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AlF_3 film deposited by IAD with end-Hall ion source using SF_6 as working gas

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ABSTRACT

A novel and effective process to fabricate high quality fluoride thin films was presented. Aluminum fluoride films deposited by a conventional thermal evaporation with an ion-assisted deposition (IAD) using SF_6 as a working gas at around room temperature were investigated. In this study, the optimal voltage and current, 50 V and 0.25 A, were found according to the optical properties of the films: high refractive index (1.489 at 193 nm), low optical absorption and extinction coefficient ($<10^{-4}$ at 193 nm) in the UV range. The physical properties of the film are high packing density and amorphous without columnar structure. It was proved that using SF_6 working gas in IAD process is a good choice and significantly improves the quality of AlF_3 films.

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1. Introduction

Fluorides such as AlF_3 are currently used as optical coatings for UV and deep UV applications [1–4]. It has been shown that aluminum fluoride thin film has better optical properties when it is deposited by resistive-heating thermal evaporation method [5,6]. Nevertheless, the optical and structural qualities of AlF_3 films are still needed to be improved. Therefore, in additional to a thermal evaporation an ion-assisted deposition (IAD) method is applied to improve film quality due to the extra momentum transfer [7] of ions produced by ion source on the substrate. The mechanism of ion-assistance film deposition has been extensively studied in order to achieve denser, adhesive and stress controlled coatings. Noble gas, such as Ar, is usually used as a working gas in the process to grow AlF_3 thin film. Even though the quality of the thin film is improved but low F concentration is found due to the thermal decomposition of AlF_3 during thermal evaporation process. Furthermore, contaminants such as C, O, and Ar, are detected in AlF_3 film.

Using F^- ions to enhance the F concentration and hence improve the quality of AlF_3 films in the deposition process has been demonstrated by Lee's group [8,9]. However, very few reports have been published on the topic of the application of SF_6 as a working gas with an IAD method in optical coating process at around room temperature. For the above reasons, sulfur hexafluoride (SF_6) was chosen as a working gas which provided more F^- ions dissociated from SF_6 in IAD process [10]. The deposition method with reported parameters was proved to successfully fabricate high quality AlF_3 films in this study.

2. Experiment

AlF_3 films were deposited on fused silica substrates by using a thermal molybdenum boat equipped with a homemade end-Hall ion source. The source material for thermal evaporation was AlF_3. The vacuum chamber was evacuated by a diffusion pump at the base pressure $<6 \times 10^{-6}$ Torr, whereas the working pressure controlled by a mass flow meter was about 2×10^{-4} Torr during the deposition process. Even through the AlF_3 films were deposited without IAD, SF_6 gas was fed into the chamber in order to keep the deposition process under the same working pressure. The deposition rate of AlF_3 films was 0.15–0.20 nm/s at around room

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temperature, and the thickness of the AlF_3 films was kept at approximately 300 nm.

The optical transmittance of the as-deposited AlF_3 film on the substrates was measured by a spectrophotometer (Cary 5E, Varian) in the wavelength range of 190–600 nm. The refractive index and the extinction coefficient at λ = 193 nm of the film were measured by variable-angle spectroscopic ellipsometry (VASE, J.A. Woollam Company M-2000U). The measurement was performed in the spectral range from 190 to 800 nm under angles of incidence ranging from 55° to 65° in steps of 5°. A Cauchy model was created and the experimental data were fitted by WVASE32 software prepared by J.A. Woollam Company. The fitting results of all the samples were inspected by using root-mean-squared-error (MSE) approach [11,12]. When the fitting results are the same with the measured data, it indicates that the created Cauchy model fits measured data very well, i.e., the model can be regard as the actual structure of the AlF_3 film.

The microstructure and crystallinity of the as-deposited film were examined by X-ray diffractometer (XRD). The cross-sectional morphology of the AlF_3 film was photographed by LEO 1530 field emission scanning electron microscope (FEG-SEM). Surface roughness of the film was analyzed by atomic force microscope (AFM) in a scanning area of 1 μm $\times$ 1 μm.

3. Results and discussions

3.1. (A) Optical properties

The energetic ions produced by a gridless ion source play an important role in the optical properties and the structure of the as-deposited film. The SF_6 working gas was decomposed into positive SF_5^+ ions and negative F^- ions in an IAD process. The massive positive ions lost their energies mainly due to momentum transfer and the negative F^- ions enhance the atomic concentration of F in the AlF_3 films on the substrate [10].

Fig. 1 shows the transmittance spectra of the as-deposited AlF_3 films deposited by IAD using SF_6 as working gas under various ion-beam voltages. The optimal voltage of IAD process was 50 V, because the spectrum of the sample denoted by "sample-50V0.50A" was above that of the bare substrate in the UV range. Moreover, the minimum transmittance at 342 nm closing to that of the bare substrate indicated that the film of sample-50V0.50A had not only the lowest absorption but also the largest amplitude of transmittance spectrum which indicated that the refractive index was the highest among the films.

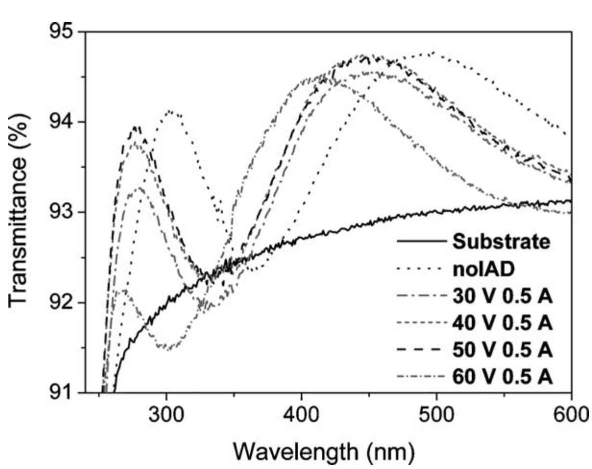

Fig. 1. The transmittance spectra of the as-deposited AlF_3 films deposited by IAD using SF_6 as working gas under various values of ion-beam voltage.

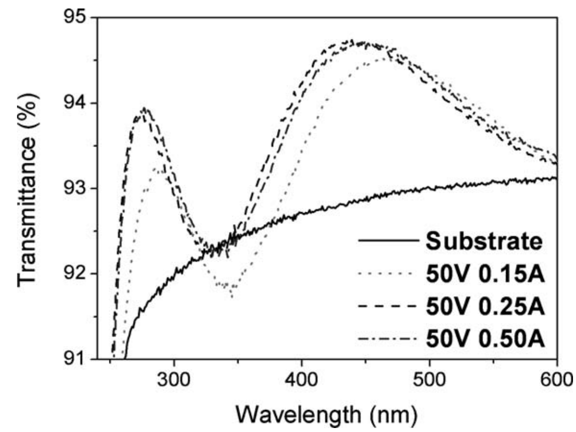

Fig. 2. The transmittance spectra of the as-deposited AlF_3 films deposited by IAD using SF_6 as working gas under various values of ion-beam current.

In order to find out the optimal current parameter of ion-assisted deposition, the ion-beam voltage was kept at 50 V and the ion-beam current was changed from 0.15, 0.25 to 0.50 A. In this way the optimal current of IAD process, 0.25 A, was found in the following reasons. The minimum transmittance spectrum of the sample denoted by sample-50V0.25A is closer to that of the bare substrate than that of sample-50V0.50A in the UV range as shown in Fig. 2. Even though the difference between sample-50V0.25A and -50V0.50A is very small, the optical properties of sample-50V0.25A, such as the refractive index and extinction coefficient, are still better than those of sample-50V0.50A as shown in Table 1.

The thickness, the root-mean-squared-error (MSE), the refractive index, and the extinction coefficient, at λ = 193 nm of all the AlF_3 films evaluated by VASE were summarized in Table 1. The refractive index of sample-50V0.25A, 1.489, is higher than that of the sample fabricated without IAD, 1.438 at λ = 193 nm, denoted by sample-noIAD. The extinction coefficient of sample-50V0.25A, $k < 10^{-4}$, is smaller than that of sample-noIAD, 1.1×10^{-3}. These results indicate sample-50V0.25A has the higher packing density and lower optical absorption. In addition, it was found that the film of sample-noIAD had inhomogeneous structure.

3.2. (B) Crystallinity, microstructure, and surface morphology

The crystallinity and structure of AlF_3 films deposited at various IAD ion-beam voltages and currents were examined by XRD, as shown in Fig. 3. All the films deposited at around room temperature were amorphous, and the broad peak at 2θ, $\sim$20°, is the signal from the fused silica substrate. Even though it is amorphous film but it does show better optical property than that of the poly-crystallization structure which causes more optical scattering [13].

The cross-sectional morphology of the sample-50V0.25A and -noIAD were photographed by FEG-SEM, as shown in Fig. 4(a) and (b), respectively. It was found obviously that the cross-sectional morphology of these two samples was amorphous and no columnar structure, and had the same XRD results. The possible reason might be due to the thermally evaporated deposition process of the AlF_3 material, which has been reported by the previous study [6,14]. Most interestingly, the cross-sectional morphology of the sample-50V0.25A was still amorphous without columnar structure. Using the IAD method is helpful to make the loose structure of the AlF_3 films become dense. Even if an amorphous structure formed by noIAD process might be destroyed, another amorphous structure was formed in the IAD process.

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