirror chip is realized, the weight of X-ray telescope system will become light drastically.



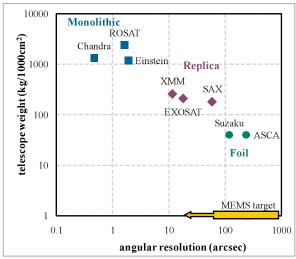


Fig. 1 Schematic of Wolter type-I X-ray optics

Fig. 2 Schematic of sub-wavelength structure

In order to reduce the weight of a mirror, the device by the mirror chip using MEMS technology was devised ^[2, 3]. The MEMS mirror chip can reduce mirror weight because of it can shrink the volume (three dimensions). To keep the same effective area, the mirror number should be increased; in fact that should be increased the density (two dimensions) of mirror slit. So the mirror weight is to be reduced. Two types of micropore optics are known and studied. One is the lead-grass microchannel plate (MCP) ^[4]. The spherically slumped square-pore MCPs are used to form lobster-eye optics, a class of imaging device consisting of an array of square pores on spherically curved surfaces. The other type is the recently proposed high-resolution pore optics (HPO) utilizing mirror-polished silicon wafers ^[5].

It was proved that silicon micro pore optics that the mirror chips made by anisotropic wet etching could reflect X-ray by Ezoe et al ^[6]. The X-ray telescope consisted of mirror chips those were put in a concentric circle, as shown in figure 3a and mirror chips are put on a support base board, like as figure 3b.

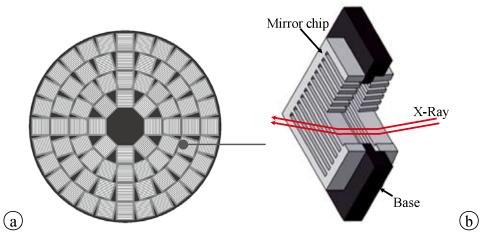


Fig. 3 Schematic of MEMS X-ray mirror optics

One mirror chip was designed as a size of $7.5 \times 7.5 \times 0.2 \text{ mm}^3$. So we can design a mirror system, collection area, distance of focal point and number of mirrors, by arranging chip flexibly [7].

However, reflectivity falls when X-ray energy is above 2 keV in order to use silicon as a mirror material, figure 2 shows the theoretical X-ray reflectivity. If the mirror made of nickel is used, it can reflect the X-ray of up to about 3 keV, that is also shown in figure 2. Then, the mirror made from a nickel plate by the LIGA process was focused on. Synchrotron radiation (SR) lithography is one of the most effective technologies for fabricating high aspect ratio micro- and nano-structures [8, 9]. Particularly in the applications of optical devices, structures fabricated by SR lithography have been possible to approach a submicron scale and a higher resolution. The relation of an X-ray reflectance and a surface roughness of a mirror is expressed with the following formula.

$$I = I_0 \times \exp\left(-\left(\frac{4\pi\sigma\sin\theta}{\lambda}\right)^2\right) - \dots (1)$$

The right-side member of the equation except I_{θ} is called Debye-Waller factor. As for the symbol, I is intensity of reflected X-ray, I_{θ} is intensity of incident X-ray, δ is a rms roughness of the X-ray reflecting surface, θ is an angle of incidence of the X-ray, λ is a wavelength of the incidence ray. As shown in the formula, reflectivity depends on rms roughness, because of θ is determined by mirror system design and λ is determined by target energy.

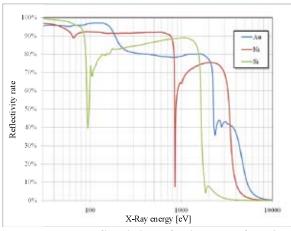


Fig. 4 X-ray reflectivity of mirror surface in various materials.

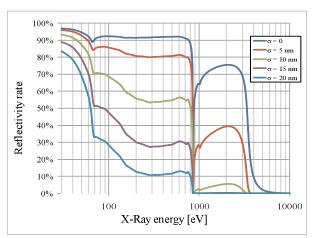


Fig. 5 X-ray reflectivity of Ni mirror surface at different surface roughness

Figure 5 shows a calculation result of a reflectance when θ is from 0 up to 20 nm, with the incident angle of the rays are 1 degree. If θ equals 0, I equals to I₀ that is shown the theoretical maximum reflectance. The reflectivity drop point at the X-ray energy of about 850 eV depends on absorption edge of nickel. It is known that nickel L-shell absorption edges are 853 eV and 870 eV. If the mirror which surface roughness is 15 nm was used, the reflectance of above 900 eV ray was about 0, and even ray of below 900 eV was very low reflectivity that is under 5 %. So this surface that had such a roughness cannot be used as an X-ray total reflection mirror. The surface roughness of a mirror needs to be below 5 nm, and roughness of below 1 nm can be more practical mirrors.

2. FABROCATION PROCESS

The fabrication processes are listed as in the following steps and shown in figure 3. At first the SR X-ray is irradiated to a PMMA (poly-methylmethacrylate) resist through an X-ray mask. Since the surface form of a PMMA structure is correctly transferred as a plating structure, The PMMA was developed with a GG developer, and then the sample was rinsed with stopper solvent and then rinsed with DI water, and finally dried in vacuum chamber. And next, Au layer for electroforming seed layer was deposited. Then electroforming was processed and the PMMA structure is completely covered with Ni. The unnecessary part of electroplating and PMMA base was chipped away using lapping machine. And finally remaining PMMA resist was completely removed with organic solvent and mirror chip was picked up.

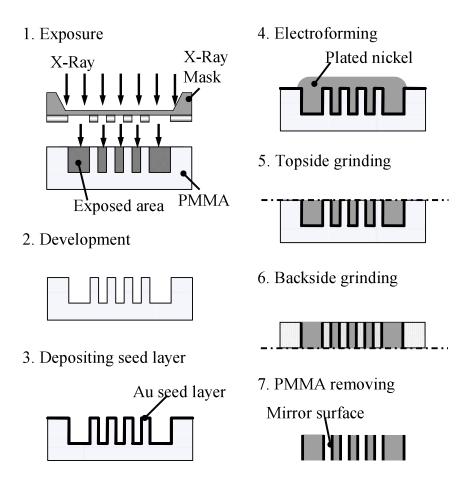


Fig. 6 Process flow

3. FABRICATION RESULT

Fig. 7 shows an X-ray mask of fabricated X-ray total reflection mirror chip. The mirror chip design had 71 slits those slits were width of 50 μ m each and each lines were width of 20 μ m, the length of slits or lines were 2400 μ m, and the width of the beam that was in center of the chip was 200 μ m. The X-ray mask with 3 μ m-thick Au absorber consisting of masking patterns formed on 50 μ m-thick polyimid membrane was used.

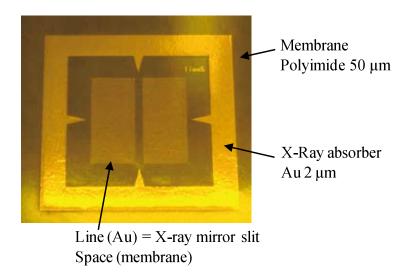


Fig. 7 X-ray mask of X-ray mirror chip

When manufacturing an X-ray mirror it becomes important how much roughness the PMMA resist side wall surface has. For controlling the PMMA resist surface roughness, processing form, and the processing depth, it was necessary to adjust parameters, such as the amount of SR beam dosage, development and rinse time, and development temperature. We adjusted some process parameters as follows. The amount of X-ray dose at 0.28 A·h was irradiated to reveal the structure with the height of 300 μ m. The developing time was 120 min, rinse time with stopper solvent was 60 min, and rinse time with DI water was 60 min, each liquids temperature were 37°C. Then the fabricated PMMA structure that was master block for nickel metal plating was shown in figure 8 and its side view was shown in figure 9. The 20 μ m lines (they look like walls at figure 9) were successfully fabricated but line width was decreased to about 16.5 μ m.

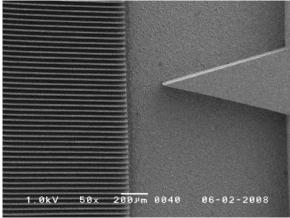


Fig. 8 PMMA master block of X-ray grazing mirror chip

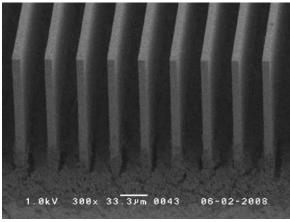


Fig. 9 The side view of PMMA master block of mirror chip

The PMMA master block was electro plated with nickel. Metal plating was processed by dipping in 350 g/l nickel (II) sulfamate tetrahydrate bath, the process time was 36 hour at electrical current density of 200 mA/dm². And then the mirror chip was ground both of top and bottom side with #8000 (size of abrasive grain was 1 μ m) lapping film. The average width of mirror chip slit was 16.4 μ m. This is because of decrease the line width of PMMA master block in developing process. The metal plated mirror slit was transfer the shape accurately from master block.

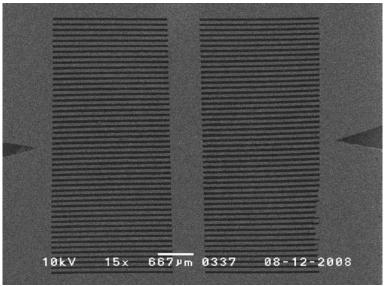


Fig. 10 SEM photograph of the fabricated X-ray total reflection mirror chip made from nickel by electroplating.

3. ROUGHNESS OF MIRROR SURFACE

The SEM photograph of the mirror sidewall was shown in figure 11. The mirror surface seems to be smooth without any blemish, streaky and undulated structures. The detailed roughness of the mirror surface was measured by scanning probe microscopy (SPM) at several points. Figure 6 shows typical example of measured mirror surface. The mirror surface roughness of the root mean square (rms) was about 12 nm at the area of $5 \times 5 \, \mu m^2$. In addition the surface roughness was grinded and reduced by Magneto-Rheological Fluids (MRF).

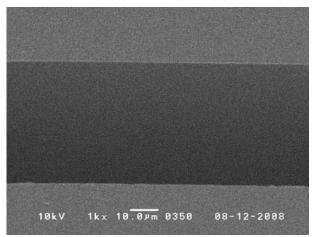


Fig. 11. SEM photograph of the fabricated X-ray total reflection mirror chip made from nickel by electroplating.

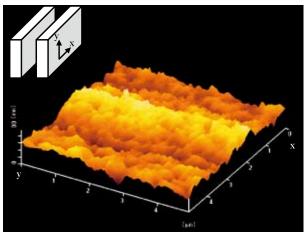


Fig. 12. Example of a SPM image of nickel mirror surface

4. X-RAYREFRECTANCE OF MIRROR CHIP

The X-ray angular response at fabricated mirror chip was measured. The X-ray generates by Manson X-ray generator and the origin of X-ray was aluminum $K\alpha$. Figure 13 shows the results. Black solid line was shown from the simple collimator profile that has no reflectance. The measured data has excess from the simple collimator line, this fact proves that X-rays reflects by this mirror chip. And the profile is similar to rms roughness of 4 nm, so the mirror surface roughness was estimated 4 nm.

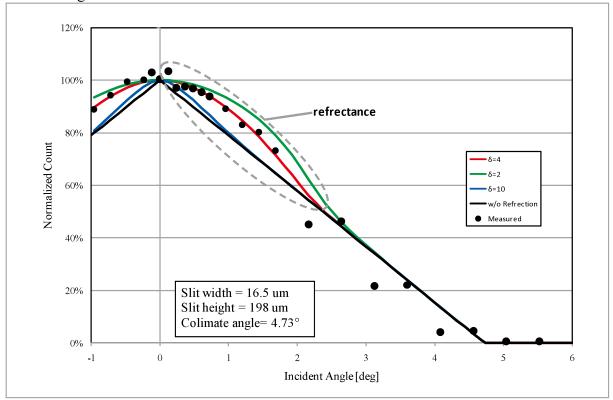


Fig. 13 The grating with 300 nm pitch

5. CONCLUSION

The X-ray total reflection mirror chip was fabricated by using SR lithography and electroforming process. The X-ray total reflection mirror chip had slits width of 50 μ m and each lines were width of 50 μ m, the length of slits or lines were 2400 μ m, and the width of the beam that was in center of the chip was 200 μ m. And the X-ray was irradiated this mirror chip and we can prove the X-rays can be reflected by this LIGA mirror chip. The roughness of a mirror surface was estimated as rms 4 nm, but it will be improved and it must reduce to below 1 nm in near future. An X-ray total reflection mirror device will be assembled by the improved mirror chip, and we aspire to realize an X-ray telescope composed with ultra light weight X-ray total reflection mirror chips.

REFERENCES

- [1] Yuichiro Ezoe, Masaki Koshiishi, and Kazuhisa Mitsuda: "MEMS open the way to ultra lightweight and low-cost x-ray optics", SPIE Newsroom, doi:10.1117/2.1200609.0392 (2006)
- [2] Yuichiro Ezoe, Masaki Koshiishi, Makoto Mita, Kazuhisa Mitsuda, Akio Hoshino, Yoshitaka Ishisaki, Zhen Yang, Takayuki Takano, and Ryutaro Maeda: "Micropore x-ray optics using anisotropic wet etching of (110) silicon wafers", APPLIED OPTICS,

- Vol.45, No.35, pp.8932-8938 (2006)
- [3] S. W. Wilkins, A. W. Stevenson, K.A. Nugent, H. Chanpman, and S. Steenstrup, "On the concentration, forcusing, and collimation of x rays and neutrons using microchannel plates and configurations of holes," Rev. Sci. Instrum, 60, 1026-1036 (1989)
- [4] M. Bavdaz, D. H. Lumb, A. J. Peacock, M.Beijersbergen, and S. Kraft, "Status of x-ray optics development for XEUS Mission" in Design and Microfabrication of Novel X-Ray Optics, A. A. Snigirev and D. C. Manicini, eds., Proc. SPIE 5488, 829-836 (2004)
- [5] G. W. Fraser, M. J. L. Turner, and M. G. Watson, eds., "Microchannel plate x-ray optics", Leicester X-ray Astronomy Group Special ReportXR97/02, p.191 (1997)
- [6] A. Chen, P. Kaaret, and T. Kenny, "Focusing of x rays using micromachined silicon lobster eye lenses", Multilayer and Grazing Incidence X-Ray/EUV Optics II, R. B. Hoover, ed., Proc. SPIE 2011, 227-231 (1994)
- [7] A. Chen, P. Kaaret, and T. Kenny, "Micromachined silicon x-ray optics", Advances in Multilayer and Grazing Incidence X-Ray/EUV/FUV Optics, R. B. Hoover and A. B. Walker, Jr., eds., Proc. SPIE 2279, 298-304 (1994)
- [8] P.Bley and J.Mohr, "The LIGA Process A Microfabrication Technology -", FED Journal, 5 (1), pp.34-48 (1994)
- [9] W. Ehrfeld and H. Lher, "Deep X-Ray Lithography for the Production of Three-Dimensional Microsystems from Metals, Polymers, Ceramics", Radiat. Phys. Chem, 45 (3), pp.340-365 (1995)