Al/Zr multilayer mirror and its thermal stability for EUV application

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Abstract. Two kinds of Al/Zr (Al(1%wtSi)/Zr and Al(Pure)/Zr) multilayers in the region of 17-19nm were deposited on fluorine doped tin oxide coated glass by using direct-current magnetron sputtering technology. Based on the fitting data of grazing incident X-ray reflection and near-normal incident (EUV) reflectance, the interfacial roughness in the Al(1%wtSi)/Zr is lower than that in Al(Pure)/Zr because of the presence of silicon in Al. For the further characterization of Al(1%wtSi)/Zr multilayers, six samples deposited on Si substrates were annealed from 100 °C to 500 °C temperature in a vacuum furnace for 1 h. Based on the results of EUV and X-ray diffraction, the Al(1%wtSi)/Zr multilayer has a stable structure up to 200 °C, and keeps almost the similar EUV reflectivity as the non-annealed sample. After 300 °C, the amorphous of Al-Zr alloy is transformed to polycrystalline in the interface, which could be the reason for the decrease of EUV reflectivity. The polycrystalline Al-Zr compound does not destroy the multilayer completely even up to 500 °C.

1. Introduction

With the research of reflective multilayer coatings in the extreme ultraviolet (EUV) spectral region in the practical applications [1-2], the Al–based [3–6] systems are widely researched in the recent years to meet the practical requirements. Owing to the low absorption below the Al L-edge near 17 nm, the Al can be a good spacer layer in the multilayer combination. However, there are two problems the Al-based multilayer: the inhomogeneous crystallization of Al and the interdiffusion [3]. Recently, Al/Zr multilayer has shown very promising performance in the EUV region [4-6]. From the analysis of Al(1.0%wtSi)/Zr periodic multilayer with a large number of periods in our previous paper [5], we found that the interfacial roughness is not constant throughout the sample, the roughness being smaller in the first 40 periods and then increasing in the upper layers in a large number of periods of
the Al(1.0%wtSi)/Zr periodic multilayer. However, the optical performance of Al(1.0%wtSi)/Zr with 40 periods was limited by the variable interfacial structure. In the extreme ultraviolet (EUV) spectral regions, the solar corona is rich with line emission from a variety of ions, formed at plasma temperatures of up to several million degrees. Therefore for the solar satellite instruments, beside the high reflectivity, the thermal stability of multilayer is another crucial problem for the practical application [1-2]. However, the thermal stability of Al(1%wtSi)/Zr multilayer has not been evaluated yet.

In this paper, we describe the performance of the Al(1%wtSi)/Zr multilayers for the purpose of coating the multilayer on normal-incidence telescopes tuned to specific emission lines (i.e. Fe-IX (λ=17.1 nm), Fe-XI (λ=18.0 nm) and Fe-XII (λ=19.3 nm)) in the wavelength range of λ~17-19nm. The experimental process is described in the Sec.2. In Sec.3-1, two different multilayer types (Al(1%wtSi)/Zr and Al(Pure)/Zr) with different periods (N=40, 60) are presented. The grazing incident X-ray reflection (GIXR) and near-normal incident EUV reflectance are used to investigate the structure properties of the multilayers. To fully estimate the Al(1%wtSi)/Zr multilayer, the effect of the thermal stability is discussed in the Sec.3-2. We conclude in Sec.4 with comments regarding the performance for the Al/Zr system.

2. Experimental methods

All samples were fabricated by direct-current magnetron sputtering technology [5-7] with the base pressure 8.0×10⁻⁵Pa. The sputtering targets with diameter of 100 mm were zirconium (99.5%), aluminum (99.999%, Al(Pure)) and silicon doped in aluminum (Al(1%wtSi)). Under a 0.18 Pa argon (99.999% purity) pressure, the samples deposited on fluorine doped tin oxide coated glass (FTO) substrates are Al(1%wtSi)/Zr (N=40, 60) and Al(Pure)/Zr (N=40, 60). While six Al(1%wtSi)/Zr multilayers were consisted of 40 bi-layers, deposited on Si polished wafers. The thicknesses of the samples on FTO are around 9.1 nm, and gamma values are 0.35, which were designed to have a high reflectivity at near 18 nm region at 5° incident angle by synchrotron radiation. While the thickness of the sample on Si wafers are around 9.3 nm, and gamma values are 0.34.

For characterizing the structure of the samples, the GIXR and XRD measurements were performed using a Cu Ka source (λ=0.154 nm). The EUV reflectivity measurements were made at a 5° incident angle, using the reflectometer at the Spectral Radiation Standard and Metrology Beamline and Station (beamline U26) at the National Synchrotron Radiation Laboratory in Hefei, China.

3. Results and discussions

3.1 Al (1%wtSi)/Zr and Al (Pure)/Zr multilayers

In order to estimate the influence of Si doping in the Al layers [3], we firstly compare two systems of Al (1%wtSi)/Zr and Al (Pure)/Zr with the same period numbers (40 and 60). Fig.1(a) shows the x-ray reflectivity obtained by GIXR and the best-fitted curves with four layer model [6] used the Bede Refs software (genetic algorithm) [8] for the Al (1%wtSi)/Zr and Al (Pure)/Zr samples with 40 periods. The calculated root-mean-square (rms) roughness was about 0.95 nm for the Al(1%wtSi) layers and was about 0.4 nm for the Zr layers. Both rms of Al-on-Zr, Zr-on-Al interlayers were 0.5 nm. While with the similar rms for the Al-on-Zr, Zr-on-Al interlayers and Zr layers in the Al (Pure)/Zr multilayers, only the rms of Al (Pure) layer was larger than that of Al(1%wtSi) layer in 0.2 nm. For the 60 periods, the
situation is similar, which the Al(Pure) layer has larger roughness. The measured and simulated EUV reflectivity curves are shown in Fig.1(b). With the same fitting models in GIXR, the reflectance of periods 40 and 60 are only 37.9 % at 17.8 nm and 33.2 % at 18.2 nm, while the values in Al(1%wtSi)/Zr system are 41.2 % at 17.8 nm and 37.8 % at 18.0 nm, respectively. The lower reflectance of Al(Pure)/Zr multilayers indicate the higher interfacial roughness in this multilayer systems, consistent with the fitting data in the GIXR. From the above analyses, it can be seen that the Si doping in Al layers can influence the reflectivity for the two system, where the Al (1%wtSi)/Zr multilayers have higher values than those of Al (Pure)/Zr. Therefore, we decide to focus on Al (1%wtSi)/Zr system to characterize its thermal stability.

Fig.1 (a) The curves of fitting data with 40 periods for Al (1%wtSi)/Zr (red solid line) and Al (Pure)/Zr (blue line and symbol) are presented, the experimental measurements (black open points) are also shown in it; (b) (Color online) Measured and simulated reflectance versus wavelength of the multilayers at 5° incident angle by synchrotron radiation.

3.2 The thermal stability of Al (1%wtSi)/Zr multilayer

In the previous section, we found that the Al(1%wtSi)/Zr multilayer had better optical performance in the region of 17-19 nm. Except for the higher reflectance, the thermal stability of the multilayers should also be concerned in the practical applications. Therefore, the five multilayer samples, except for room temperature (RT) sample, were annealed at temperatures of 100, 200, 300, 400 and 500 °C in a vacuum furnace for 1 h, respectively. After annealing at different temperatures, the samples were cooled to room temperature naturally in a vacuum furnace with a base pressure of 3×10^{-4} Pa. In Fig.2(a), the measured EUV reflectivity curves are shown. The measured reflectivity (40.4%) is lower that the value calculated theoretically (70.9%) because of different impact factors [6]. With the increasing annealing temperature, the reflectivity increases to 41.7 % at 100 °C, and then down to 16.2 % at 500 °C. Before 300 °C, the peak positions of different annealing samples maintains at 18.5 nm. From 300 °C, the peak position moves to 18.3 nm, and apparently shifted to 17.9 nm at 500 °C.
Fig. 2 (a) (Color lines) Measured reflectivity versus wavelength of the multilayers at 5° incident angle by synchrotron radiation. b: Diffraction curves of the samples of Al(1%wtSi)/Zr (color lines) with different annealed temperatures (RT, 100 °C, 200 °C, 300 °C, 400 °C and 500 °C).

As shown in Fig. 2 (b), different XRD patterns are observed for different annealed samples. Before 300 °C, there are three main peaks: 38.7 ° (Al<111>), 35.3 ° (Zr<002>), 36.5 ° (Zr<101>) in the XRD pattern of the samples (RT, 100 °C, 200 °C). Based on the results of EUV measurements for 100 °C sample, we found that the crystallization of Zr could influence EUV reflectivity and decrease the interfacial roughness. From 300 °C, all main peaks are changed. When the temperature is at 500 °C, the peak positions of both Al<111> and Zr<101> are decreased 0.2 °, which show that a new polycrystalline Al-Zr alloy compound may be formed at the interface. The Zr<002> peak still has a small contribution around 35.3 ° in the curves. Thus, we can conclude the Zr material still exists at 500 °C. The phases of polycrystalline Al-Zr alloy could be about the similar positions as Al<111> and Zr<101>, which shift the peak positions in the Fig.2 (b). Based on our previous paper [6], the interlayers of RT sample consisted of an amorphous Al-Zr alloy in the multilayer. Considering the results in the XRD (Fig. 2 (b)), the multilayer consist of a similar multilayer structure before 300 °C, which cannot influence the main peaks in the XRD patterns. While from 300°C, the amorphous Al-Zr alloy may be transformed to polycrystalline Al-Zr alloy. That is why all the peaks show the different peak positions in the XRD patterns. This structural change of the multilayer between 200 and 300 °C should be the main reason for the decrease of EUV reflectivity (Fig. 2 (a)). It can be concluded that the new polycrystalline Al-Zr compounds increases the interfacial roughness and degrades the optical contrast of the multilayer structure.

4. Conclusion

The two systems of the Al(1%wtSi)/Zr and Al(Pure)/Zr multilayers in the 17-19 nm are presented. According to the analyses of the GIXR and EUV, the Si doping in Al layer has a great influence on the roughness of Al layer and increases the reflectivity of the Al(1%wtSi)/Zr multilayer in both periods (N=40, 60). To further evaluate performance of Al(1%wtSi)/Zr multilayer, we deposited six samples to detect the thermal stability in different annealing temperatures. Based on the analyses of EUV, the multilayer samples annealed up to 100 °C, show higher peak reflectivity than the RT sample. The reflectivity does not decrease apparently before 300 °C. However from 300 °C, the interfacial structure of Al-Zr alloy was transformed from amorphous to the polycrystalline, which increases the interdiffusion between the Al and Zr layers and decreases the EUV reflectivity. At 500 °C, the new formed compounds Al-Zr alloy in the interfaces cannot destroyed the periodic structure of the Al(1%wtSi)/Zr multilayer completely. Therefore, in our experiment, we can concluded that the Al(1%wtSi)/Zr multilayer has a good optical performance and stable structure up to 200 °C, which is considered to be very valuable for further practical applications.

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