Design and Fabrication of Soft X ray Suppermirrors

Hanlin Wang
University of Chinese Academy of Sciences

Haifeng Wang
Changchun Institute of Optics, Fine Mechanics and Physics

Shuai Ren
Changchun Institute of Optics, Fine Mechanics and Physics

Peng Zhou
Changchun Institute of Optics, Fine Mechanics and Physics

Xin Zheng
Changchun Institute of Optics, Fine Mechanics and Physics

Bo Chen
Changchun Institute of Optics, Fine Mechanics and Physics

Zhongjun Chen
Institute of High Energy Physics

Shuhu Liu
Institute of High Energy Physics

Xiaodong Wang (wangxiaodong@ciomp.ac.cn)
Changchun Institute of Optics, Fine Mechanics and Physics

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Abstract

W/Si, Ir/SiC, and Pt/C supermirrors were designed by block method and fabricated by magnetron sputtering method. The glancing incident angles are 1.0, 1.4 and 1.7 degree. Pt/C supermirrors show better spectral results than W/Si, Ir/SiC. Pt/C supermirrors also have good angular tolerance, which will benefit opt-mechanic alignment. In addition, Pt layer as outmost one can withstand harsh space environment.

Main Text

In X ray region, the refractive index of all materials is near to unity, extinction coefficient is near to zero, which means that all materials are transparent. In normal incidence, X ray has extremely low reflectance. Thus, reflective optical system works in glancing mode in X ray region by virtue of total reflection of materials [1]. With increasing of energy, total reflection glancing angle decreases significantly, and this means that optical system ought to have a long size (tens or hundreds of meters magnitude), which leads to high cost, alignment difficulty, and low collecting area. Supermirror is a good solution to enhance glancing incident angle [2-4]. It is composed of alternating two materials, each material has different thickness, and it has a good reflection in a broad waveband at a fixed angle or in a broad angle range at a fixed energy by Bragg diffraction law.

There are three kinds of methods to design supermirrors: power law method [3], numerical analysis method [4], and block method [2]. In block method, a supermirror is composed of several blocks, each block is a periodic multilayer, and it has a good reflectance at specific waveband by Bragg diffraction law. Different block has different period thickness, and this will achieve a good reflectance at broad wavelength or angle range. The supermirror designed by block method is easier to be fabricated than other two methods because it has lower requirement of thickness control accuracy.

Supermirrors are widely used in space exploration [5-7]. W/Si supermirrors were used in NuStar [5], Pt/C supermirrors were used in ASTRO-H [6] and NuStar, and Ir/SiC will be used in Athena [7]. In Athena, the working energy range is from 0.1 to 10keV, and the biggest glancing angle is 1.752 degree. However, they did not give the details of multilayer design, and it seems that their theoretical and experimental results were not so good [7].

In this paper, W/Si, Ir/SiC, and Pt/C supermirrors were designed by block method and fabricated by magnetron sputtering method. The glancing incident angles are 1.0, 1.4 and 1.7 degree.

Supermirrors designed by block method are easy to be fabricated because they do not need high precision of control accuracy for deposition rate. Thus, we used block method to design supermirrors. As shown in Fig. 1, supermirrors designed by block method compose of several blocks, and one block is a periodic multilayer. Two alternating materials with fixed thickness (dH, dL) constitute a period with a period thickness of d in the periodic multilayer, and two materials have high (H) and low (L) refractive index (n). One block has N periods, and this block has a high reflectance at fixed wavelength based on
Bragg diffraction law. Bragg diffraction law is defined as Eq. (1), where $\theta$ is glancing incident angle, $\lambda$ is wavelength, $d$ is periodic thickness. Since with increasing of energy, absorption decreases, to reduce absorption, we put long-wavelength block near to air. That is to say, $\lambda_5 > \lambda_4 > \lambda_3 > \lambda_2 > \lambda_1$, and $d_5 > d_4 > d_3 > d_2 > d_1$, $N_5 < N_4 < N_3 < N_2 < N_1$, and total thickness of Block5 < Block4 < Block3 < Block2 < Block1. The details about block method can be found in Ref. 2 and 8. W/Si, Ir/SiC, and Pt/C supermirrors were designed by block method. The glancing incident angles are 1.0, 1.4 and 1.7 degree. The substrate is fused silica, and the roughness of the substrate and films is assumed to be 0.45 nm. To resist space high-energy particles, we put metal layer as the final one near to air. It was reported that Ir and Pt have good stability when exposed to space environment, especially atomic oxygen [9-11].

$$2d \sin \theta = \lambda$$ (1)

| Grazing incident angle | 1.0 degree | 1.4 degree | 1.7 degree |
|------------------------|------------|------------|------------|
| Pt/C supermirror       | 18.89%     | 7.37%      | 3.83%      |
| Ir/SiC bilayer coating | 6.60%      | 0.52%      | 0.15%      |

Table 1

Comparison of average reflectance between supermirrors and bilayer at 5-10 keV.

Figure 2 shows theoretical reflectance curves of Pt/C supermirrors designed by block method at 1.0, 1.4, 1.7 degrees. In Athena, Ir/SiC bilayer is the baseline coating, and the optimized structure consists of 10 nm Ir with 4 nm SiC. For comparison, results of Ir/SiC bilayer coating are provided. Supermirrors demonstrate significantly better spectral performance than Ir/SiC bilayer coating at high energy, especially at 5-10 keV. Table 1 gives comparison of average reflectance between Pt/C supermirrors and Ir/SiC bilayer coating at 5-10 keV. The improvement magnitudes at 1.0, 1.4, 1.7 degree are 6.6, 14.2, 25.5, respectively.

Table 2, 3, 4 gives designed structure of W/Si, Ir/SiC and Pt/C supermirrors. Periodic thickness for 1.0 degree ranges from 3.6 to 8.0 nm, for 1.4 degree from 2.6 to 6.8 nm, and for 1.7 degree from 2.2 to 6.1 nm.
Table 2
Design results of W/Si supermirrors.

| E (keV) | 4    | 4.5  | 5    | 6    | 7    | 8    | 9    | 10   |
|---------|------|------|------|------|------|------|------|------|
| 1.0 degree |   d (Å)   | ---  | 79.43 | 53.42 | 48.89 | 44.19 | 40.61 | 36.81 |
|          | \( \Gamma_W \) | ---  | 0.525 | 0.467 | 0.458 | 0.501 | 0.469 | 0.514 |
|          | N    | ---  | 1    | 2    | 2    | 3    | 5    | 7    |
| 1.4 degree |   d (Å)   | 67.44 | 43.11 | 38.34 | 33.48 | 30.27 | 28.49 | 26.69 |
|          | \( \Gamma_W \) | 0.558 | 0.378 | 0.476 | 0.468 | 0.483 | 0.507 | 0.503 |
|          | N    | 1    | 2    | 3    | 5    | 7    | 10   | 15   |
| 1.7 degree |   d (Å)   | 60.4 | 34.19 | 40.3  | 33.89 | 29.67 | 26.82 | 24.79 | 22.89 |
|          | \( \Gamma_W \) | 0.5  | 0.364 | 0.426 | 0.484 | 0.468 | 0.459 | 0.502 | 0.495 |
|          | N    | 1    | 1    | 1    | 3    | 5    | 8    | 12   | 18   |

Table 3
Design results of Ir/SiC supermirror.

| E (keV) | 4 | 5 | 6 | 7 | 8 | 9 | 9.5 | 10 |
|---------|---|---|---|---|---|---|-----|----|
| 1.0 degree |   d (Å)   | --- | 79.78 | 53.06 | 47.87 | 43.86 | 40.62 | --- | 36.92 |
|          | \( \Gamma_{Ir} \) | --- | 0.569 | 0.414 | 0.38  | 0.453 | 0.46  | --- | 0.541 |
|          | N    | --- | 1    | 2    | 2    | 3    | 5    | --- | 7    |
| 1.4 degree |   d(Å)  | 52.63 | 43.95 | 36.57 | 34.04 | 30.21 | 28.03 | --- | 26.46 |
|          | \( \Gamma_{Ir} \) | 0.499 | 0.461 | 0.412 | 0.5   | 0.469 | 0.503 | --- | 0.49  |
|          | N    | 1    | 1    | 2    | 4    | 6    | 9    | --- | 12   |
| 1.7 degree |   d(Å)  | 59  | 37.13 | 34.87 | 30.33 | 29.15 | 26.06 | 23.89 | 22.11 |
|          | \( \Gamma_{Ir} \) | 0.5  | 0.395 | 0.379 | 0.469 | 0.449 | 0.442 | 0.49  | 0.499 |
|          | N    | 1    | 2    | 2    | 3    | 5    | 8    | 12   | 18   |
Table 4  
Design results of Pt/C supermirrors.

| E (keV) | d(Å)    | 4   | 4.5  | 5   | 6   | 7   | 8   | 9   | 10  |
|---------|---------|-----|------|-----|-----|-----|-----|-----|-----|
| 1.0 degree |       | --- | ---  | 79.96 | 52.95 | 47.74 | 43.26 | 40.43 | 36.58 |
|          | Г_{Pt}  | --- | ---  | 0.577 | 0.434 | 0.401 | 0.472 | 0.463 | 0.528 |
|          | N       | 0   | 1    | 2    | 2   | 3   | 5   | 7   |     |
| 1.4 degree | d(Å)    | 57.93 | 42.74 | 38.54 | 33.75 | 31.03 | 28.77 | 26.72 |     |
|          | Г_{Pt}  | 0.563 | 0.462 | 0.43  | 0.473 | 0.565 | 0.508 | 0.491 |     |
|          | N       | 1   | 1    | 2    | 4   | 6   | 9   | 12  |     |
| 1.7 degree | d(Å)    | 58  | 33.53 | 38.58 | 32.63 | 28.59 | 25.89 | 23.95 | 22.13 |
|          | Г_{Pt}  | 0.5  | 0.357 | 0.407 | 0.463 | 0.457 | 0.453 | 0.498 | 0.484 |
|          | N       | 1   | 1    | 1    | 3   | 5   | 8   | 12  | 18  |

Six materials were deposited by Direct current magnetron sputtering. Metals were deposited at 25-35 W, and Si, C and SiC were deposited at 40-150 W. The base pressure is 4.0×10^-4 Pa, and working pressure is (4-8)×10^-2 Pa. Thickness was controlled by time. Deposition rates for W, Si, Ir, SiC, Pt, C are 0.02417, 0.02874, 0.01629, 0.01883, 0.02346, 0.01195 nm/s, respectively. Since Ir layer has a high stress, to mitigate stress [12], Cr layer was deposited on the substrate prior to Ir layer deposition.

Reflectance of supermirrors were measured at Beijing Synchrotron Radiation Facility (BSRF). Reflectance at 2.0-5.5 keV was measured at 4B7A station, and reflectance at 6-10 keV was measured at 4B9A station. As shown in Fig. 3a, two axuv detectors were used, first one was used to detect intensity of incident light, and second one was used to detect reflecting light from supermirrors after the first one moved away from optical route. First detector was electronically controlled, and measurement was automatically conducted. The step was 0.02 keV. To assure that light beam beats effective area of supermirrors, the widths of slit were manufactured by laser lithography to be 0.05mm for 1.0 degree, 0.16 mm for 1.4 degree, 0.26 mm for 1.7 degree, respectively. The base noise is less than 20 pA. As shown in Fig. 3b, only one EDEc detector was used. Measurement was manually performed. The detector detected intensity of incident light, then the sample was put onto the stage, and detector moved to 2θ position to detect intensity of reflecting light from the supermirror. The step was 0.5 keV.

Figure 4 shows experimental reflectance results of W/Si supermirrors. There is large deviation between experiment and theoretical calculation. The reason for this discrepancy is that high deposition rate leads to big thickness error. Figure 5 demonstrates experimental reflectance results of Ir/SiC supermirrors. At 1.0 and 1.4 keV, there is a good agreement between experiment and theoretical calculation. The shortcoming is that reflectance is close to zero at 6 keV. At 1.7 degree, there is a good agreement between experiment and theoretical calculation at 2-5 keV, but the agreement gets worse at 5.5-10 keV. As shown
in Fig. 6, there is an excellent agreement between experiment and theoretical calculation for Pt/C supermirrors. The average reflectances are 18.19%, 9.39%, 4.87% at 5-10 keV for 1.0, 1.4, 1.7 degree, respectively. In Opto-mechanic alignment, mirrors are also required to have a good angular tolerance. That is to say, when there is a little angle deviation, the reflectance ought not decrease significantly. Figure 7 shows angular tolerance of Pt/C supermirrors at 7.0 keV. At 1.0 degree of supermirror, there is still larger than 10% of reflectance up to 1.5 degree, at 1.4 degree of supermirror, there is still 8% of reflectance up to 1.9 degree, and at 1.7 degree of supermirror, there is still larger than 5% of reflectance up to 2.2 degree. This will benefit opto-mechanic alignment.

In summary, W/Si, Ir/SiC and Pt/C supermirrors were designed by blocked method and fabricated. Pt/C supermirrors show excellent spectral performance, and the average reflectances are 18.19%, 9.39%, 4.87% at 5-10 keV for 1.0, 1.4, 1.7 degree, respectively. They still have a good angular tolerance, and this will benefit opto-mechanic alignment.

**Declarations**

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**Author contributions**

X.D.W. designed and fabricated supermirrors and wrote the paper, H. W., H. W., Z. C. and S. L. did reflectance measurement, S. R., P. Z. and X. Z. prepared some samples, B.C. reviewed the manuscript.

**Competing interests**

The author(s) declare no competing interests.

**References**

1. Bajt, “X-ray focusing with efficient high-NA multilayer Laue lenses,” Light Sci. Appl. 7, 17162 (2018).
2. Yao, H. Kunieda, and Z. Wang, “Design and fabrication of a supermirror with smooth and broad response for Hard X-Ray Telescopes,” Appl. Opt. 52, 6824 (2013).
3. D. Joensen, P. Voutov, A. Szentgyorgyi, J. Roil, P. Gorenstein, P. Høghøj, and F. E. Christensen, “Design of grazing-incidence multilayer supermirrors for hard-x-ray reflectors,” Appl. Opt. 34, 7935 (1995).
4. V. Kozhevnikov, I. N. Bukreeva, and E. Ziegler, “Design of X-ray supermirrors,” Nucl. Instrum. Methods Phys. Res. A 460, 424–443 (2001).
5. E. Christensen, A. C. Jakobsen, N. F. Brejnholt, K. K. Madsen, A. Hornstrup, N. J. Westergaard, J. Momberg, J. Koglin, A. M. Fabricant, M. Stern, W. W. Craig, M. J. Pivovaroff, and D. Windt “Coatings for the NuSTAR mission,” Proc. SPIE 8147, 81470U (2011).

6. Tamura, H. Kunieda, Y. Miyata, T. Okajima, T. Miyazawa, A. Furuzawa, H. Awaki, Y. Haba, K. Ishibashi, M. Ishida, Y. Maeda, H. Morl, Y. Tawara, S. Yamauchi, K. Uesugi, Y. Suzuki, and HXT Team, “Supermirror design for Hard X-Ray Telescopes on-board Hitomi (ASTRO-H),” J. Astron. Telesc. Instrum. Syst. 4, 011209 (2018).

7. D. M. Ferreira, S. Massahi, F. E. Christensen, B. Shortt, M. Bavdaz, M. J. Collon, B. Landgraf, N. C. Gellert, J. Korman, P. Dalampiras, I. F. Rasmussen, I. Kamenidis, M. Krumrey, and S. Schreiber, “Design, development, and performance of x-ray mirror coatings for the ATHENA mission,” Proc. SPIE 10399, 1039918 (2017).

8. D. Wang, B. Chen, H. F. Wang, L. Zhang, S. Ren, and P. Zhou, “Design of W/Si supermirror by block method,” Nuclear Inst. and Methods in Physics Research, A 957, 163435 (2020).

9. Garoli, L. V. R. D. Marcos, J. I. Larruquert, A. J. Corso, R. P. Zaccaria, and M. G. Pelizzo, “Mirrors for space telescopes: degradation issues,” Appl. Sci. 10, 7538 (2020).

10. Nardello, P. Zuppella, V. Polito, A. J. Corso, S. Zuccon, and M. G. Pelizzo, “Stability of EUV multilayer coatings to low energy alpha particles bombardment,” Opt. Express 21, 28334 (2013).

11. R. Gull, H. Herzig, J. F. Osantowski, and A. R. Toft, “Low earth orbit environmental effects on osmium and related optical thin-film coatings,” Appl. Opt. 24, 2660 (1985).

12. Probst, T. Begou, T. DÖHRING, S. Zeising, M. Stollenwerk, J. Stadtmüller, F. Emmerich, and J. Lumeau, “Coating stress analysis and compensation for iridium-based x-ray mirrors,” Appl. Opt. 57, 8775 (2018).

Figures
Figure 2

Theoretical reflectance curves of Pt/C supermirrors designed by block method at 1.0, 1.4, 1.7 degrees, and for comparison, results of Ir/SiC bilayer coating are provided.

Figure 3

Sketch of setup of reflectance measurement in 4B7A (a) and 4B9A (b) stations of BSRF.
Figure 7

Angular tolerance of Pt/C supermirrors.