

XRD Characterization of AlN Thin Films Prepared by Reactive RF-Sputter Deposition

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ABSTRACT

AlN thin films have been grown on R((1-12) surface-cut)- Al_2O_3 , SiO_2 -glass and C((001) surface-cut)- Al_2O_3 substrates, by using a reactive-RF-sputter-deposition method. X-ray diffraction (XRD) shows that AlN film has (110) orientation of wurtzite crystal structure for R- Al_2O_3 and (001) orientation for SiO_2-glass and C- Al_2O_3 substrates. The film thickness was analyzed by Rutherford backscattering spectroscopy (RBS) and it appears that XRD intensity does not show a linear increase with the film thickness but a correlation with the stress, *i.e.*, deviation of the lattice parameter of the film from that of bulk. The film composition and impurities have been analyzed by ion beam techniques. Effects of highenergy ion beams are briefly presented on atomic structure (whether stress relaxation occurs or not), surface morphology and optical properties.

Keywords: Aluminum Nitride Film; Composition; Impurities; Atomic Structure; Surface Morphology; Optical Properties

1. Introduction

It has been known that aluminum nitride (AlN) has a wide direct-bandgap (6.2 - 5.8 eV) [1,2] with hexagonalwurtzite crystal structure [3] and unique properties: good thermal conductivity (~3 W/cmK at 300 K) [4]. good insulator (>10¹¹ Ω ·cm) [5], high dielectric constant [6], relatively small linear-expansion coefficients (5.3 and 4.2 \times 10⁻⁶ K⁻¹ along a- and c-axis) [7], high sound velocity (6 km/s) [8] and large hardness [9]. Owing to these properties, AlN films have potential applications to electronic devices [10], surface acoustic wave (SAW) devices [11], actuator [12], transparent hard coatings and AlN composites to light-emitting devices [13]. Also, AlN films have been used as buffer layer for GaN [14] and ZnO [15] film growth. For these applications, X-ray diffraction (XRD) technique have been extensively employed to evaluate the crystalline quality and growth orientation of AlN films which have been grown by various techniques, chemical-vapor atomic-layer deposition (a special type of CVD) [2], metal organic CVD [16], molecular beam epitaxy [17], ion beam enhanced deposition (electron beam evaporation of Al combined with N ion bombardment) [5], reactive radio-frequency (RF) magnetron sputtering

deposition [6,10,18], pulsed laser deposition (PLD) [19] on various substrates, sapphire [2,19], Si [5,14-16,18], SiC [17], Al [6], Mo [12] etc. For AlN films grown on Si(111), the authors have shown that oxygen impurities near the substrate surface affect the growth orientation and suggest that the XRD intensity decreases with increasing the stress and nearly diminishes when the stress exceeds 2%, irrespective of the film thickness (27 - 470 nm) [20]. Here, the stress is defined as the difference of the lattice parameters between film and bulk. Use of the stress can be justified based on the fact that c-axis length increases with the residual-stress [18] and temperature dependence of the lattice parameter is similar to that of the residual-stress in terms of pressure [19]. The result does not agree with the lattice relaxation around 50 nm of AlN on SiC [17] and favors the constant stress throughout the AlN film on Si(111) [16]. It is of interest to study whether the suggested stress is useful for the quality evaluation of AlN film grown on different substrates other than Si(111).

In this paper, we have grown AlN on R-plane cut sapphire (R-Al₂O₃), SiO₂-glass and C-plane cut sapphire (C-Al₂O₃) substrates by a reactive RF-sputter deposition method. We have measured XRD, the composition, thickness and impurities, and examined use of the stress for the film quality evaluation. We also have measured surface morphology (grain size, shape and surface smoothness), which may affect the crystalline quality, since films are polycrystalline, and optical absorption. These properties might be important for applications mentioned above. For AlN on R-Al₂O₃, irradiation with high-energy (90 MeV Ni) ions was performed in order to study whether stress relaxation, surface smoothing and bandgap modification occur or not by ion irradiation.

2. Experimental

AlN films were grown on R-Al₂O₃, SiO₂-glass and C-Al₂O₃ substrates by using a reactive-RF-sputter-deposition method with Al target (purity of 99.999%) in pure N_2 gas of ~0.3 Pa with a method described in [20,21]. A reason for usage of pure N₂ gas is to avoid Ar inclusion into films, considering that conventionally Ar and N₂ mixture gas has been employed. The substrates were subjected to ultrasonic rinse in ethanol prior to the film deposition. XRD with Cu-k α radiation was performed to examine crystalline quality and orientation. The thickness, composition and impurities of films were analyzed by RBS. The growth rate was obtained to be approximately 3 nm/min for AlN on three substrates used in this study. Light impurities such as carbon and oxygen near the film surface were analyzed by using nuclear reaction analysis (NRA), ${}^{12}C(d, p){}^{13}C$ and ${}^{16}O(d, \alpha){}^{14}N$ with 1.2 MeV d at the reaction angle of 160° [20]. In RBS and NRA, stopping powers are taken after [22] with the AlN density of 3.26 g·cm⁻³ (4.8×10^{22} Al cm⁻³). Surface morphology was observed by atomic force microscopy (AFM) and optical absorption was measured by using a conventional spectrometer. Irradiation with 90 MeV Ni ions was performed by using a TANDEM accelerator at Japan Atomic Energy Agency at Tokai.

3. Results and Discussion

3.1. Characterization

Figure 1 shows XRD patterns and rocking curves of AlN film on R-Al₂O₃, SiO₂ and C-Al₂O₃ substrate. The substrate temperature T_s was optimized, 150°C, 200°C and 200°C for these substrates, respectively so that the XRD peak intensity is maximized and the full-width at halfmaximum (FWHM) of XRD rocking curve is minimized. It is found that AlN film has exceptionally a-axis, *i.e.*, (110) orientation on R-Al₂O₃ (diffraction angle $2\theta \approx 59^\circ$), in contrast to c-axis ($2\theta \approx 36^\circ$), *i.e.*, (001) orientation grown on other substrates, Si, SiO₂, C-Al₂O₃ etc. FWHM of the rocking curve of as-deposited film on R-Al₂O₃ is order of 2° (**Figure 1(a)** and **Table 1**). AlN on SiO₂ glass-substrates has (001) orientation and FWHM is much larger (~10°) (**Figure 1(b)** and **Table 2**). FWHM



Figure 1. XRD patterns of as-deposited AlN film on R-Al₂O₃ (a), SiO₂ (b) and C-Al₂O₃ (c) substrates. Rocking curves of as-deposited films are shown in the inset and FWHM is indicated by horizontal lines. Deposition time was 55, 30 and 30 min for AlN on R-Al₂O₃, SiO₂ (sample 70c in Table 2) and C-Al₂O₃. Peaks at $2\theta \approx 59^{\circ}$ and 36° are (110) and (002) diffraction of AlN, and 52.5° and 41.7° R- and C-plane of sapphire.

of the rocking curve of as-deposited film on C-Al₂O₃ is order of 0.5° (**Figure 1(c)**). Hence, the crystalline quality of AlN on SiO₂-glass is poorer than that on R-Al₂O₃ and is the best for AlN on C-Al₂O₃. For AlN on C-Al₂O₃ used in this study, deposition time was 10 to 65 min or the film thickness ~30 to 200 nm.

A typical RBS of AlN on R-Al₂O₃ is shown in **Figure 2**. Similar RBS spectra were obtained for AlN on SiO₂ and C-Al₂O₃. The film thickness was deduced from the N-width illustrated in **Figure 2**. The thickness derived from RBS and XRD results are summarized in **Tables 1** and **2** for AlN on R-Al₂O₃ and SiO₂. Here, accuracies of

Sample	Deposition Time (min)	L (nm)	Relative Intensity	FWHM (deg.)	a-axis length (nm)
Z1d	15	42.7	0.85	2.68	0.3149
78b	30	95	0.002		
71a	30	115	3.2	2.22	0.3157
C3d	45	148	4.4	2.14	0.3147
95c	55	171	1.0	2.81	0.3189
X6a	67	200	2.5	2.34	0.3177
98a	55	208	8.5	2.0	0.3149
X8h	100	271	5.2	2.33	0.3170
C0f	120	368	0.79	3.7	0.3214
Y6c	160	449	10.6	2.18	0.3124

Table 1. A summary of RBS (thickness, L) and XRD (intensity, FWHM and a-axis length) characterization of as-deposited AlN films on R-Al₂O₃.

Table 2. A summary of RBS (thickness, L) and XRD (intensity, FWHM and c-axis length) characterization of as-deposited AlN films on SiO₂.

Sample	Deposition Time (min)	L (nm)	Relative Intensity	FWHM (deg.)	c-axis length (nm)
Z0c	15	37.7	0.01		
70c	30	108	2.7	6.0	0.50764
Z6e	45	148	0.54	10	0.50887
96a	55	167	0.32	16	0.51039
85h	60	184	0.08		0.50699
85c	60	190	0.07		0.50754
Y1d	65	199	1.8	8.1	0.50685
89f	56	213	0.97	9.3	0.50936
Z5e	80	233	0.016		
Y5a	100	325	2.5	8.8	0.50866



Figure 2. RBS of AIN on R-Al₂O₃ (sample 98a in Table 1). The spectra were obtained using 1.8 MeV He, and incident and outgoing angle are 30° and 50° measured from surface normal. Energies of He scattered from Al and N located at surface and interface are indicated by vertical lines. Ar and Fe impurities are also indicated.

thickness, XRD intensity, FWHM and axis length are estimated to be 10%, 20%, 3% and 0.3%, respectively. The composition appears to be nearly stoichiometric

(N:Al = 1:1), within the RBS accuracy of 10%. One sees some impurities in the RBS spectra and tentatively identified as Fe (main component of stainless steel) and Ar, considering that stainless steel is the main material of RF-sputter deposition chamber and Ar gas has been often employed. We find that for 16 AlN films on R-Al₂O₃ including those given in Table 1, Fe and Ar impurity concentration relative to Al concentration ranges from 0.04% - 0.1% and 0.045% - 0.14%, respectively. Slightly larger amounts (Fe: 0.05% - 0.17%, and Ar: 0.4% -0.17%) were observed for AlN on SiO₂. Similar amounts of Ar and Fe impurities were detected for AlN on C-Al₂O₃. No noticeable relation is found between the Fe impurity concentration and the XRD intensity, and between the Ar impurity concentration and the XRD intensity. NRA was performed for the films on R-Al₂O₃ given in Table 1 and shows that the areal density of C (no information of depth profile is available because of poor depth resolution of the NRA) on/in AlN films ranges from 5 to 18×10^{15} cm⁻², larger than $\sim 3 \times 10^{15}$ cm⁻² for virgin R-Al₂O₃ substrate. NRA also shows that the areal density of O near the film surface ranges from 8 - 16 \times 10^{15} cm⁻². Similar amounts of C and O were detected for

AlN on SiO₂ (C: 7 to 15×10^{15} cm⁻², O near the film surface: 6 to 17×10^{15} cm⁻²). Again no clear relation is observed between the XRD intensity and C impurity density, and between the XRD intensity and O impurity density near the film surface. The former result leads to a speculation that the majority of C impurities are located near the surface. For AlN on C-Al₂O₃, similar amounts of impurities are assumed.

One sees that the XRD intensity does not follow a linear increase with the film thickness for AlN on R-Al₂O₃ and SiO_2 (Tables 1 and 2) and the similar situations is observed for AlN on C-Al₂O₃. As suggested in [20], XRD intensity vs stress is shown in Figure 3. Here, the stress is defined by the axis length of the film divided by the bulk value (0.31111 and 0.49788 nm for a- and c-axis [23]) minus unity, assuming that the stress defined above represents the residual-stress as mentioned earlier. Usually, several samples were prepared in the same run. For a particular run for AlN on R-Al₂O₃, having the film thickness around 380 nm, the results of four samples are shown. Accidentally, the sample (C0f) given in Table 1 is the poorest in the crystalline quality (XRD intensity is the lowest). As shown in Figure 3, it is found that the XRD intensity decreases with increasing the stress, except for the thinnest films, regardless of the substrates, as observed for AlN with c-axis orientation on Si(111) substrate [20]. These results indicate no relaxation of the stress regardless of the film thickness and substrates, implying that the stress is an import factor determining the crystalline quality, and that a simple explanation by misfit (lattice parameter mismatch between the film and substrate) is not applicable to the present results. Introduction of the stress might be affected by O, C and possibly H impurities in the film as well as substrate surface condition, and these are to be investigated.



Figure 3. XRD intensity vs stress for AlN films on R-Al₂O₃ (O, Δ , +), SiO₂ (x) and C-Al₂O₃ (\Box , •). Data (Δ) are taken for the films prepared in the same run having the thickness of ~380 nm. Datum indicated by + is the result of the thinnest film (sample of Z1d in Table 1). For AlN on C-Al₂O₃, film thickness is less than 50 nm (•) and 90 - 200 nm (\Box).

3.2. Surface Morphology, Optical Absorption and High-Energy Ion Irradiation Effects

Surface morphology is mainly studied for AlN on R-Al₂O₃ and SiO₂. An AFM image of as-deposited AlN film on R-Al₂O₃ is shown in Figure 4(a). One sees that a column with c-axis orientation lays down parallel to the surface. The grain is often non-spherical, columnar and thus the grain size is less well-defined. It appears that the smaller size of columnar grains ranges from 20 to 40 nm and their length extends to over 250 nm, as shown in Figure 5(a) and surface smoothness (or roughness) in terms of root mean square (RMS) of the surface height ranges from 0.2 - 3 nm as shown in Figure 5(b). It appears that surface roughness increases linearly with the film thickness for AlN on R-Al₂O₃. Figure 4(b) shows an AFM image of AlN film on R-Al₂O₃ after irradiation of 90 MeV Ni ions at 1×10^{13} cm⁻², and ion irradiation effects will be described later. Figures 4(c) and (d) show AFM images of AlN on SiO₂ and C-Al₂O₃ and cross section of grains on these substrates is nearly circular. The grain size of AlN on SiO₂ is 10 - 40 nm (Figure 5(a)) and RMS is 0.8 - 2 nm (Figure 5(b)). Grain size is ~20 nm for AlN on C-Al₂O₃ shown in Figure 4(d), where RMS is 0.36 nm. In this study, surface smoothness is the best for AlN on C-Al₂O₃ and poorest on SiO₂. For 90 MeV Ni ion irradiation on AlN on R-Al₂O₃ at 10¹³ cm⁻², change was not observed in RBS (see Figure 2) and AFM image remained nearly the same as before irradiation, but surface roughness (RMS) slightly decreases (from 0.75 nm to 0.55 nm).

For AlN on R-Al₂O₃ under irradiation with 90 MeV Ni ions up to 6×10^{13} cm⁻², which appears to cause significant inelastic-collision-effects [25], no change in the axis-length was observed within 0.1%, *i.e.*, no stress relaxation by ion irradiation and the XRD intensity at the fluence of ~ 6×10^{13} cm⁻² decreases to half of that of as-deposited film. No appreciable but slight (several %) reduction in FWHM of the XRD rocking curve was observed at ~around 10¹³ cm⁻².

Optical absorption spectra of AlN on $R-Al_2O_3$ are shown in **Figure 6**. The bandgap as of deposited film is obtained to be 5.7 eV in reasonable agreement with the reported value of ~6.0 eV [1,2]. Optical absorption has little changed, except for the wavelength below 300 nm (**Figure 6**) and the bandgap decreases by ~0.2 eV. Similarly, the bandgap of 5.7 eV is obtained for as-deposited AlN film on SiO₂.

4. Summary

We have presented characterization of AlN films on R-Al₂O₃, SiO₂-glass and C-Al₂O₃ substrates by means of XRD, ion beam technique, AFM and optical absorption. Good quality of AlN films with exceptional orientation



Figure 4. (a) AFM image of as-deposited AlN film on R-Al₂O₃. Deposition time was 55 min. Surface roughness (RMS) is 0.75 nm; (b) AFM image of AlN film on R-Al₂O₃ (sample shown in Figure 4(a)) after irradiation with 90 MeV Ni ions at 1×10^{13} cm⁻²; (c) AFM image of as-deposited AlN film on SiO₂ (sample: 85h in Table 2). Deposition time was 60 min. Surface roughness (RMS) is 1.7 nm; (d) AFM image of as-deposited AlN film on C-Al₂O₃. Deposition time was 60 min. Surface roughness (RMS) is 0.36 nm.



Figure 5. (a) Grain size (nm) vs film thickness (nm) for AlN on R-Al₂O₃ (O, \blacklozenge) and SiO₂ (x) and (b) surface roughness in terms of root mean square (RMS) vs film thickness for AlN on R-Al₂O₃ (O) and SiO₂ (x).



Figure 6. Optical absorption spectra before and after irradiation with 90 MeV Ni ions at 1×10^{13} cm⁻². Inset shows the square of absorbance times photon energy vs photon energy, illustrating bandgap determination.

of a-axis orientation has been obtained on R-Al₂O₃ substrate and a correlation is found between the stress and crystalline quality in terms of XRD intensity. Effects of irradiation with 90 MeV Ni ions have been briefly described.

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