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Ion beam sputtered aluminum based multilayer mirrors for extreme ultraviolet solar imaging

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ABSTRACT

In this paper, we report on the design, synthesis and characterization of extreme ultraviolet interferential mirrors for solar imaging applications in the spectral range 17 nm–34 nm. This research is carried out in the context of the preparation of the European Space Agency Solar Orbiter mission. The purpose of this study consists in optimizing the deposition of Al-based multilayers by ion beam sputtering according to several parameters such as the ion beam current and the sputtering angle. After optimization of Al thin films, several kinds of Al-based multilayer mirrors have been compared. We have deposited and characterized bi-material and also tri-material periodic multilayers: aluminum/molybdenum [Al/Mo], aluminum/molybdenum/boron carbide [Al/Mo/B_4C] and aluminum/molybdenum/silicon carbide [Al/Mo/SiC]. Best experimental results have been obtained on Al/Mo/SiC samples: we have measured reflectivity up to 48% at 17.3 nm and 27.5% at 28.2 nm on a synchrotron radiation source.

1. Introduction

During the last 40 years, there has been a considerable development of normal incidence multilayer mirrors for the extreme ultraviolet (EUV) spectral domain [1]. In the field of EUV solar imaging [12 nm–40 nm], conventional multilayer coatings use silicon as low absorption material. Barbee reported on Mo/Si multilayers in 1985 [2]. Such coatings have been widely used in EUV telescopes [3,4]. Other Si-based structures have been developed and used in space in order to improve the stability, the selectivity or the reflectivity of the coating, as for example Mo_2C/Si [5], MoSi/Si [6], B_4C/Mo/Si [7,8] or SiC/Si [9].

In this paper, we present the development of ion beam sputtered multilayers based on aluminum in the spectral band [17 nm–34 nm]. The replacement of silicon by aluminum is motivated by the lower absorption of aluminum in this spectral region, which provides a better theoretical reflectivity. This implies major technological issues that are due to the tendency of aluminum to crystallize easily, which causes high roughness at the interfaces. Moreover, the high reactivity of aluminum with oxygen can cause oxidation problems. Previous results obtained in our laboratory on Al-based multilayers deposited by magnetron sputtering have shown great potentiality of such structures: normal incidence reflectivity as high as 55% at 17 nm, 50% at 21 nm and 42% at 30 nm has been measured on Al/Mo/B_4C multilayer mirrors [10,11]. Other groups have also reported interesting results on Al-based multilayers deposited by magnetron sputtering. A reflectivity of 27% at 19 nm was reported in the case of Al/SiC multilayers [12] and of 41% at 17.8 nm for the Al/Zr system [13]. Alternatively, other periodic structures with two materials have been considered for space missions, such as Mg/SiC [14]. This pair of material is very effective in the [25 nm–80 nm] range. It could achieve 40% of reflectivity at 30 nm. The goal of this study is to optimize the deposition parameters of aluminum-based multilayer by ion beam sputtering (IBS). The IBS process has been used in the past for EUV mirror coatings for several space missions (EUVI/STEREO, SWAP/PROBA2, HECOR/HERSCHEL).

In the first part, we focused on the optimization of aluminum thin films by studying the effect of several deposition parameters. Then, in the second part, we compare the results obtained with different types of Al-based multilayer mirrors.

2. Experimental details

For deposition, we have used an ion beam sputtering machine (IBS) located in a clean room of class ISO6. This system has been described in details in the previous work [15,16]. Briefly, it consists of a cryogenic vacuum chamber equipped with an ion gun, a neutralizer, a sample holder, a rotating target holder and a quartz microbalance. The sample holder is rotated in order to improve the thickness uniformity. An ion gun (Veeco, 3 cm Hollow Cathode Ion Gun) sputters successively the targets that are mounted on a rotating target holder. A neutralizer provides electrons in the chamber in order to avoid the accumulation of charges on the target in the case of insulating materials. The argon ion

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energy is set in the range of 450 eV–1000 eV and the ion current in the range of 20 mA–70 mA. Single films and multilayers have been deposited onto 2 cm diameter float glass and on Si (100) wafers. The initial surface roughness is lower than 0.3 nm for float glass and silicon substrates, as estimated from grazing incidence X-ray reflectometry (GIXR) measurements. Before sputtering, the deposition chamber was pumped down to a pressure of a few 10^{-6} Pa. Deposition takes place with a total pressure of 2×10^{-2} Pa. The deposited thickness is monitored in situ by using a quartz microbalance. In this paper, we focus on the optimization of deposition parameters for aluminum. Based on previous research by magnetron sputtering [10], we decided to compare two different target compositions for aluminum: Si doped aluminum (Al:Si, 1.5%) and Cu doped Al (Al:Cu, 2%). The deposition parameters for the other materials (Mo, SiC and B4C) were already optimized in the past [8,15].

The structural characterization of thin films and multilayer mirrors is carried out with a grazing incidence X-ray reflectometer at 0.154 nm using a Bruker Discover D8 diffractometer (Cu Kα radiation). X-ray diffraction (XRD) patterns were obtained using a Seifert 3003 PTS diffractometer (Cu Kα1 radiation). Conventional θ–2θ patterns were recorded with a 0.01° step size and 4 s counting time by step. The EUV reflectivity measurements were performed with a EUV reflectometer equipped with a laser plasma source. The low photon flux of this source does not allow measuring the absolute EUV reflectivity with good accuracy but we use this technique for preliminary analysis and comparison of the samples. Based on these results, some selected multilayer mirrors were characterized with synchrotron radiation at the BEAR beamline of Elettra Synchrotron Trieste [17]. The accuracy of the EUV reflectivity measurements is about 0.5%.

3. Results and discussion

3.1. Optimization of Al thin films

First, we have studied the influence of the ion beam current on the quality of the Al thin films with thickness ranging from 50 nm to 60 nm. All samples have been deposited with the Si doped aluminum target. We observe an increase of the number and contrast of the Kiessig fringes with increasing ion gun current from 20 mA to 70 mA (Fig. 1). Other samples, not shown on Fig. 1, have been deposited with intermediate values of ion current under similar conditions and confirm the improvement of the sample quality at higher current values. This is mainly due to the reduction of surface roughness of deposited films (see Table 1). Indeed, simulations of the data plotted on Fig. 1a show that the surface roughness decreases from 4.6 nm to 3.6 nm when the ion current increases from 20 mA to 70 mA.

Fig. 1b shows that the position of the critical angle depends on the beam current value. We see that the Al2 curve is shifted towards higher angles compared to the Al1. This means that the sample made with a current of 70 mA is denser than the one made with a current of 20 mA. By fitting these curves, we deduce an increase of the density from 2.5 to 2.65 g/cm^3 (the theoretical density of aluminum is 2.7 g/cm^3). As shown in Table 1, by increasing the ion current, we increase also the ion energy. Thus, the improvement of density and roughness at higher current is probably due to an increase of the energy of deposited Al species on the substrate. We initially assumed that this was due to the crystallization of the layers as reported on the literature [18]. However XRD measurements at large angles reveal no crystallization of Al thin films whatever the ion current. Fig. 2 present the XRD spectra of Al1 and Al2 samples and of a float glass substrate. The broad peak that appears around 25° on the three spectra is due to the amorphous phase of the glass substrate. Actually, one can clearly see in Fig. 2 that the layers deposited at 20 mA (sample Al1) or 70 mA current (sample Al2) are amorphous. Consequently, the variation of aluminum density is probably due to the layer porosity. This hypothesis is supported by the fact that GIXR measurements give differences in thicknesses

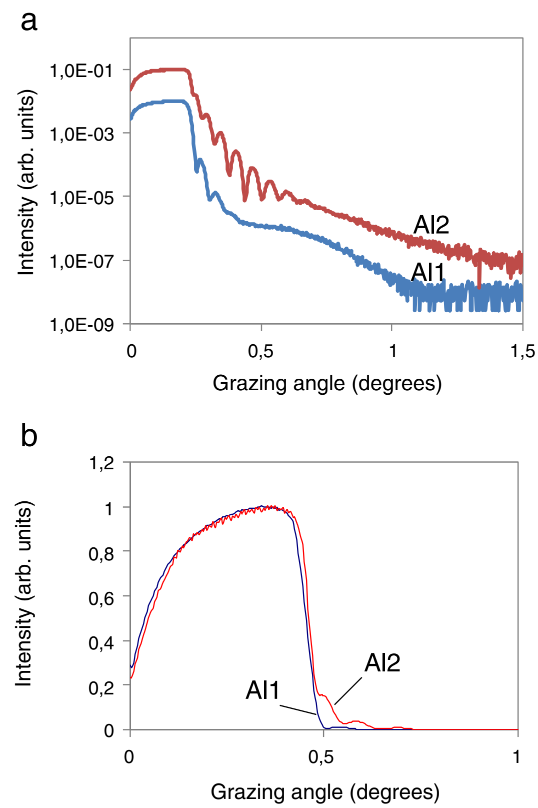

Fig. 1. Grazing X-ray reflectometry measurements of aluminum thin films deposited with 20 mA Al1 and 70 mA ion current Al2. (a) Logarithmic scale in arbitrary unit (the 2 curves are shifted for more visibility) and (b) linear scale in arbitrary unit.

between the two samples, whereas the frequencies given by the quartz microbalance were identical for both. So, we can conclude that the use of a low ion current produces porous layers. It is necessary to have an ion current high enough to obtain dense layers in the design of our multilayers.

We have also studied the influence of the target angular position, with respect to the ion beam, on the quality of the deposited aluminum films. In the initial configuration, the target is horizontal and the incident angle of the ion beam (α) is 30° (Fig. 3a). We define in Fig. 3a, the average emission angle of sputtered atoms φ_0 with respect to the normal to the target surface. We have estimated the value of φ_0 from the thickness profile of a layer deposited on a static substrate (non-rotating substrate holder). The maximum thickness of the deposited film is obtained at a distance $x_{max} = 8 \pm 1$ cm from the center of the sample. This corresponds to an emission angle φ_0 between 14° and 18°. This value is in good agreement with the values ranging from 15° to 30° reported in the literature for similar conditions [19,20].

When rotating the target, we change the angle α between the target and the ion beam (Fig. 3b). The position of the target relative to the gun also affects the direction of the lobe of sputtered particles in the deposition machine. By optimizing the position of the target, one can redirect a maximum of sputtered particles to the center of the sample.

According to X-ray reflectometry measurements (Fig. 4), we find that increasing the angle α significantly improves the quality of the deposited films. By fitting the GIXR spectra of samples Al2 to Al5, we deduced the surface roughness values reported in Table 1. Indeed, the surface roughness decreases from 3.6 nm for the nominal position ($\alpha = 30°$) to 1.3 nm for α equal to 45°. Beyond this position, the improvement stops and the risk of causing damage to the ion gun increases, because some sputtered particles are redirected to the gun.

When the target is tilted 15°, the emission lobe of sputtered particles is centered on the sample (Fig. 3b). The deposition rate is higher and the