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Comparison of AlN films grown by RF magnetron sputtering and ion-assisted molecular beam epitaxy

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ABSTRACT

Crystalline aluminum nitride (AlN) thin films were formed on various substrates by using RF magnetron sputtering of an Al target in a nitrogen plasma and also by ion-assisted molecular beam epitaxy (IAMBE). Basal-oriented AlN/(111) Si showed a degradation of crystallinity with increased substrate temperature from 550 to 770 °C, while the crystallinity of AlN/(0001) Al_2O_3 samples improved from 700 to 850 °C. The optical absorption characteristics of the AlN/(0001) Al_2O_3 films as grown by both deposition methods revealed a decrease in sub-bandgap absorption with increased substrate temperature.

INTRODUCTION

Aluminum nitride (AlN) is a promising wide bandgap semiconductor material (~6.2 eV) for UV light emitters and detectors[1]. It has also been investigated as an insulated-gate material for III-V field effect transistors [2,3], and as a suitable material for surface acoustic wave devices [4]. Furthermore, the large thermal conductivity (2 W/cm–K) of AlN makes it a good packaging material for integrated circuits [5].

Various methods of fabricating AlN have been reported. They include ammonalysis [1], MOCVD [6], DC magnetron sputtering [7], RF magnetron sputtering [8], reactive MBE [9] and ion-assisted MBE (IAMBE) [10,11]. In this work, we report the results of AlN thin films prepared using RF magnetron sputtering and IAMBE. We will compare the crystalline quality and optical absorption characteristics of these AlN films.

RF magnetron sputtering was selected because it can provide high plasma ion density as well as high deposition rates. The IAMBE system was employed because it offered several advantages over sputtering: the separate control of both Al and N_2^+ fluxes and the absence of Ar gas contamination.

EXPERIMENT

Synthesis Methods

A) RF Magnetron Sputtering System

A RF magnetron gun mounted with a 2" Al target (99.999%purity) was used to prepare AlN thin films. To remove contamination from the Al target surface, the target was pre-sputtered at a RF power of 200 W with Ar at a chamber pressure of 40 mTorr for 60 minutes prior to each deposition. A high ratio of N_2:Ar gas was selected to provide active nitrogen to react with the sputtered Al atoms to enhance the film crystallinity [12]. The sputtered samples were prepared at a N_2:Ar flow rate of 140 sccm:20 sccm, at a chamber pressure of 10 mT and RF power of 200 W. Silver paste was applied between the heater substrate and sample to provide good thermal contact. The deposition rates varied between 80-120 Å/min. (from 110 W to 200 W at 10 mT). The target-substrate spacing was 4 cm.

B) Ion-assisted molecular beam epitaxy (IAMBE)

A WA-Tech Knudsen cell was heated to evaporate Al, which reacts with nitrogen ions bombarding the substrate surface to form AlN thin films. A resistive heater was used to heat the substrates. The nitrogen ions were supplied by a Kaufmann ion gun. A 2 cc PBN crucible loaded with 0.5 g of 6N pure Al charge was inserted inside the Ta heater foils of the Knudsen cell. The deposition rate of AlN thin film was 0.2 Å/sec., which was considerably smaller than the RF magnetron sputtering rate. The activated nitrogen, composed of both ions and neutrals were created with an ion beam voltage of 35 V at a plasma discharge voltage of 30 V. The N_2^+ flux is 2 times greater than the Al flux. The sapphire substrates were heated to temperatures between 700 to 800 °C. Platinum foil was inserted between the sapphire substrates and the heater substrate to provide good thermal contact. Prior to the start of deposition, the Al_2O_3 substrates were immersed in the N_2^+ beam for 5 minutes. Chamber pressure was at the 10^{-4} torr range with a N_2 flow of 3.5 sccm during growth. The ion-gun to substrate spacing and the Knudsen cell to substrate distances were 5 cm and 15 cm, respectively.

Substrates

A variety of substrates were employed, such as (111) Si, $(1\bar{1}02)$ Al_2O_3 and (0001) Al_2O_3. Sapphire substrates (basal orientation, polished on both sides) were degreased using hot acetone followed by hot methanol rinse. Silicon substrates (111 orientation) were first etched in piranha ($5H_2SO_4$:$1H_2O_2$) for 15 min., followed by a BHF dip to remove any residue surface oxide, and DI water rinse.

Thin Film Analysis

The crystallinity of the AlN thin films was determined using a Siemens AT-5000 X-ray diffractometer (Cu K_α, λ=0.154 nm). The optical absorption spectra of the AlN thin films sputtered on α-Al_2O_3 (basal sapphire) and $(1\bar{1}02)$ Al_2O_3 were obtained using a Perkin-Elmer Lambda-9 spectrometer. The transmissivity of the AlN films was measured, from which the absorption coefficient of the film was calculated. Film thickness was measured using both Rutherford backscattering analysis and a Dektak profilometer.

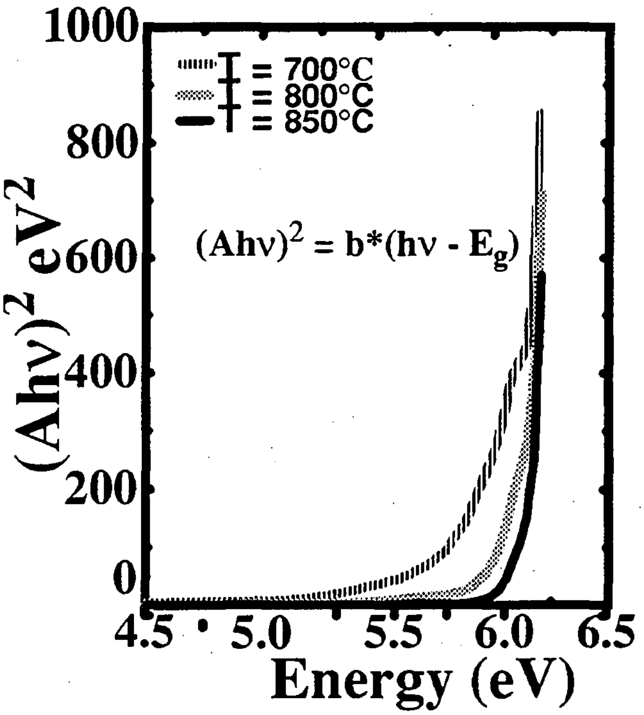

Figure 3: Optical Absorption Spectrum of sputtered AlN/(0001) Al_2O_3.

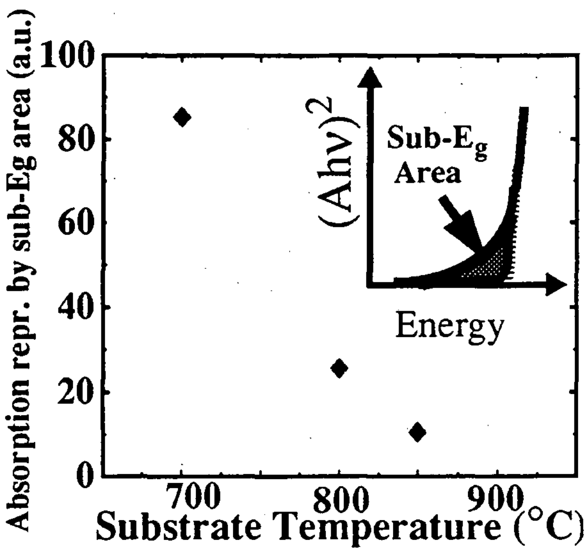

Figure 4: Integration of absorption within the sub-bandgap region vs. substrate temperature of sputtered AlN.

Analogous to the XRD data of the sputtered AlN samples, for IAMBE AlN, the X-ray peak increased with increasing substrate temperature. Furthermore, the FWHM decreased with temperature. Figure 5 shows the FWHM versus growth temperature. The optical absorption behavior of the MBE AlN also followed that of the sputtered films. Figure 6 shows that the higher the substrate temperature, the smaller the absorption in the sub-bandgap region.

Substrate Dependence

For sputtered AlN on (111) Si, crystalline film was achieved starting at a substrate temperature of 550 °C. The $(1\bar{1}02)$ Al_2O_3 substrate was found not to be amenable to epitaxial hexagonal AlN growth. Using (0001) Al_2O_3, wurtzite AlN was obtained by both sputtering and IAMBE, which indicated that it formed a good template for basal-oriented formation of AlN thin film. At 550 °C for (111) Si substrates, the AlN film was about as good in crystalline quality as 700 °C for ion-assisted MBE AlN/(0001) Al_2O_3.

RESULTS AND DISCUSSION

General Discussion

Figure 1 shows the X-ray diffraction (XRD) data of AlN/(111) Si substrate using sputtering, indicating basal orientation. The X-ray peak intensity decreased with increasing substrate temperature. The FWHM of the AlN peak was found to increase with increasing temperature (Figure 2).

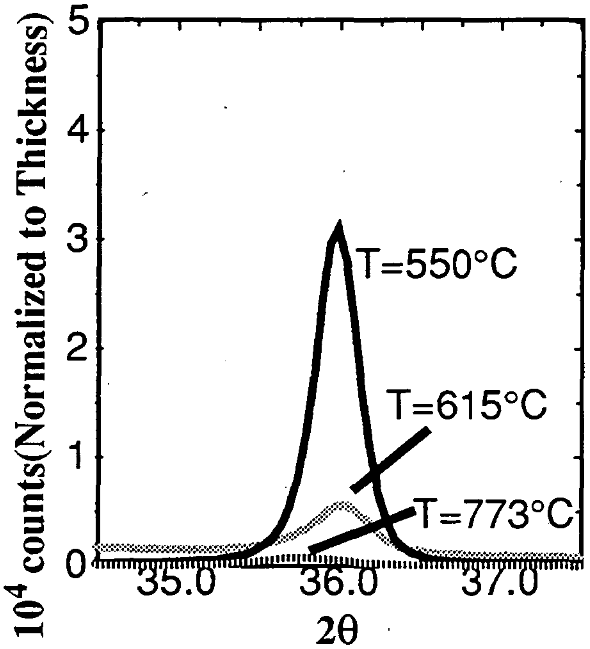

Figure 1: XRD θ-2θ scan of (0002) AlN/(111) Si as a function of substrate temperature.

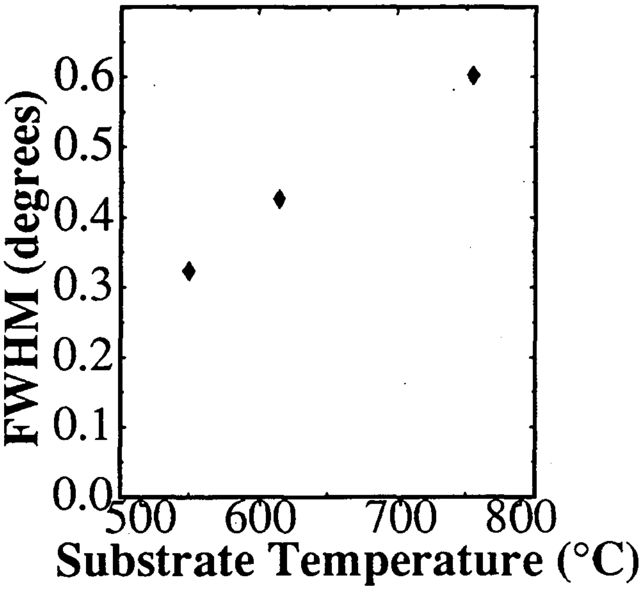

Figure 2: FWHM of AlN/(111) Si vs. substrate temperature.

For films sputtered on the $(1\bar{1}20)$ Al_2O_3 substrates in the temperature range 25 - 900 °C, the presence of strong preferential orientation was not detected with X-ray diffraction analysis. However, for basal Al_2O_3 substrates, (0002) AlN X-ray peaks were observed at 700 °C, and the intensity increased with substrate temperature to 850 °C. Figure 3 shows the optical absorption spectrum of the same set of sputtered AlN films. AlN grown at a higher temperature gave a sharper absorption edge than at lower temperature. All the AlN films grown in this temperature range exhibited a bandgap value of 6.1 eV, which was extracted from the steepest part of the absorption curve. The increased signal at the higher energy region can be attributed to the band-to-band absorption of states at the band edges. The absorption below the bandgap region was more pronounced for AlN films prepared at lower substrate temperatures. By integrating the area underneath the absorption spectrum in the sub-bandgap region, we observed that the amount of sub-bandgap absorption in the film decreased with increasing substrate temperature (T_{sub}). Figure 4 shows the sub-bandgap absorption level (in arbitrary units) vs. growth temperature for sputtered AlN.

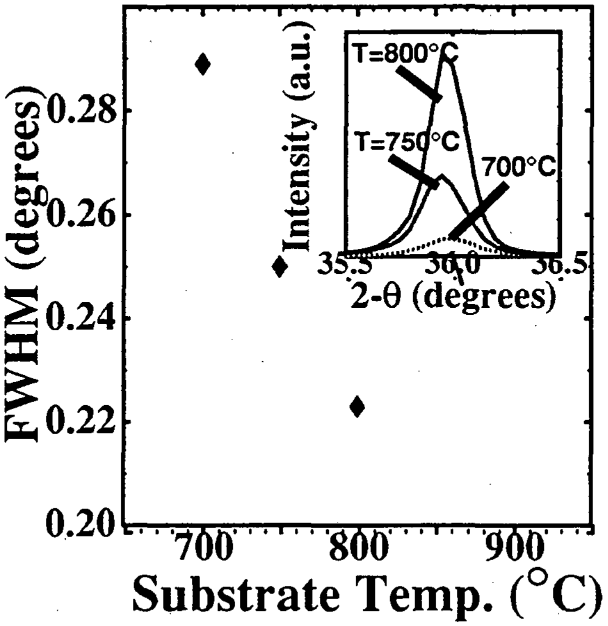

Figure 5: FWHM vs. substrate temperature for IAMBE AlN/(0001) Al_2O_3.

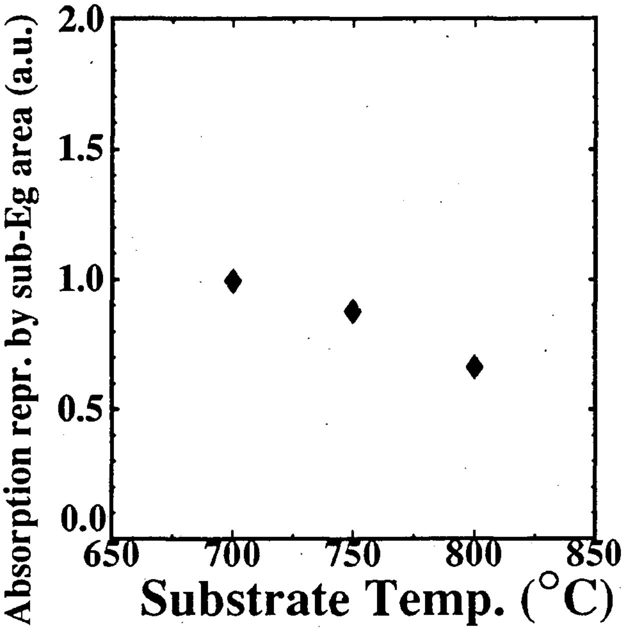

Figure 6: Optical absorption in sub-bandgap region vs. substrate temperature for (0002) AlN/Al_2O_3.

Temperature Effects

AlN grown on (111) Si exhibited a wurtzite structure perpendicular to the Si (111) plane starting at 550 °C. With increasing substrate temperature, the crystallinity of the AlN degraded. We expect a reaction at the surface between the Al and Si atoms during the growth. Al forms an eutectic solution with Si at 560 °C. For (0001) sapphire substrates, the improvement in crystalline quality of the AlN thin film for both RF sputtering and IAMBE was seen for increased substrate temperatures. The higher growth temperatures are believed to be important in overcoming the kinetic barriers of epitaxial AlN growth including formation of atomic nitrogen, adsorption, surface diffusion, and incorporation into the lattice. AlN is expected to be formed in the stable phase regime up to a substrate temperature of 1200 °C [13], and in the metastable regime to 1500 °C [14]. AlN thin film growth at higher substrate temperatures is planned.

The bandgap of sputtered AlN films on $(1\bar{1}02)$ Al_2O_3 ranged from 5.2 to 5.84eV depending on growth conditions. Consistent with AlN/(0001) Al_2O_3 films, these bandgap values were extrapolated from the steepest portion of the optical absorption spectrum. On the other hand, the sputtered AlN films on (0001) Al_2O_3 possessed the same bandgap of 6.1 eV, and did not change over the temperature range 700 - 850 °C.

CONCLUSION

We have successfully grown basal-oriented AlN on both (111) Si and (0001) Al_2O_3 substrates. AlN/(111) Si achieved crystallinity along the c-axis at 550 °C, but the crystallinity began to degrade with increased substrate temperature due to the reaction between the growing overlayer and substrate. On the other hand, the crystallinity of the AlN thin films grown on (0001)

Al_2O_3 improved with increasing substrate temperature when grown by both RF magnetron sputtering (700 - 850 °C) and IAMBE (700 - 800 °C). Furthermore, optical absorption measurements revealed that AlN/(0001) Al_2O_3 grown by the two methods possessed less sub-bandgap absorption when grown at higher temperatures.

REFERENCES

[1] W.M. Yim, E.J. Stofko, P.J. Zanzucchi, J.I. Pankove, M. Ettenburg, and S.L. Gilbert, J. Appl. Phys., vol.44, pp.292-294,1973.
[2] M. Mizuta, S. Fujieda, and Y. Matsumoto, Ext.Abstr.19th Conf. on Sol.St.Dev. and Matl., pp.135-136, 1987.
[3] J.S.Chan, N.W.Cheung, and K.M.Yu, Mat.Res.Soc.Symp.Proc.,vol.268,pp.377-82,1992.
[4] M.T. Duffy, C.C. Wang, G.D. O'Clock Jr., S.H. McFarlane III, and P.J. Zanzucchi, J. Electr. Matl., vol.2, no.2, pp.359-374, 1973.
[5] R. Tummala, Microelectronics Packaging Handbook, edited by R. Tummala and E. Rymaszewski (Van Nostrand Reinhold, New York, 1989), pp.493-494.
[6] K.L. Ho, K.F. Jensen, S.A. Hanson, J.F. Evans, D.C. Boyd, and W.L. Gladfelter, Mat.Res.Soc.Symp.Proc., vol.162, pp.605-610, 1990.
[7] F.C. Stedile, J.R.B. Baumvol, W.H. Schreiner, and F.L. Freire Jr.,J. Vac. Sci. Technol. A,vol.10(5), pp.3272-3275, 1992.
[8] W.J.Meng, J.A.Sell, and R.A.Waldo, J. Vac. Sci. Technol.A, vol.9, pp.2183-2186,1991.
[9] S. Yoshida, S. Misawa, and S. Gonda, J. Appl. Phys., vol.53, pp.6844-6847, 1982.
[10] Z. Sitar, M.J. Paisley, B. Yan, J. Ruan, W.J. Choyke, and R.F Davis, J. Vac. Sci. Technol. B, vol.8, pp.316-321, 1990.
[11] M.Miyauchi, Y.Ishikawa, and N.Shibata, Jpn. J. Appl. Phys., vol.31, pp.L1714-20,1992.
[12] C.R. Aita, J. Appl. Phys., vol.53, pp.1807-11, 1982.
[13] J.B.MacChesney,P.M.Bridenbaugh and P.B.O'Connor,Mater.Res.Bull.,vol.5,p.783,1970.
[14] N.Newman,J.Ross and M.Rubin, Appl.Phys.Lett.,vol.62, pp.1242-1244.

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