High reflectivity Co/Mg multilayer working in the broad soft X-ray range of 350-770 eV

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Abstract: Co/Mg multilayer was proposed and optimized to work in the broad soft X-ray range of 350-770 eV. The multilayers fabricated using magnetic sputtering technique showed relatively large interface width and surface roughness. It was improved by using a higher Co sputtering power which made the polycrystalline structure more ordered. The effect of Ar sputtering pressure on the layer structure was also studied. The measured reflectivity results indicated high reflectivity from 22% to 31% at 400-650 eV can be reached if a saturated number of bilayers of 50 is deposited.

1. Introduction

Soft X-ray spectroscopy and imaging techniques working in the photon energy range of 350-770 eV are widely used in biology, material science, archaeology researches and plasma diagnostics [1-3]. To perform such experiments, high performance monochromator composited with multilayer mirrors and ~~the~~ recently developed multilayer gratings [4, 5] are required for selecting desired spectrum efficiently. Nevertheless, due to the large absorption of materials and multiple absorption edges located in this range, a pool of different multilayer design can cover this broad energy range with practical efficiency. For instance, Cr/Sc, Cr/Ti, Cr/V multilayers have been widely used in the range below the Sc-L (398 eV), Ti-L (453 eV) and V-L (512 eV) absorption edges, respectively with high efficiencies[6, 7], while the efficiency drops dramatically in the energy range above the edges. Other common multilayers, like W, Mo for absorbing layers and Si, C for spacer layers, have no absorption in this range, while their absorption coefficient are quite large in the low energy range [8, 9]. It makes the theoretical reflectivity only 5%-10% at around 350 eV (Figure 1). Mg has almost the lowest absorption in this range which makes it an ideal material for the spacer layer of a multilayer. For the absorbing layer, SiC is typically used to combine with Mg given to their high performance at around 30.4 nm for EUV astronomical observation [10]. However, the optical contrast between Mg and SiC at soft X-ray region is quite small which presents similar low reflectivity as W/Si. Compared with SiC, Co has a much larger optical contrast with Mg, and its absorption is much smaller than W, Mo, etc. Replacing SiC with Co can provide a much higher reflectivity of 26%-46% in the whole range of 350-700 eV. Although the reflectivity of Co/Mg multilayer starts to decrease at above 700 eV, the reflectivity at 750 eV is still 24%. Therefore, Co/Mg multilayer can be an ideal candidate to work in the whole range of 350-770 eV theoretically.

To estimate the optical performance of Co/Mg multilayer, the reflectivity in the photon energy range of 350-800 eV was simulated using IMD software [11]. The interfaces were assumed to be ideal, which meant there was no roughness or diffusion. The period was 6.0 nm, which was selected for the Co/Mg multilayer blazed grating applied on soft X-ray spectrometer that will be fabricated in our future work. The bilayer number was 50 to reach the saturated reflectivity. The thickness ratio of Co was set as 0.4. The optical constants were derived from the Centre for X-ray Optics website (CXRO). For comparison, the reflectivities of Mg/SiC, Cr/Sc, Co/C, W/Si, and Mo/Si multilayers were also simulated and the theoretical reflectivity at different energies are shown in Fig. 1. The reflectivities of Co/Mg multilayer reaches 26% to 46% from 350 eV to 700 eV and start to decrease when the photon energies are larger than approximately 700 eV. Co/C multilayer shows slightly lower reflectivity than that of Co/Mg multilayer because of the lower optical contrast. Mg/SiC, W/Si and Mo/Si multilayers exhibit gradually increased reflectivity with photon energy up to 800 eV. However, the reflectivities in low energy range of these multilayers are extremely low. For Cr/Sc multilayer, because the optical contrast between Cr and Sc is small and the absorption of Sc is strong in this range, it exhibits an extremely low reflectivity when the photon energy is larger than 400 eV. According to the simulation results, Co/Mg multilayer had highest theoretical reflectivity over the broad energy range from 350 eV to 700 eV, and the refletivity is still useable at 700-770 eV. Therefore, we proposed using Co/Mg multilayer to fabricate reflection and dispersion multilayer optics in this range.

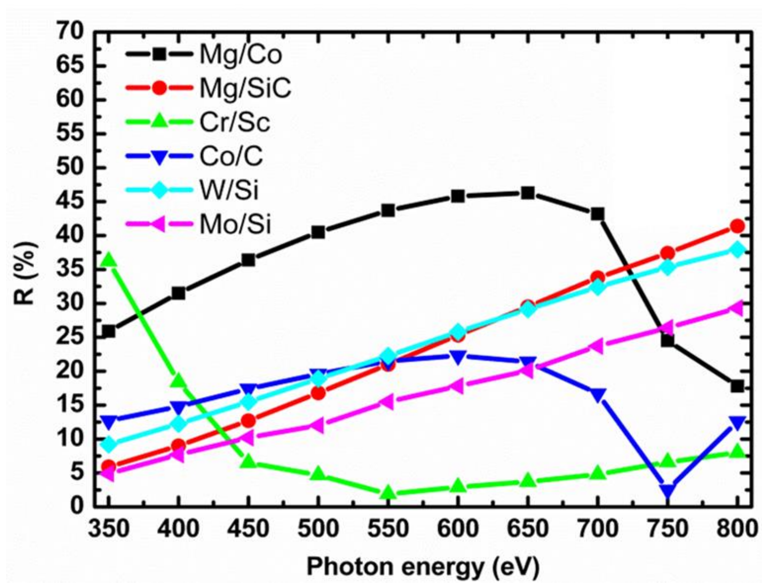

Fig. 1. The simulated reflection curves of Co/Mg, Mg/SiC, Cr/Sc, Co/C, W/Si, and Mo/Si multilayer. The period of all multilayers was 6.0 nm and the bilayer number was saturated. The thickness ratio of layer with larger absorption to the multilayer period was set as 0.3~0.4. The interfaces were assumed to be ideal, i.e. no interface width was considered here.

The layer properties of Co/Mg multilayers with relatively large period of around 16 nm have been studied for the application in EUV region [12-14]. The Mg and Co individual layers were demonstrated to be partially crystallized with hcp structure. The interface widths were about 0.5 nm as fitted from the hard X-ray reflectivity curves [12], which are larger than other high quality multilayers, like W/Si [15]. No chemical reaction was found between Co and Mg atoms as measured by the X-ray emission spectroscopy. A high EUV reflectivity of 40.3% of the Co/Mg multilayer was measured at 30.4 nm under near normal incidence [12]. The thermal stability of Co/Mg multilayer was also studied. The reflectivity began to decrease at 300 °C and the layer structure was destroyed at 400 °C. The thermal stability can be improved by adding Zr barrier layer at the interfaces [13].

In soft X-ray range, due to the much shorter optical wavelength, the period of Co/Mg multilayer is decreased to only a few nanometers to realize Bragg reflection. This can significantly change the layer growth behavior and interface quality of the multilayer. The layer structure and optical performance of Co/Mg multilayers with such small period remain unknown. On the other hand, the smoothening effect of the multilayer growth needs to be suppressed if deposited on a nanoscale grating structure to form a multilayer grating [4]. A simple way is to increase the sputtering pressure during deposition of the multilayer, which can make the deposited atoms having less kinetic energy and smoothening effect. It has been demonstrated on the Mo/Si multilayer blazed grating, in which the smoothening of the grating structure was obviously reduced by increasing the sputtering pressure from 1.0 mTorr to 3.5 mTorr [16]. One negative effect of increasing the sputtering pressure is causing the relative larger surface roughness of multilayers while the exact effect on different multilayers need to be studied specifically.

Thus, driven by the demand of high reflectivity multilayer working in the whole photon energy range of 350 -770eV, Co/Mg multilayers with a small period of approximately 6 nm were deposited and studied. The multilayers were deposited under different sputtering power of Co and sputtering pressure (Ar gas) to optimize the layer growth quality. A larger sputtering power of Co significantly reduced the interface width of the multilayer which can be explained by the formation of well-ordered polycrystalline structure. However, the larger sputtering pressure increased the interface width and the surface roughness, and the rough surface is not suitable for the coating of nanoscale gratings. Based on the optical characterization results, a high reflectivity of 22%-31% can be reached in this soft X-ray region.

2. Experiments

Five Co/Mg multilayers were fabricated using a direct-current magnetron sputtering system. The multilayers were deposited on polished Si (100) wafer segments with 20 mm × 20 mm, which have surface roughness of 0.2 nm. High purity Ar gas (99.999%) was used for the sputtering. The background vacuum pressure was less than 3×10^{-5} Pa during the deposition. The power applied on Mg target was 15 W due to the high growth rate of Mg. The period of all multilayers was approximately d=6.0 nm and the bilayer number was N=20.

Three of the multilayers were deposited under different Co target sputtering power at 20 W, 40 W and 60 W. The sputtering pressure was kept at 1.0 mtorr during the deposition process. The other two multilayers were deposited under different Ar pressure of 2.0 and 3.0 mtorr. The power applied on Co target was set at 60 W. After the deposition, the grazing incident X-ray reflection (GIXRR) measurements were firstly performed on a X-ray diffractometer (D1 system, BEDE Inc.) with Cu K-α line (Photon energy E=8.04 keV) light source. The obtained GIXRR curves were fitted using BEDE Ref software [17], to determine the multilayer period and interface widths. Atomic force microscopy (AFM) measurements were performed on the samples using a Bruker Dimension Icon system, to analyze the surface morphology. The scanned area was 1 μm × 1 μm with 256×256 pixels. One-dimensional power spectrum density (PSD) functions were computed from the height data. The algorithm for PSD calculation was described in our previous study [18].

In order to find out the mechanism of the effect of Co sputtering power on the multilayer structure, the samples fabricated under Co sputtering power of 20 W and 60 W were characterized by Transmission Electron Microscopy (TEM) measurement. The bright field with magnification factor of 30 K was used to observe the uniformity of the layers and the roughness of the interfaces. The high resolution bright field measurement with 1000 K magnification factor was used to characterize the interface diffusion and polycrystalline structure of the layers. In addition, the Selected Area Electron Diffraction (SAED) measurement was conducted to determine the crystal structure.

The X-ray diffraction (XRD) measurement were performed on the Co/Mg multilayers fabricated under sputtering power of 20 W, 40 W and 60 W, using the θ-2θ scanning mode. Only the crystal grains with atomic planes parallel to the multilayer interface can be detected in this mode. The measurement process was explained in our previous study [18]. By matching the angular positions of diffraction peaks with the International Centre for Diffraction Data (ICDD) powder diffraction file, the crystal structure can be determined.

The soft X-ray reflectivity measurements were performed on the reflectometer at the Optics Beamline of BESSY II Synchrotron Radiation Laboratory in Berlin, Germany. The size of the beam incident on the sample was approximately 0.35 mm × 0.2 mm. The energy scans were measured in a broad energy range from 350 eV to 770 eV at different grazing angles of 8.0°, 9.4 º, 11.2° and 14.9°, respectively.

3. Results and Discussion

3.1 Co/Mg multilayers fabricated under different Co sputtering power

The measured GIXRR curves of the three Co/Mg multilayers fabricated under different sputtering powers of 20, 40, 60 W are shown in Fig.2. The same-order Bragg peaks of different samples had similar angular position, indicating consistent periods of the multilayers. More Bragg peaks were observed with higher sputtering power, which meant the quality of the multilayer structure was significantly improved. These curves were fitted using a two-layer model consisting of 20 bilayers of Mg and Co. The fitted curves are shown as red lines in Fig.2, and the fitted parameters are listed in Table. 1. The periods and Co thickness ratio were close to the designed value, which are 6.0 nm and 0.4, respectively. The interface widths of the multilayers fabricated at 20 W were 1.95 and 1.07 nm for Mg-on-Co and Co-on-Mg interfaces, respectively. The large interface widths decreased the reflectivity and degraded high-order Bragg peaks, especially the higher orders. As the sputtering power increased to 40 W and 60 W, the interface widths decreased. When the power was 60 W, the interface widths were only 0.62-0.65 nm. Further increasing the power to 80 W did not bring smaller interface width. It is evident that the sputtering power of 60 W is optimal for depositing Co/Mg multilayers with good interface quality.

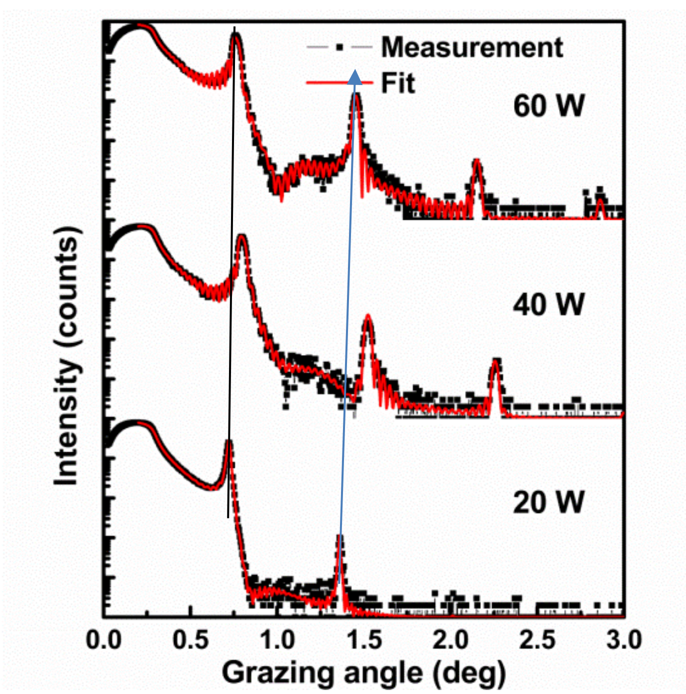

Fig. 2. The measured and fitted GIXRR curves of Co/Mg multilayers fabricated under different Co sputtering power.

Table 1. Fitted results of the sample fabricated under different Co sputtering powers.

Co target sputtering power (W)	Thickness (nm)		Period (nm)/Thickness ratio	Interface width (nm)	
	Co	Mg		Mg-on-Co	Co-on-Mg
20	2.78	3.76	6.54 (0.42)	1.95	1.07
40	2.55	3.36	5.91 (0.43)	0.79	0.67
60	2.39	3.81	6.20 (0.39)	0.65	0.62

The surface morphologies of the Co/Mg multilayers fabricated under different sputtering powers of 20 W and 60 W are shown in Fig. 3(a) and (b). Both images exhibited rough surfaces with grain-like features of 20-40 nm. However, the heights of the grains of the sample fabricated at 20 W were significantly larger, indicating a rougher multilayer surface. The one-dimensional PSDs of the AFM images are shown in Fig. 3(c). It shows that the PSD of the sample fabricated at 20 W was obviously higher than that of the sample with 60 W in the whole spatial frequency range. The RMS values of surface roughness were 1.0 nm and 0.52 nm for the sample fabricated at 20 W and 60 W, respectively. These results indicated that the interface roughness of Co/Mg multilayers can be reduced by increasing Co sputtering power, which were consistent with GIXRR fitting results.

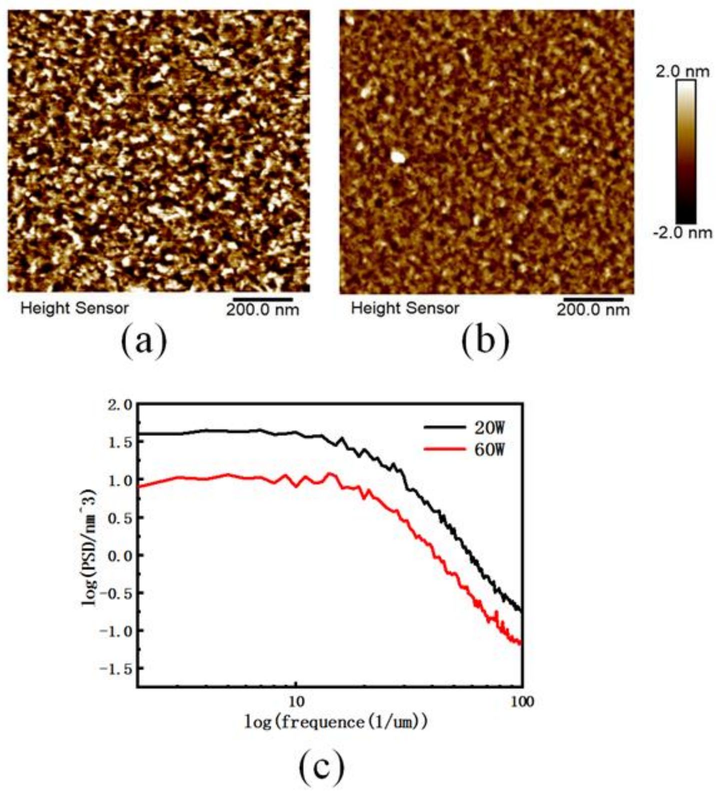

Fig. 3. (a), (b) AFM images of the Co/Mg multilayers fabricated under Co sputtering powers of 20 W and 60 W. (c) The PSD curves derived from the AFM images.

3.2 Co/Mg multilayers fabricated under different Ar pressure

The measured GIXRR curves of the three samples fabricated under different Ar pressures are shown in Fig. 4. Four Bragg peaks can be observed in the GIXRR curve of the sample fabricated under Ar pressure of 1.0 mtorr. As the Ar pressure increased to 2.0 mtorr, the fourth Bragg peak disappeared. Further increasing the pressure to 3.0 mtorr caused significantly decrease of the third Bragg peak. The GIXRR curves were fitted and the results are listed in Table. 2. For the Ar pressure of 1.0 mtorr, the interface widths are 0.65 and 0.62 nm for Mg-on-Co and Co-on-Mg interface, respectively. For the pressure of 3.0 mtorr, the interfaces widths increased to 0.79 and 0.74 nm. The increasing trend of the interface widths with higher pressure is slightly larger than other multilayer systems like Mo/Si [19] and W/Si [15]. Considering the smallest interface widths of the Co/Mg multilayers fabricated here is already large, the higher Ar pressure is not preferred.

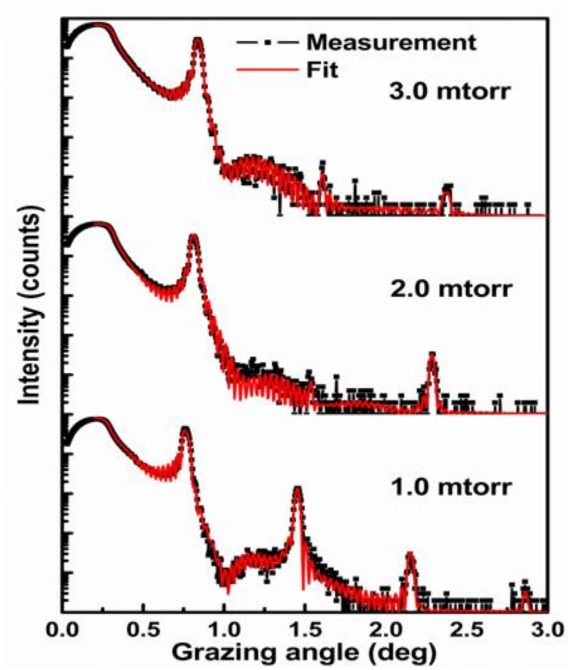

Fig. 4. The measured and fitted GIXRR curves of Co/Mg multilayers fabricated under different Ar pressures.

Table 2. Fitted results of the sample fabricated under different Ar pressures.

Ar pressure (mtorr)	Thickness (nm)		Period (nm)/Thickness ratio	Interface width (nm)	
	Co	Mg		Mg-on-Co	Co-on-Mg
1.0	2.39	3.81	6.20 (0.39)	0.65	0.62
2.0	2.38	3.44	5.82 (0.41)	0.70	0.66
3.0	2.25	3.39	5.64 (0.40)	0.79	0.74

The AFM images of the Co/Mg multilayers fabricated under different Ar pressures are shown in Fig. 5(a)-(c). The surface morphology changed little when the Ar pressure increased from 1.0 mtorr to 2.0 mtorr. With the Ar pressure increasing to 3.0 motrr, the multilayer surface was significantly rougher. The PSD curves are shown in Fig. 5(d). As the pressure increased from 1.0 mtorr to 2.0 mtorr, the PSD curve only increased slightly at the middle spatial frequency range of 20-50 um^{-1}. The PSD of the sample fabricated under 3.0 mtorr was higher than that of the other two samples in the almost whole spatial frequency range of 8-100 um^{-1}. The surface roughness were 0.52 nm, 0.56 nm, 0.66 nm (RMS) for the corresponding multilayer fabricated at 1.0 mtorr, 2.0 mtorr, 3.0 mtorr, respectively. For the high line density X-ray gratings with only a few nanometer groove height, these rough surface morphology can significantly modify the groove profile and decrease the efficiency which need further optimization.

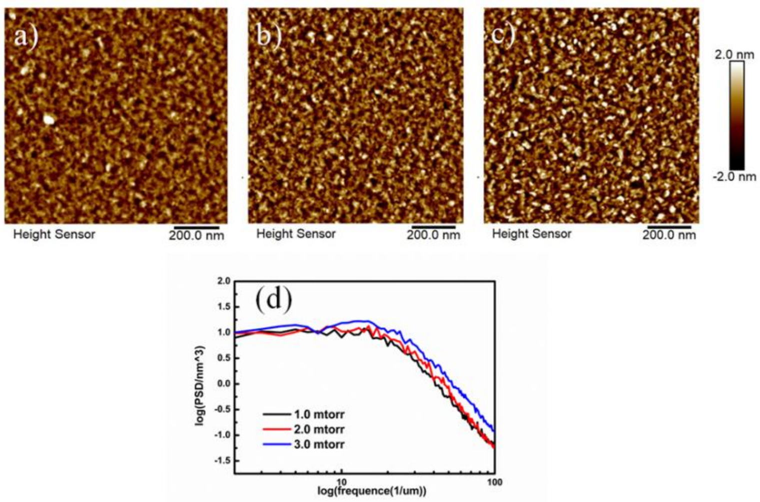

Fig. 5. (a), (b), (c) AFM images of the samples fabricated under different Ar pressures. (d) PSD curves derived from the AFM images.

3.3 Transmission electron microscopy (TEM) measurement

The bright field images of the Co/Mg multilayer fabricated under Co sputtering power of 20 W and 60 W are shown in Fig. 6(a) and (b), respectively. The dark and white stacks were Co and Mg layers, respectively. When the sputtering power was 20 W, the interfaces of the Co/Mg multilayer were rough and the diffusions at the interfaces were strong. The measured thicknesses of Mg and Co layers at different spot were not consistent because of the non-continuous agglomerations. With the Co sputtering power increasing to 60 W, the interfaces were smoother and flatter; the diffusions were suppressed. These changes were related with the crystallization state variations of the layers. The measured thicknesses of Mg and Co layers were 3.83 nm and 2.39 nm, which were consistent with the GIXRR results. Fig. 6(c) and (d) were the bright field images with magnification factor 1000 K of the Co/Mg multilayer which can be used to observe the details of the interfaces and the crystallization state of the layers. For the Co/Mg multilayer fabricated under Co sputtering power of 20 W, the Mg layers were partially crystallized and the crystal grains were arranged disorderly, which leaded the rough interfaces. In addition, large amounts of amorphous Mg atoms diffused into the Co layers strongly, which made the interfaces rough and blurred. According to Fig. 6(d), when the Co sputtering power was increased to 60 W, the crystallization in Mg layers was enhanced significantly and the crystal grains had preference orientation which was parallel to the multilayer interface. Clear crystal grains can also be observed in the Co layers, which indicated the crystallization was increased. These made the interfaces smoother and sharper. Fig.6 (e) and (f) shows the SAED patterns of the Co/Mg multilayers fabricated under Co sputtering power of 20 W and 60 W, respectively. When the Co sputtering power was 20 W, the pattern diffracted from Mg layers were two non-continuous rings which were diffracted from Mg (002) and (100). The pattern diffracted from Co layers was a broad and faint ring which indicated the crystallization in Co layers was extremely weak. When the Co sputtering power was increased to 60 W, strong diffraction spots of Mg (002) were observed which indicate a more ordered crystalline structure of Mg. The Mg (100) crystal plane was not observed. The patterns diffracted from Co layers also changed from broadened ring to several bright spots, implying a significantly enhanced crystallization.

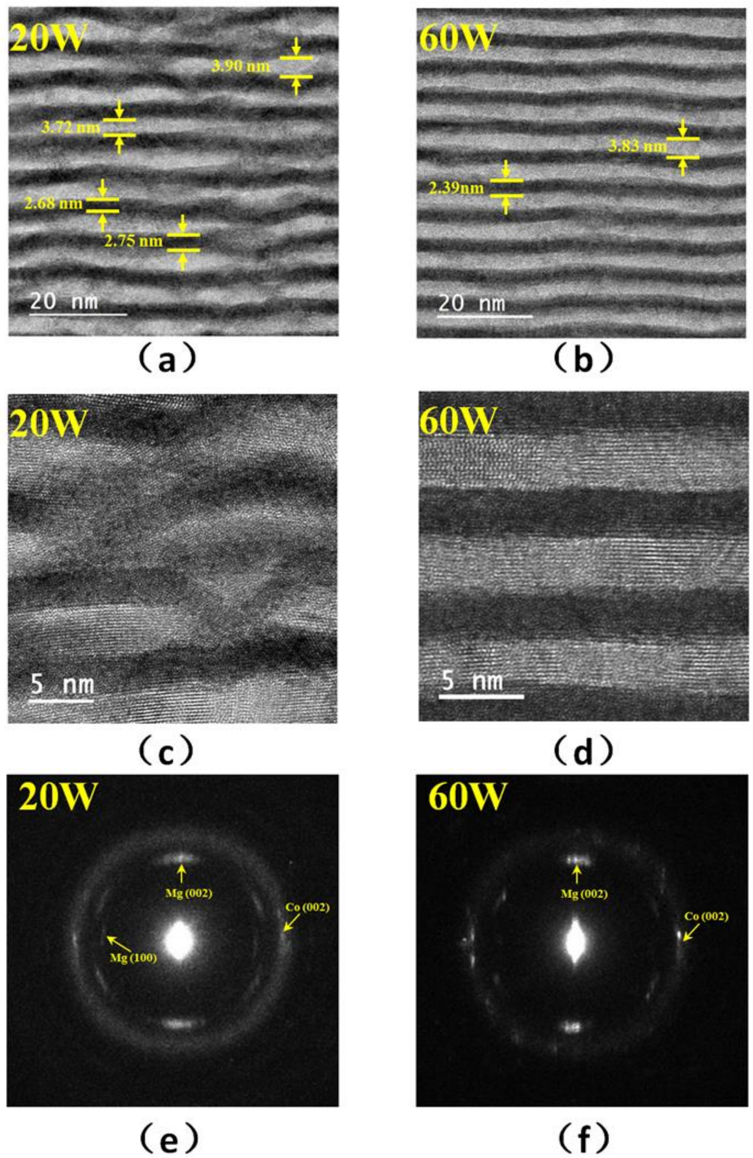

Fig. 6. The TEM results of the Co/Mg multilayers fabricated under Co sputtering power of 20 W and 60 W. (a) and (b) are the bright field images with magnification factors of 300K. (c) and (d) are the bright field images with magnification factors of 1000 K. (d) and (e) are the measured SAED patterns.

3.4 X-ray diffraction measurement

Fig. 7 shows the results of the XRD measured in θ-2θ mode of the Co/Mg multilayers fabricated under Co sputtering power of 20 W, 40 W and 60 W. In this mode, only crystallites with lattice planes parallel to the layer interface can be detected. The TEM results showed the Mg and Co layers were partially crystallized disorderly for the samples fabricated under sputtering power of 20 W. However, there was no observable diffraction peak for this sample because the lattice with parallel to the layer interface was almost non-existent. With the Co sputtering power increasing to 40W, only one diffraction peak at diffraction angle of 34.1° can be observed, which was identified as Mg (002). The appearance of this peak suggested the crystallization in Mg layers increased and the crystal planes were preferred to be parallel to the multilayer interface. With the Co sputtering power increasing to 60 W, the crystallization with lattice planes parallel to the interfaces increased dramatically which corresponds to the phase of Mg (002). The crystallization in Co layers was also increased, which is consistent with the TEM results. The strong crystallization of Mg and Co helped to form well-ordered atomic structure at the interfaces and reduced the interface roughness and interdiffusion.

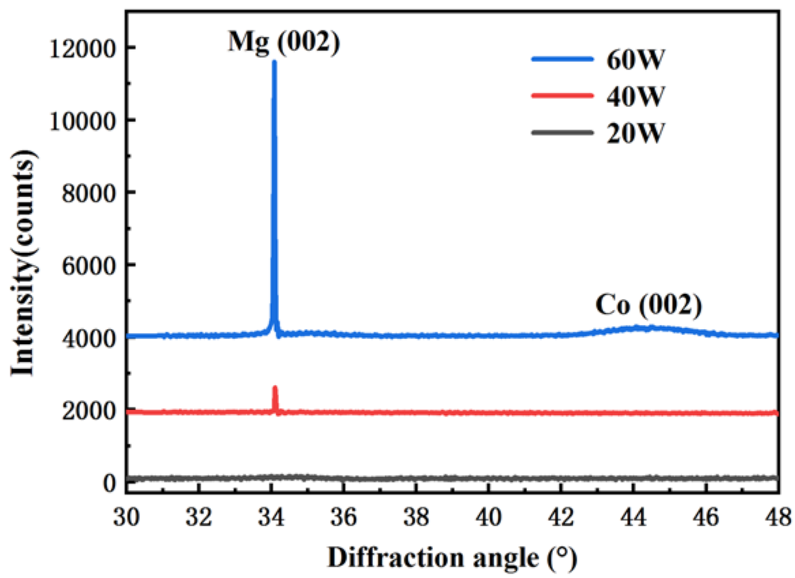

Fig. 7. The XRD results of the Co/Mg multilayers fabricated under different Co sputtering powers.

3.5 Soft X-ray reflectivity measurement

The reflectivity of the Co/Mg multilayer fabricated under Co sputtering power of 60 W and Ar pressure of 1.0 mtorr was measured at various grazing angles of 14.9°, 11.2°, 9.4°, 8.0°. The results are shown in Fig. 8 as scatter lines. The reflectivity at the photon energies of 410 eV, 543 eV, 650 eV and 740 eV are 15.8%, 19.5%, 18.2%, and 5.0%, respectively. The maximum reflectivity was obtained at 543 eV. The stronger absorptions of Co layers and the rough interfaces decreased the reflectivity to only 5%-10% at high energy range. The energy scan curves were calculated using the model with the GIXRR fitted structure parameters. The calculated curves are shown in Fig 8 as solid lines. The measured and calculated curves matched very well with each other, which indicates that the GIXRR fitted parameters were accurate and can be used to predict the reflectivity in soft X-ray range. Based on that, the saturated reflectivity of the Co/Mg multilayer with bi-layer number of 50 was calculated, the results were shown as the STARs in Fig 8. The reflectivity in soft X-ray range of 400eV to 650eV was increased to 22% to 31% with the maximum value of 31% at 543 eV. To the best of our knowledge, this is the highest reflectivity of a multilayer that ever reported in this range.

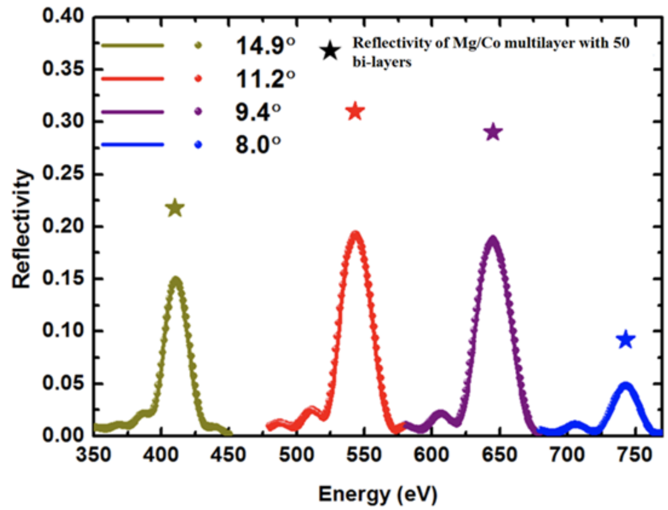

Fig. 8. The soft X-ray reflectivity of the Co/Mg multilayer. Scatter and solid lines are the measured and calculated reflectivity curves, respectively. The stars represent the reflectivity with saturated bi-layers.

4. Conclusion

In this paper, we proposed to use Co/Mg multilayer to fabricate reflection and dispersion multilayer optics in the photon energy range of 350-700 eV. The Co/Mg multilayers were fabricated under different Co sputtering power and Ar pressure. The GIXRR fitted results showed that the average interface width decreased from 1.51 nm to 0.63 nm when the Co sputtering power increased from 20 W to 60 W. Meanwhile, when the Ar pressure increased from 1.0 mtorr to 3.0 mtorr the average interface increased from 0.63 nm to 0.77 nm. The TEM and XRD results showed that as the Co sputtering power increased to 60 W, the crystallization of Mg and Co layers were significantly increased, especially for Mg. The lattice planes of the crystallites were mainly parallel to the multilayer interface, which made the interfaces smoother and sharper. A high soft X-ray reflectivity of 22%-31% can be achieved over the range of 400 ev to 650eV if a saturated number of bilayers was deposited. This investigation proved that Co/Mg multilayer has high reflectivity in the broad soft X-ray range. It can be used to fabricate high efficiency reflection and dispersion optics in this range. In the future, the relatively large surface roughness of the multilayer need to be further optimized for its application in grating optics.

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