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Oxygen Control on Nanocrystal-AlON Films by Reactive Gas-Timing Technique R.F. Magnetron Sputtering and Annealing Effect

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Abstract. The AlON films grown on Si(100) substrates by using radio frequency (r.f.) magnetron sputtering from high purity aluminum (99.999% Al) target with a novel reactive gas-timing technique. The 100 nm thick of AlON films were deposited with 200 watts r.f. power and the substrate temperature is maintained at room temperature by the technique of gas-timing which varying flow-in sequence of high purity of Ar (99.999%) and N_2 (99.9999%) gases fed into the sputtering chamber at 10:90 (sec) ratio. The composition and crystal orientation of AlON films affected by gas-timing of Ar and N_2 were analyzed by Auger Electron Spectroscopy (AES) and X-ray diffraction (XRD). The oxygen atoms revealed by AES formed into a corporation in films was studied. This suggests that the oxygen contamination formed as AlO_XN_Y compound may due to the residual oxygen in base pressure of 10^{-7} mbar and higher reactivity of oxygen in the reactor compared to nitrogen. The gas-timing technique used in the sputtering growth system shows the advantage of the oxygen quantity control, while the general sputtering process (without gas-timing technique) shows an increase of the oxygen composition depended on film thickness. The characterizations results clearly indicate that the gas-timing r.f. magnetron sputtering technique plays an important role to control the incorporation of oxygen and to form the nanocrystal-aluminum oxynitride films which very attractive for various sensors applications.

Introduction

Aluminum Oxynitride (AlON) is a transparent polycrystalline ceramic material of high strength and hardness. AlON films are widely applied as protective coatings against wear, diffusion and corrosion [1-3], optical coatings [4, 5], optoelectronics, and other fields of technology. This is due to the possibility for a broad combination of the physical and chemical properties of the oxynitride films with variable concentrations of aluminum, oxygen, and nitrogen. The film properties can be tailored between those of pure aluminum oxide (Al_2O_3) and aluminum nitride (AlN), depending on different applications. The synthesis of AlON films has been commonly reported using physical vapor deposition (PVD), such as ion-beam-assisted evaporation and sputtering [1–3, 5]. The optical, mechanical and gas barrier properties of the AlON films have also been investigated [1–3]. However, little attention has been paid to the preparation of AlON films by chemical vapor deposition (CVD). Unfortunately, AlON is a refractory material; it requires high substrate temperatures (1300 K), which is complicated to obtain [6]. Then reactive gas-timing r.f. magnetron sputtering technique is utilized to implement AlON films with uniformity at low temperature [7].

In this paper, the oxygen concentration profiles of AlON films grown with and without reactive gas-timing r.f. magnetron sputtering method were reported. The crystalline structure and surface morphology of theses films affected by annealing temperature were also studied.

Experimental

To study on oxygen concentration profiles of AlON films with different sputtering methods, the reactive gas-timing and general reactive r.f. magnetron sputtering methods were used to prepare AlON films on Si(100) substrates. The sputtering target was 99.999% aluminum (Al) material. In the sputtering growth of AlON films, the argon (Ar) gas carry out particular function as ions bombardment, while nitrogen (N_2) gas performs a function of reactive ions. The flow sequence of Ar:N_2 gases is shown in Fig. 1, for the example gas-timing. The solid line denotes the sequence of Ar that bombarded the Al target for 10 sec, and the dashed line denotes the sequence of N_2 that reacted with Al atoms for 90 sec. The flow rate of Ar was fixed at 12 standard cubic centimeters per minute (sccm), while the flow rate of N_2 was 7 sccm. The 100 nm thick of AlON films were deposited at room substrate temperature with r.f. power of 200 W. The composition of AlON films was evaluated by Auger electron spectroscopy (AES) depth profiling.

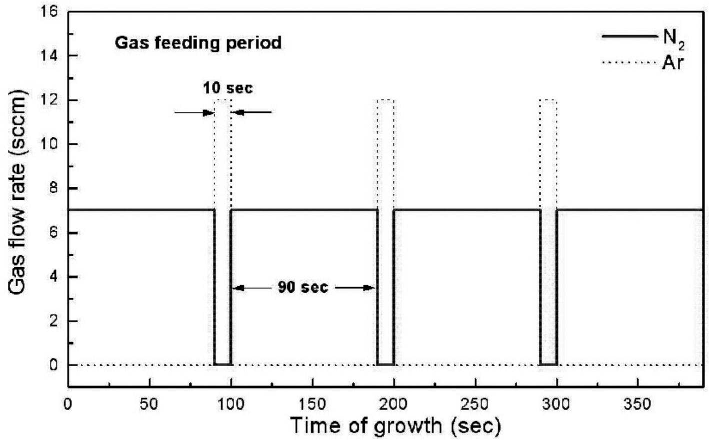


Figure 1. Time sequence of gases flow rates of Ar (12 sccm) at 10 sec and N_2 (7 sccm) at 90 sec.

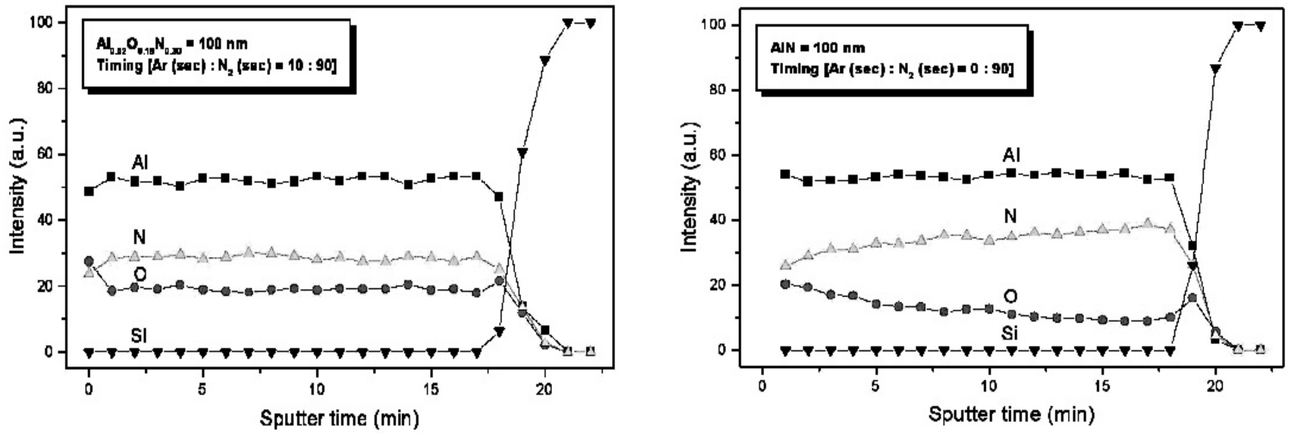
To study on annealing effect, the 100 nm thick of AlON films on Si(100) substrates deposited at room temperature with reactive gas timing r.f magnetron sputtering were used. After deposition process, the films were annealed in N_2 flow-furnace at 200, 350, and 450 °C, respectively.

Results and Discussion

Oxygen Concentration Profiles

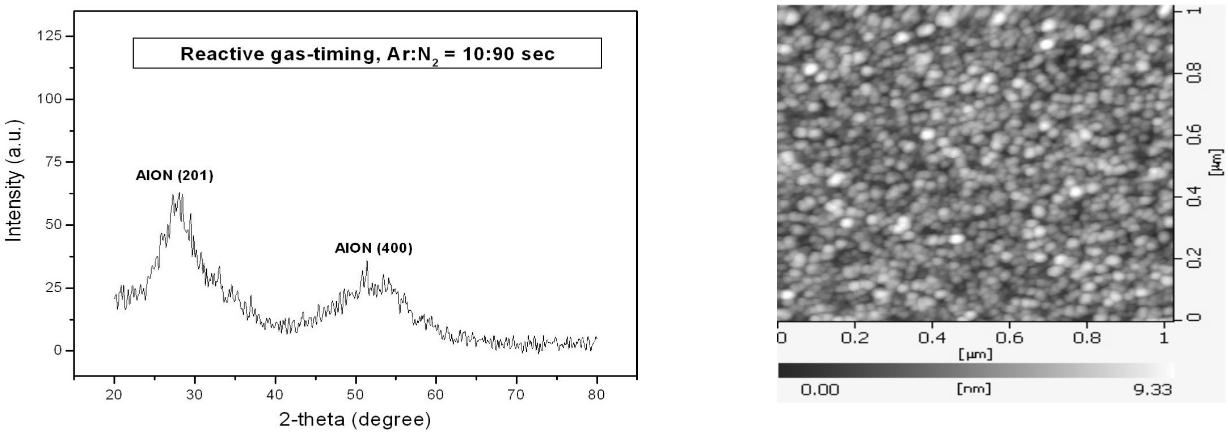
The composition of deposited films was evaluated by AES depth profiling. The oxygen profiles of AlON films grown with and without reactive gas-timing r.f. magnetron sputtering are shown in Fig. 2(a) and (b), respectively. According to AES measurement, the results indicate that the ratio of oxygen composition is more or less constant regarding the film thickness in case of gas-timing process. But in case of sputtering with general sputtering (in pure N_2 gas), the ratio of oxygen composition is increased in proportional to film thickness. The results of our experiment clearly indicate that the reactive gas-timing technique applied for sputtering method plays an important role to the formation of AlON films. It is effectively to control of oxygen in sputtered films.

The crystallinity of AlON thin films was characterized by XRD. Figure 3(a) shows the XRD pattern of deposited film on Si(100) substrate by reactive gas-timing rf magnetron sputtering at room temperature. The result exhibits the crystalline orientation of (201) plane at the 2-theta angle of 27.54˚ and (400) plane at 2-theta angle of 53.58˚ as AlON tetragonal structure. The surface morphology of AlON film obtained from atomic force microscope (AFM) shows the nanocrystal grain size around 20 nm and film uniformity as shown in Fig. 3(b). These results indicate that the reactive gas-timing used in sputtering growth process have an important influence on the crystallinity of AlON films.



(a) (b)

Figure 2. AES depth profiling of AlON films grown (a) with and (b) without reactive gas-timing r.f. magnetron sputtering



(a) (b)

Figure 3. (a) XRD pattern from AlON film deposited on Si (b) The surface morphology obtained from AFM

The exhibited oxygen in deposited film with pure N_2 sputtering may due to the reasons of an unavoidable residual agent (O_2) in the vacuum system at base pressure of about 10^{-7} mbar, the higher reactivity of oxygen in the reactor which compared to nitrogen and the impurity of N_2 gas introduced to the chamber. These suggestions can imply the oxygen contamination in deposited film without feeding of O_2 gas into the vacuum chamber.

Annealing Temperature Effect

Influence of annealing temperature on 100 nm thick of AlON films, was studied by AES measurement. Figure 4 shows atomic concentration of O relates with annealing temperature. The O concentration is high proportion to annealing temperature as shown in Figure 4 (a-d). At room temperature growth of AlON film, the oxygen concentration is about 18% throughout the thickness of the film, while the oxygen concentration of annealed films is increased. These results clearly exhibit that the oxygen can incorporate effectively into the film at high temperature of annealing process. At annealing temperature of 450 °C on AlON films, the oxygen was found at higher than 35%. So we can adjust the oxygen concentration in AlON films by annealing process.

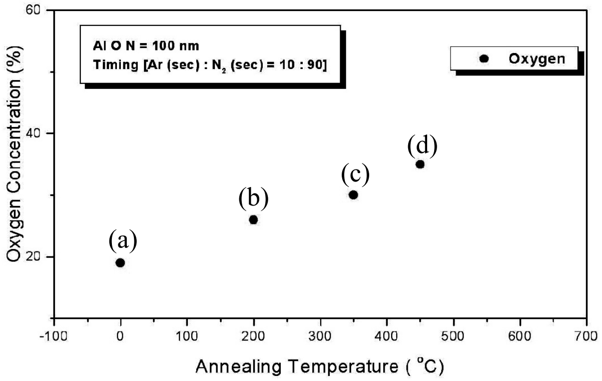


Figure 4. The O atomic concentration of annealed films at (a) RT (b) 200 °C, (c) 350 °C and (d) 450 °C, relates with annealing temperature.

Summary

The AlON thin films were prepared on Si(100) substrates with and without reactive gas-timing r.f. magnetron sputtering at room temperature. The XRD result of room temperature sputtered film exhibits the crystalline orientation of (201) plane at the 2-theta angle of 27.54˚ and (400) plane at 2-theta angle of 53.58˚ as AlON tetragonal structure. The AES revealed the oxygen concentration of annealed AlON thin films increase from 18% to 40% depended on annealing temperature. The 18% of oxygen concentration incorporates in deposited AlON film at room temperature may due to the residual oxygen in vacuum base pressure of 10^{-7} mbar and the higher reactivity of oxygen in the reactor which compared to nitrogen. According to the AES results, the oxygen can effectively incorporate into film at high temperature and the concentration increases when annealing temperature is risen. These results indicate that the annealing temperature plays the key role on the composition of AlON films. The annealing process is very effective to form AlON film with different oxygen concentration. The results indicate that the reactive gas-timing have a great impact on the crystallinity and oxygen concentration of nanocrystal-AlON films.

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