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Spectroscopic Analyses of Sputtered Aluminum Oxide Films with Oxygen Plasma Treatments

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Abstract. X-ray photoelectron spectroscopy (XPS) and Spectroscopic Ellipsometry (SE) were used to analyse the effect of oxygen plasma treatment on properties of aluminum oxide thin films. The aluminum oxide films were fabricated using a reactive sputtering system. The as-deposited films were treated with oxygen plasma powered by an RF generator. During the plasma treatment, the pressures were set at 1 x 10^{-1} to 1x 10^{-2} mbar, while the RF supplied powers at 100 W and 200 W. It was observed that lower plasma powers and higher pressures resulted in smoother films. The O/Al ratio of the films were found to decrease with increasing plasma powers and pressures. The thickness and refractive index of the films were significantly affected by the oxygen plasma treatment process, which could be related to the change in films' packing density and the etching at the surface.

Introduction

Aluminum oxide is an important insulator used in various microelectronic devices [1]. Among many processing methods, the sputtering technique can be used to produce aluminum oxide thin films with the precise control of their purity and composition [2]. The application of post-processing steps, such as annealing or plasma treatment, is often necessary for some applications. These postdeposition treatments are introduced to enhance several properties of the films including their crystallinity [3-4], electrical conductivity [5] and optical properties [5-6]. Unlike the annealing process, the plasma treatment process has gained much interest recently because it can be performed at lower temperatures. Thus, it is compatible with a wide range of substrate materials [7]. However, various conditions in the plasma treatment may also cause surface defects, oxygen vacancies, impurity atoms, etc. These defects are prone to limit the performance of electronic devices. Thus, it is important to study effects of various conditions used during the plasma treatment on the properties of thin films. This work investigates the effect of oxygen plasma treatment on sputtered aluminum thin films. X-ray photoelectron spectroscopy (XPS) and spectroscopic ellipsometry (SE) were employed to study the chemical composition, optical property and thickness of the films. The relations among thin films' stoichiometry, the change of thickness, and the change of refractive index were determined and discussed.

Experimental

Aluminum oxide films were grown on (100) silicon wafers and glass substrates using a pulsed direct current (DC) reactive magnetron sputtering system (AJA International, Inc ATC2000-F). An aluminum target with 99.999% purity (KJ Lesker) was used in this experiment. The based pressure was set at 8×10^{-6} mTorr. Ar and O_2 flow rates used were 15 sccm and 2 sccm, respectively. The operating pressure was set at 10 mTorr. After the deposition, the aluminium oxide films were treated with oxygen plasma for 10 min at room temperature. The pressure and power were set at 1×10^{-1} – 1×10^{-2} mbar, and 100 – 200 W, respectively.

The field-emission scanning electron microscope (Hitachi SU8030 FESEM) and X-ray photoelectron spectroscopy (XPS beamline 3.2b, SLRI) were employed to characterize surface morphology and chemical composition of the oxygen plasma treated samples, respectively. The rotating-analyzer spectroscopic ellipsometer (J.A. Woollam Co. VASE2000) was used to analyze the refractive index and thickness of the films.

Result and Discussion

Fig. 1 shows the surface morphologies of the aluminum oxide thin films after being treated with oxygen plasma at different powers and pressures. The results have shown that the films treated at higher pressure of 1×10^{-1} mbar appear to have smoother surfaces. At lower pressure of 1×10^{-2} mbar, more surface defects can be observed. This is because the oxygen plasma at lower pressure possesses higher energy per atom; thus the bombardments on alumina surface cause more surface defects. Higher plasma powers also cause films to have rougher surfaces.

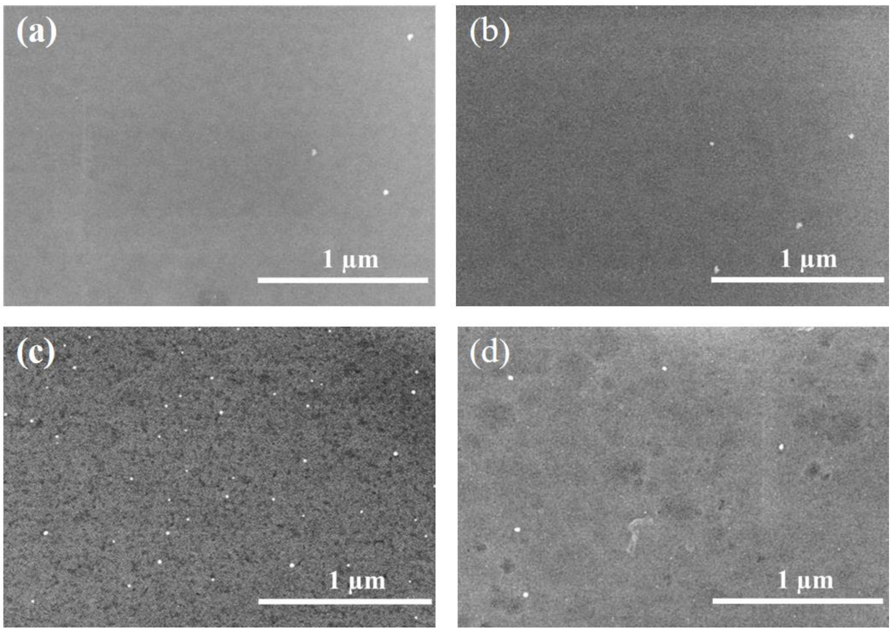

Fig. 1. FE-SEM micrographs of aluminum oxide films treated with (a) plasma power 100 W pressure 1×10^{-1} mbar, (b) plasma power 200 W, pressure 1×10^{-1} mbar (c) plasma power 100 W, pressure 1×10^{-2} mbar and (d) plasma power 200 W, pressure 1×10^{-2} mbar.

Fig. 2 shows the XPS spectra of the oxygen plasma treated aluminum oxide thin films. The overview of the spectra (fig. 2a) was obtained from the sample treated with the oxygen plasma at the pressure of 1×10^{-1} mbar and the power of 100 W. The significant peaks included Al2p, Al2s, C1s and O1s peaks. The O1s peak can be deconvoluted into two components as shown in fig. 2b. The first component at the binding energy of 531 eV corresponded to O^{2-} ions associated with lattice oxygen atoms. The peak near 532.5 eV could be associated with O^{-} ions from adsorbed oxygen species at the surface of the aluminum oxide [8]. Fig. 2c shows the XPS spectrum of Al 2p orbital. The peak at 74.8 eV could be associated with the Al-O bonding.

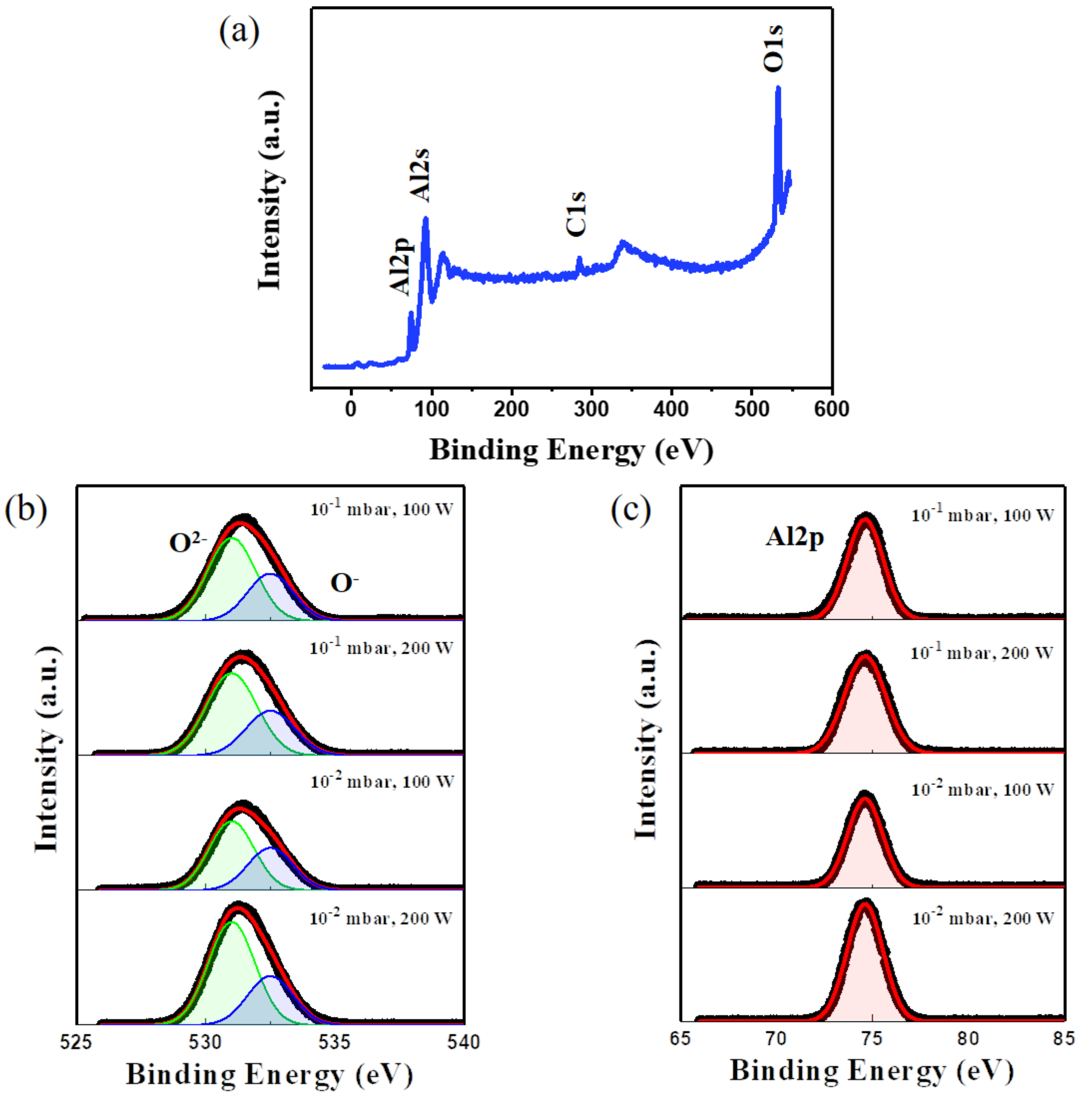

Fig. 2. X-ray photoelectron spectra of aluminum oxide films treated with oxygen plasma (a) the survey interval, (b) high-resolution O1s region and (c) high-resolution Al2p region.

Table 1 shows the O/Al ratio of thin films treated with different RF powers and pressures. The atomic ratio was calculated from areas of deconvoluted peaks in the XPS spectra. The as-deposited film was found to have O/Al ratio of 1.63, which is considered an oxygen-rich film, as compared to the stoichiometric value of Al_2O_3. The treatment using the plasma power of 100 W and the pressure of $1\text{x}10^{-2}$ mbar yielded the film with O/Al ratio closest to the Al_2O_3 stoichiometric condition of 1.5. Higher plasma powers and pressures caused the films to have lower O/Al ratios, and further deviated from the stoichiometric condition.

Table 1. The O/Al ratios of the aluminum oxide films treated with different plasma pressures and powers

Pressure (mbar)	Power (W)	O/Al
As-deposited		1.63
$1\text{x}10^{-1}$	100	1.33
$1\text{x}10^{-1}$	200	1.27
$1\text{x}10^{-2}$	100	1.50
$1\text{x}10^{-2}$	200	1.44

The influence of plasma power and pressure on the thickness of aluminium oxide thin films is shown in table 2. The thickness was indirectly determined through the spectroscopic ellipsometry (SE) technique. It was observed that the thickness of the films significantly decreased after the oxygen plasma treatment. This could be due to the following reasons. First, the plasma treatment process altered the packing density of the films. Second, higher energy plasma also etched surface atoms, which was more pronounced at lower pressure of 10^{-2} mbar and higher power of 200 W (higher energy/atom).

Table 2. The thicknesses of the aluminum oxide films treated with different plasma pressures and powers

Pressure (mbar)	RF Power (W)	Thickness (nm)
As-deposited		548
$1x10^{-1}$	100	496
$1x10^{-1}$	200	494
$1x10^{-2}$	100	490
$1x10^{-2}$	200	462

The refractive index of oxygen plasma treated aluminium oxide thin films measured by SE is demonstrated in fig 3. The results have shown that the oxygen plasma treatment affects optical properties of the films. The change in optical properties after the plasma treatment is in agreement with several works found in literature [9-10]. In our experiment, the refractive index was observed to decrease in all samples. The change in the refractive index may be associated with the change in the packing density of the films as well as surface roughness caused by energetic particle bombardments on the films' surfaces.

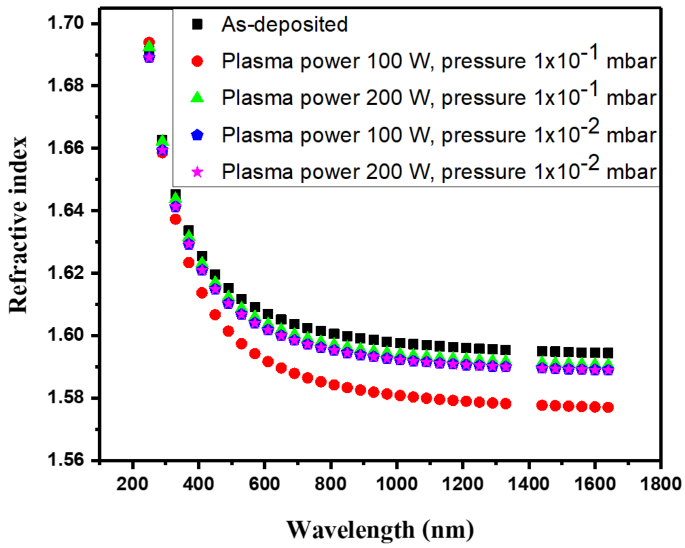

Fig. 3. The influence of plasma powers and pressures on the refractive index of aluminium oxide thin films.

Summary

This work investigated the effects of oxygen plasma treatment on the surface morphology, chemical composition and refractive index of sputtered aluminium oxide thin films. The results demonstrated that the oxygen plasma treatment strongly influenced surface and optical properties of the films. Lower plasma pressures and higher powers caused films to have rougher surfaces. The results also suggested that the oxygen plasma altered the films' composition from oxygen-rich to aluminium-rich. The lowest O/Al ratio of 1.27 was found in the sample treated with the highest plasma power of 200 W and pressure of $1x10^{-2}$ mbar. The decrease in films' thicknesses was quite significant after the oxygen plasma treatment. Similarly, the oxygen plasma treatment also affected the refractive index of the films. These could be related to the change in films' packing density and the etching on the surface of the films.

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