The characterization of aluminum nitride thin films prepared by dual ion beam sputtering

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Abstract
Aluminum nitride (AlN) films were prepared between 800 and 1200 V at 450 °C and room temperature. The polycrystalline films were deposited but textured along the (100) and (002) plane. The AlN (002) gradually decreased as the argon ion beam voltages increased at 450 °C. The AlN (002) decreased at 1000 V and then was sustained at 1200 V at room temperature. The AlN film deposited at 450 °C was thinner than that at room temperature at a given voltage, which might account for the higher mobility of the adatoms. The surface roughness decreased monotonously as the argon ion beam voltages and deposition temperature increased. The β-values steadily decreased as the argon ion beam voltages increased at room temperature, but significantly drop at 450 °C. All β-values revealed that the growth is stabilized and a texture is formed. The binding energy of the O-1s spectra was around at 532.0 eV which is attributed to the adsorbed water. The relationship between both the argon ion beam voltages and the deposition temperatures and the microstructure of AlN films is also discussed.

Keywords: Aluminum nitride; Surface roughness; X-ray diffraction; X-ray photoelectron spectroscopy

1. Introduction
Aluminum nitride (AlN) has many excellent physical and chemical properties, such as high thermal conductivity [1,2], high hardness, good corrosion resistance [3], outstanding insulation properties [1], optical transparency in the visible and near-infrared ranges [2,3], and high longitude sound velocity [1]. The main applications of the films include heat sinks [4], thin-film resistors [5], hard coatings [6], and high-frequency surface acoustic wave devices [1]. The dual ion beam sputtering (DIBS) system concurrently uses two ion sources—the Kaufman-type and/or the end-Hall ion source. Although the variations of nitrogen beam energy on the microstructure of AlN films in DIBS system have been studied [7–10], the fact that the argon ion beam voltages influence the microstructure of the films has received little investigation. Hence, this study employs the DIBS system to deposit AlN films at various argon ion beam voltages and deposition temperatures. The microstructure and growth model of the films are also investigated.

2. Experimental details
The AlN films were deposited on p-type (100) Si wafers (Toshiba Ceramics Co. Ltd.) using a DIBS apparatus, which was described elsewhere [11]. An accelerating voltage from 800 to 1200 V was applied in the Kaufman ion source and the end-Hall ion source was simultaneously operated at 130 V as the reactive nitrogen/neutral nitrogen was 15–25%. The flow rate of the nitrogen gas (99.995% purity) was three times greater than that of the argon gas (99.995% purity). The deposition temperatures were room temperature and 450 °C, with a deposition time of 60 min. A MacScience MXP 3 diffractometer with Cu Kα radiation was utilized to examine the crystal structure. Furthermore,
the texture coefficient of AlN (002) orientation was evaluated from the spectra using $I_{\text{AlN (002)}} / (I_{\text{AlN (002)}} + I_{\text{AlN (100)}})$, where $I$ corresponds to the integrated intensity of the peak. The thickness of the films was measured by Hitachi S-4100 scanning electron microscopy. The root-mean-square surface roughness was measured using an atomic force microscope model Nanoscope IIIa with a D3100 scanner (Digital Instrument Co. Ltd.) in air at room temperature. The scanned area was 1 μm². The surface roughness ($w$) generally varied with the growth time ($t$) according to a power law, such that $w \propto t^b$, where $b$ is the growth exponent and describes the surface roughening process, which indicates the growth mode of the film [12]. The chemical state of O-1s was determined using an X-ray photoelectron spectroscopy (XPS) model Physical Electronics ESCA PHI 1600, which was described elsewhere [11].

3. Results and discussion

3.1. Crystallographic analysis

The orientation of AlN films is critical factor when the films are used as a surface acoustic device. Therefore, the effect of deposition parameters on the microstructure of a film must be studied. Fig. 1(a) displays XRD spectra of AlN films for various argon ion beam voltages and deposition temperatures. The films were polycrystalline but textured, and the various patterns were dominated by (100) and (002) peaks [13]. Additionally, all peaks were broadening, possibly suggesting nano-crystallinite or non-uniform strains/stresses. Fig. 1(b) represents the AlN (002) as a function of film thickness, argon ion beam voltage and deposition temperature. The AlN (002) gradually decreased as the argon ion beam voltages and the films thickness increased at 450°C. Nevertheless, the AlN (002) dropped at 1000 V and then remained at 1200 V at room temperature. The orientation may be related to the high residual stress caused by the bombardment of the films by high energetic particles at high ion beam voltages [9,10], which may result in the growth of other crystallographic planes. Furthermore, the AlN films deposited at 450°C are slightly thinner than those deposited at room temperature at the same argon ion beam voltage. That fact clearly indicated that adatoms have a high surface mobility at higher deposition temperature, resulting in the growth of thinner films.

3.2. Surface morphology analysis

Fig. 2(a) plots the surface roughness, measured using AFM, as a function of the thickness of the films and argon ion beam voltage at various deposition temperatures. The figure revealed that surface roughness decreased monotonously as the thickness of the film and argon ion beam voltages increased, which in fact may be related to the formation of the films. The formation of the films follows nucleation, yielding rough surfaces. Additionally, the surfaces of the thick films were smooth perhaps because of coarsening. The variation in surface roughness may also be related to the effect of argon ion beam bombardment of the aluminum target. The aluminum atoms have relatively low kinetic energy at low argon ion beam voltages, so the atoms that arrive on the surface as adatoms have a low capacity to be rearranged. Therefore, a rough surface is formed. Moreover, the aluminum atoms have relatively high kinetic energy at high argon ion beam voltages, so adatoms have a high capacity to be rearranged on the surface. Therefore, a smooth surface is formed.

As mentioned above, $b$ can be used to determine the growth feature of the films; a value of unity revealed a random growth mode; otherwise, the growth is stable [12]. Fig. 2(b) plots the growth exponent ($b$) values against argon ion beam voltage and deposition temperature. Although $b$-values decreased slightly as the argon ion beam voltages
increase at room temperature, significant changes occurred at 450 °C. All β-values were smaller than that reported for sputtered AlN films (β = 0.37 [12]). The growth of AlN films stabilized and yielded textured growth according to the above XRD spectra.

3.3. Chemical composition analysis

Although Al-2p3/2 and N-1s spectra [11] have been well characterized as indicating Al–N bonds (Al-2p3/2 = 74.2 eV and N-1s = 397.4 eV) in all cases, O-1s spectra are merely discussed before. Fig. 3 presents O-1s spectra of AlN films deposited between 800 and 1200 V at (a) room temperature and (b) 450 °C. The dashed lines represent the experimental data and the solid lines represent the fitting results using a nonlinear least squares fit with a Gaussian/Lorentzian peak shape (G/L mixing ratio = 0.3) after the Shirley background subtraction routine was applied. The weak oxygen spectra are centered at ~532.0 eV, which was attributed to the adsorbed water [14,15]. The adsorbed water may originate in the chamber or may have been present as impurities in the reactive gas. The XPS results demonstrated the high-quality AlN films were deposited herein.

4. Conclusions

Aluminum nitride films were synthesized at various argon ion beam voltages (800–1200 V) and at deposition temperatures of room temperature and 450 °C in a DIBS system. The polycrystalline AlN films grow, with textured growth, in (100) and (002) orientations. The AlN (002) steadily decreased as the argon ion beam voltage increased at 450 °C, but AlN (002) dropped at 1000 V and then remained at 1200 V at room temperature. The surface roughness decreased monotonously as the argon ion beam voltages and the deposition temperature increased. Moreover, the AlN film deposited at 450 °C was thinner than that deposited at room temperature. The growth exponent (β)
was deduced from the surface roughness and the value dropped as either argon ion beam voltages or the deposition temperature increased. In this work, the stable and textured growth was indicated by $\beta$-values. The binding energy of the O-1s spectra was around at 532.0 eV which was attributed to the adsorbed water.

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