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Research of multilayers in EUV, soft X-ray and X-ray

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Abstract: To develop beam splitters for soft X-ray laser Michelson interferometer at 13.9 nm, Mo/Si multilayers of 100 nm thickness deposited on both sides of silicon nitride were fabricated by using DC magnetron sputtering. Initial evaluation of their reflectivity and transmission showed that reflectivity and transmission were above 10% and 25%. The broadband analyzers have been designed, fabricated and characterized for 13～20 nm polarization measurements. The measured results are in good agreement with the design. The supermirrors with different angular intervals at 0.154 nm have been designed, fabricated and characterized.

Key words: multilayer; analyzer; broadband; supermirror

1 Introduction

The surface reflectivity of every material for radiation at wavelengths below 30 nm at normal incidence is almost zero. In order to get an enough reflection, optical components adopt grazing angular incidence because at small incident angles high reflectivity can be obtained in condition of total reflection. The grazing incidence optics is generally complex to align. Moreover, the great magnitude of aberrations limits their spatial resolution. High reflectivity can be got at normal incidence in the extreme ultraviolet (EUV) and soft X-ray region and at non-total reflection angle in X-ray range by using multilayer coatings. In general the reflectivity of the multilayer is optimized by selecting materials and deposition conditions that (1) maximize the contrast in the index of refraction, (2) minimize the absorption that is always appreciable at EUV, soft X-ray and X-ray range, and (3) produce continuous layers with compositionally abrupt and smooth interfaces. The best material combinations found to data are different at different wavelength ranges, such as Mo/Si for 12.4～30 nm[1].

High efficiency and high resolution EUV and soft X-ray optics could be used in many research fields. Extreme ultraviolet lithography has a great interest in making next generation integration circuit[2]. In astrophysics the observation of selected solar radiation in EUV and soft X-ray is very important for studying the sun[3]. A monochromator based on multilayers has been set up in some synchrotron radiation facilities[4]. Some of multilayers can be used in ICF diagnostic purposes[5]. We are interested in the applications of multilayers in China and have made some beam splitters for soft X-ray laser interfer-

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ometer, some analyzers and phase retarder for EUV and soft X-ray polarization measurements, and X-ray supermirrors for future space telescope made in China.

2 Experimental technique

All the multilayers used in this study were deposited using by a DC magnetron sputtering machine. The deposition system contains four sputtering guns mounted on the bottom of the chamber and spaced 90° apart. All of the sputtering guns are circular (100 mm diameter). The substrates, which are spun during the depositions, are facing down and affixed to the platter, which rotates over the sputtering targets. The individual layer thickness is controlled by the substrate stay time above the target. A turbo pump is used to evacuate to a base pressure of less than 5×10^{-5} Pa. Ultrahigh purity Ar gas at 1.96 Pa was used to sputter different targets.

The sputtering rates were calibrated by varying the stay time of the substrate on the targets at a constant power and determining the multilayer periods by fitting the Bragg peaks in the grazing angle X-ray diffraction spectra. X-ray diffraction measurements were performed using a Bede D1 diffractometer with a Cu $K\alpha$ source ($\lambda = 0.154$ nm). The small angle X-ray diffraction measurements were obtained over the angular range of $\theta \sim 2\theta$ between 0.3～10°.

Reflectivity of the multilayers was measured with a synchrotron-based reflectometer at National Synchrotron Radiation Laboratory in Hefei. The reflectometer has been designed for the characterization of multilayers, filters and mirrors.

3 Soft X-ray beam splitters for the Michelson interferometer

Due to their first realization about 16 years ago, soft X-ray transmissive optics allowed new investigations to soft X-ray optics application. It is possible to extend the applications to soft X-ray interferometer with the objective to probe the electron density of laser-produced plasma. The maximum electron density and the size of the probed plasmas are severely limited by absorption and refraction of the probe beam. For these reason, Da Silva and his colleagues developed an amplitude-division soft X-ray laser Mach-Zehder interferometer, successfully used to probe a dense plasma at 15.5 nm in 1995[6]. The soft X-ray laser Mach-Zehder interferometer working at 13.9 nm has been successfully developed in China in 2003. The experiments have been used two beam splitters made by depositing Mo/Si multilayers on back side of the silicon membranes with a clear area 10 mm × 10 mm. The beam splitters provided a good product of reflectivity and transmission and were suitable to use. Two beam splitters needed at the same time for accomplishing the interferometer. Every time, two beam splitters were broken when probing the laser-produced plasma because of the debris of the plasma. So it is very expensive to do the relative interferometeric experiments. With the aim of decreasing the quantities of used beam splitters, we need to develop a Michelson interferometer instead of Mach-Zehder.

The great difference between the beam splitters used in Michelson and Mach-Zehder interferometer is that beam splitters used in Michelson need to deposit the Mo/Si multilayers on both sides of the silicon membranes, while one side used in Mach-Zehder. Delmotte and his colleagues[7] developed the beam splitters for the Michelson interferometer operating at a 45° incidence angle at 13.9 nm. According to the phenomena of polarization in soft X-ray range, the reflectivity of p polarization is very low (typically, 100 times lower than for s polarization). So, when mounted in a Michelson interferometer at 45° incidence angle, the beam splitter will be effective only for the s component of the soft X-ray laser. The situation can be changed when putting the incidence angle less than 10°, just as 7.2° used in the previous Mach-Zehder interferometer. The simulation of this kind of beam splitter can give the optimization the relative thicknesses of Mo in the period and the number of periods on each side. We have calculated the variation of the reflectivity-transmission product ($R \times T$ product) as a function of the relative thickness of Mo in the period thickness with optimized number of periods for each thickness and with a period thickness of 7.0 nm. Fig. 1 shows the variation of the $R \times T$ product as a function of the number of the periods on each side with the relative thickness of Mo in the period of 0.4 and periods of 7.0 nm for incidence angle of 7.2° and 10.8 nm for incidence of 45°. Approximately 2% of $R \times T$ product can be obtained with the number of periods equal to 4 or 5 for incidence angle of 45°, while 4.5% of $R \times T$ product with the number of periods equal to 6 or 7 for incidence angle of 7.2°. So the throughput can be increased more than 4 times working at 7.2° instead of 45°.

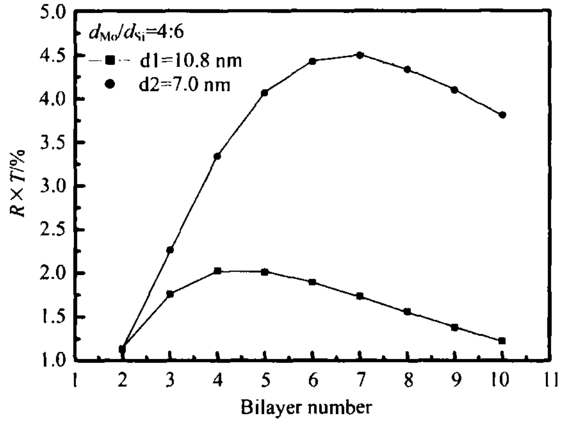
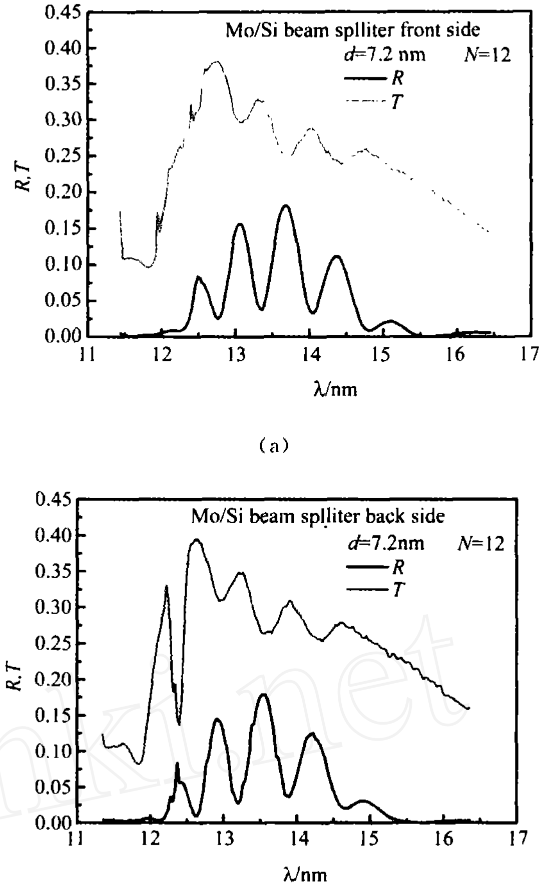


Fig. 1 Variation of the reflectivity-transmission product as a function of Mo/Si multilayer periods

The initial experiments have been carried out and the reflectivity of the both sides of beam splitters has been measured. Fig. 2(a) and Fig. 2(b) showed the measured reflectivity results of front side and back side of beam splitter. The Fabry-Perot oscillations can be seen in the reflectivity curves. There is a wavelength shift appeared in the oscillations because of either a variation of silicon nitride membrane thickness or a variation of the thickness periods. Fig. 2 shows that the reflectivity of the back side is higher than that of the front side which is related to the difference of roughness between the silicon nitride membrane's front side and back side (the roughness of a back side is lower). Further studies are in progress in order to meet the requirements of a Michelson interferometer at 13.9 nm.



(a)

(b)

Fig. 2 Experimental reflectivity measured on two both-sides coated membranes versus wavelength with the angle of incidence 7.2°

4 Broadband analyzers in EUV and soft X-ray region

Circularly polarized synchrotron radiation has attracted growing interest at various synchrotron radiation centers for the study of a wide range of phenomena in biology, chemistry, physics and material science. In the soft X-ray region, accurate evaluation of state of polarization of light emerging from a monochromator is basically important. The polarized soft X-rays have been used in many experiments, such as circular dichroism spectroscopy, spin-polarized photoelectron spectroscopy. Magnetic circular dichroism (MCD) and the Faraday effect at the 2p absorption edges of the transition materials and at the 3d edge of rare earths are starting to be used for quantitative determination of the degree of circular polarization. Helical undulators designed to produce intense radiation polarized circularly as well as linearly very well meet these requirements. In the EUV region, traditionally, reflection analyzers are used, for the soft X-ray range the use of multilayers in transmission as phase retarders has been suggested. The linear analyzers and phase retarders based on multilayer interference structures, and circular polarizing filters based on magnetic circular dichroism are applied in magneto-optical studies utilizing synchrotron radiation.

The narrow-band property of soft X-ray multilayer analyzers loses their polarizing power and limits their application because of certain angle essential to keep a reasonable throughput at these wavelengths, which limits their application. The original approach is the design method using the double-multilayer or depositing the depth-graded X-ray multilayers can improve the wideband property of the analyzers. However, variation of the angle of incidence causes the direction of reflected beam to change or the process of design and fabrication of multilayer analyzers is very complicated. To overcome the weak points of the multilayer analyzers, we designed the non-periodic multilayer structure as the analyzer, which would greatly simplify the experiment because the analyzers would have fixed shape and angles of incidence.

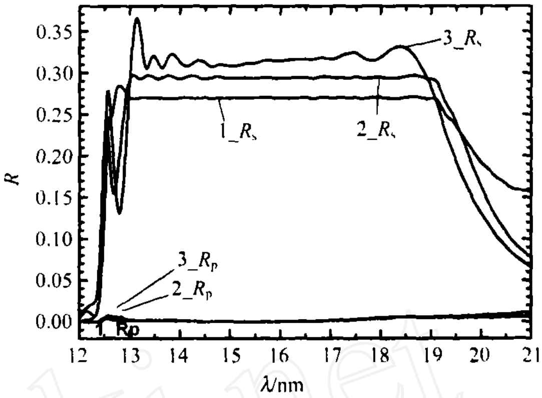


Fig. 3 S-reflectivity and p-reflectivity of Mo/Si wideband analyzer optimized with the use of direct computer algorithm to provide the plateau s-reflectivity. R_0 = 0.27(1), 0.3(2) and 0.34(3) in the [13, 19 nm] spectral range. The bilayer number N is equal to 40, and all the three wideband analyzers are designed at the normal incident angle of 40°

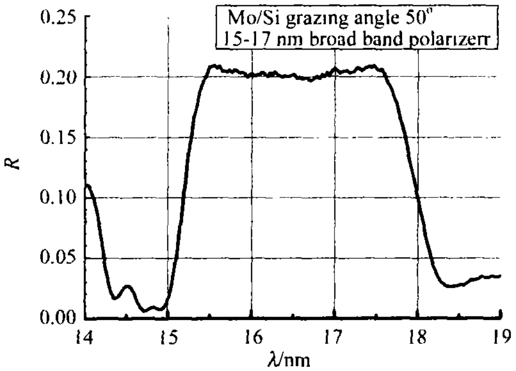


Fig. 4 Reflectivity measurements for broadband multilayers vs wavelength

The s-reflectivity and p-reflectivity of Mo/Si wideband analyzer optimized with different target reflectivity R_0 to provide the plateau s-reflectivity is shown in Fig. 3. The s-reflectivity value is too large to obtain an even reflectivity

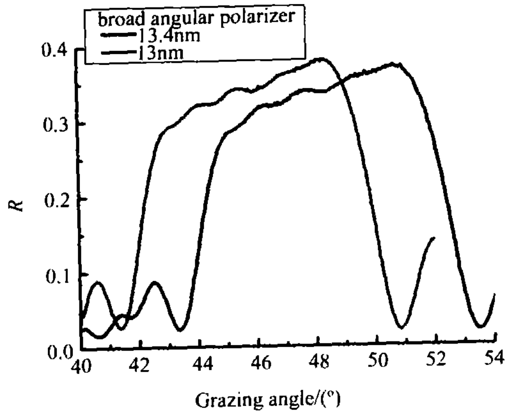


Fig. 5 Reflectivity measurements for broadband angular multilayers vs angle

plateau. One way can be used in practice to obtain an even reflectivity plateau, that is, decreasing the target reflectivity value R_0 of the plateau at fixed number of bi-layers, which results in a reduction and plateau of the finial s-reflectivity. As shown in curve 3 ($R_0 = 0.34$), the s-component reflectivities oscillate greatly. R_s turns slightly oscillatory with the decrease of $R_0 = 0.3$ in curve 2, while R_s becomes very flat in curve 1 ($R_0 = 0.27$).

The EUV and soft X-ray reflectivity measurement at grazing angle of 50 degrees indicated that the broadband Mo/Si multilayer had about 20% reflectivity from 15.5～17.5nm region (Fig. 4). Fig. 5 shows that the reflectivity results at 13 nm and 13.4 nm of a broadband angular Mo/Si multilayer had above 30% from 43° to 48° at 13 nm and 30% from 45° to 51°. Further studies are in progress for broadband analyzers.

5 Design, fabrication and characterization of X-ray supermirrors

Recently, with the development of hard X-ray telescopes and third generation synchrotron sources, the hard X-ray focus optics have been applied widely. For such applications, the reflective mirrors with relatively broad angular range at hard X-ray radiation, such as Cu $K\alpha$ line ($\lambda = 0.154$ nm), are required to extend the view field and flux of optics, where bent crystal and single layer metal mirrors can't be used because of too small grazing incident angle. The period multilayer mirrors have higher reflective angle than the critical angle of the single layer metal mirrors, but their narrow angular range limits their application in hard X-ray focus optics. More currently, depth-graded hard X-ray multilayer mirrors are being developed because they have broad grazing-incidence angular range or wider energy band. The depth-graded multilayer mirrors can provide a large angular range to extend the view field and flux of optical system.

The layer structures of X-ray supermirrors are the results of the mathematical optimization design. The optimized method usually used in X-ray supermirrors design is the purely numerical techniques which are starting from an initial multilayer structure. An optimization algorithm (such as the random optimization, the simplex optimization and global optimization) tries to minimize a given merit function to obtain the target reflectivity profile. The initial multilayer is vital to designing X-ray supermirror. Different optimization algorithms need different initial multilayers to get expected result and expend less calculated time. Both periodic multilayer and non-periodic multilayer structures are often used as initial structure.

The multilayers fabricated by DC magnetron sputtering were characterized using an X-ray diffractometer (XRD) made by Bede company with high accuracy at 8.0 keV. Fig. 6 shows the experiment curves of the supermirrors, which have the reflectivity profiles as a function of the grazing incident angle, and the intensity is normalized according to the maximum beam in-

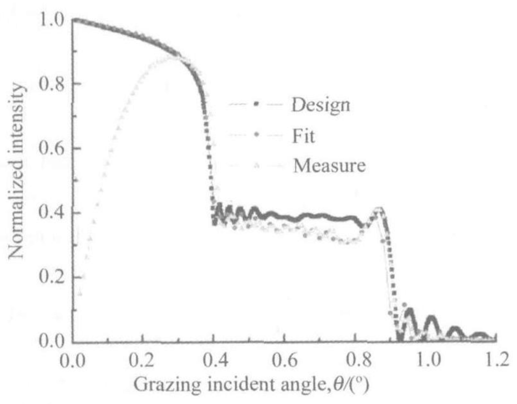


Fig. 6 Reflectivity measurements and fitting results of broadband angular 0.4～0.85 ° W/Si multilayers at wavelength of 0.154 nm

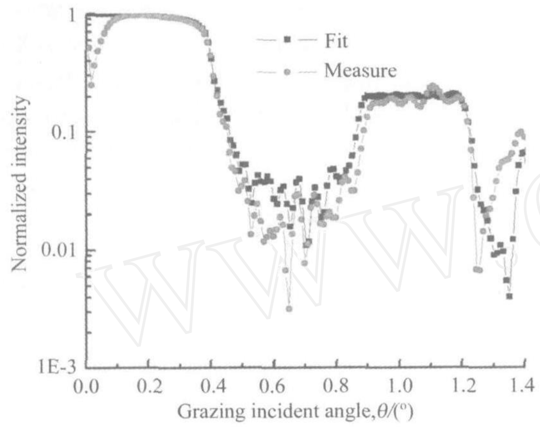


Fig. 7 Reflectivity measurements and fitting results of broadband angular 0.9～0.15 ° W/B_4C multilayers at wavelength of 0.154 nm

tensity. We also fitted several reflectivity curves choosing the interface roughness as independent parameter using the calculated model with roughness. The interface roughness of the W-on-Si was found to be about 0.5 nm, as same as the Si-on-W, and the interface roughness of the W-on-B_4C was found to be about 0.32 nm, as same as the B_4C-on-W, smaller than W-on-Si.

6 Conclusions

The design, fabrication and characterization of soft X-ray beam splitters for Michelson interferometer working at 13.9 nm, broadband analyzers for measurements of EUV and soft X-ray polarization, and X-ray supermirrors with broadband angular interval were presented in this paper. All the multilayers were deposited by DC magnetron sputtering with a half-automated deposition system made in China.

The working angle of a beam splitter for Michelson interferometer is different from that of visible light. The both sides of silicon membranes needed to coated, while just one side for Mach-Zehder interferometer. The primary experimental results show that the product of reflectivity and transmission can achieve above 3 %. The periodic multilayers have a very narrow bandwidth and are not convenient to measure the polarization of state in EUV and soft X-ray range. The measurements show that the reflectivities of broadband analyzers have a broadband which is in agreement with the design. The X-ray supermirrors with different angular bands have been made and the reflectivity measurements demonstrated that a W/Si supermirror has above 30 % reflectivity in 0.4～0.85 ° region and a W/B_4C supermirror has about 20 % reflectivity in 0.9～1.2 ° range at 0.154 nm.

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References:

[1] D. G. Stearn, R. S. Rosen, S. P. Vernon. Fabrication of high-reflectance Mo-Si multilayer mirrors by planar-magnetron sputtering[J]. *J. Vac. Sci. Technol.*, 1991, A9: 2662-2669.

[2] G. Seitz1 ,S. Schulte ,U. Dinger ,O. Hocky ,B. Fellner ,M. Rupp. EUV lithography:a challenge for optical metrology [J]. *Proc. SPIE*,2004 ,5533 :20-26.

[3] D. L. Windt ,S. Donguy ,J . Seely ,B. Kjornrattanawanich. Experimental comparison of extreme-ultraviolet multilayers for solar physics[J]. *Appl. Opt.* , 2004 ,43 :1835-1839.

[4] J . H. Underwood ,A. C. Thompson ,J . B. Kortright , K. C. Chapman ,D. Luntt. Focusing X-rays to a 1 μm spot using elastically bent , graded multilayer coated mirrors[J]. *Rev. Sci. Instrum.* ,1996 ,57 :3359.

[5] T. Aota ,N. Yamaguchi ,M. Yoshikawa , K. Ikeda , T. Ishijima , Y. Okamoto ,A. Mase , T. Tamano. Two-dimensional imaging X-ray spectrometer for plasma diagnostics[J]. *Fusion Engineering and Design*1997 ,34-35 , 189-191.

[6] L. B. DaSilva ,T. W. Barbee ,R. Jr , Cauble , *et al*. Electron density measurements of high density plasma using soft X-ray laser interferometry[J]. *Phys Rev Lett.* ,1995 , 74 :3991-3994.

[7] F. Delmotte ,M. F. Ravet ,F. Bridou ,F. Varnière ,P. Zeitoun ,S. Hubert ,L. Vanbostal ,G. Soullie. X-ray-ultraviolet beam splitters for the Michelson interferometer[J]. *Appl. Opt.* 2002 ,41 :5905-5912.

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