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Effect of Thickness on Structural and Morphological Properties of AlN Films Prepared Using Single Ion Beam Sputtering

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Abstract

Aluminum nitride (AlN) thin films have potential applications in microelectronic and optoelectronic devices. In this study, AlN thin films with different thicknesses were deposited on silicon substrate by single ion beam sputtering method. The X-ray diffraction (XRD) spectra revealed that the structure of films with thickness of 50-150 nm was amorphous, while the polycrystalline hexagonal AlN with a rough surface was observed at a thickness of 300 nm. Also, the formation of AlN in amorphous films is identified by Fourier transform infrared (FTIR) spectroscopy. Atomic force microscopy (AFM) study confirms that the surface roughness and average grain size of films increased with film thickness.
Key words: *AlN, Ion beam sputtering, optical properties, Structural properties.*

Introduction

Aluminum nitride (AlN) thin films due to its many excellent properties such as, thermal and chemical stability, high melting point, high acoustic velocity, good dielectric properties, transparency in the visual and infrared region, large optical band gap energy [1-6], have been used in microelectronic and optoelectronics devices especially in surface and bulk acoustic wave devices[3-6]. Also, AlN thin film has been applied as buffer layer to grow of ZnO nanostructures on silicon and sapphire [7]. AlN thin films can be deposited by several techniques including, RF or DC reactive magnetron sputtering [8-10], pulsed laser deposition [11], molecular beam epitaxy (MBE)[12], plasma nitriding [13,14], dual ion beam sputtering (DIBS) [15], and

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single ion beam sputtering (SIBS) [16,17]. Among these, ion beam sputtering method has advantages over other sputtering methods, such as independent control over the incident beam energy, the current density and incidence angle of bombarding ions. Also, ion beam sputtering method is useful for the manufacturing of optical films. However, preparing of AlN thin films by SIBS has rarely been reported and the objective of the present work is synthesis of AlN films by this method. Also, the effect of film thickness on structural and morphological properties of prepared films has been studied using different analyzing methods such as, X-ray diffraction (XRD), atomic force microscopy (AFM) and Fourier transform infrared spectroscopy (FTIR).

Experimental

Materials and Methods

The AlN films with different thicknesses have been deposited on silicon (p-Si <400>, 5-10 Ω.cm) by means of a single reactive ion beam sputtering system using a pure aluminum target (purity 99.999%) and a mixture of argon (99.999%) and nitrogen (99.999%) gases with equal contents. To achieve different thicknesses the deposition time was varied from 10 to 70 minutes while the other parameters, such as working gas partial pressure ratio (Ar and N2), ion beam energy and current were kept constant. Prior to any deposition, the deposition chamber was carefully cleaned and then evacuated to background pressure of 6×10^{-6} torr before introducing the gas mixture. Also, the substrates were ultrasonically cleaned in acetone and ethanol for about 30 min and dried with dry argon before loading into the deposition chamber. The ion beam energy and ion beam current for sputtering were kept at 2.2 keV and 25 mA respectively during the deposition. The working pressure was 2×10^{-4} torr and the flow rates of argon and nitrogen were 25 sccm. The substrate temperature was held at 400 ºC during film deposition with using a heater and thermocouple. The deposition parameters are summarized in Table 1. A Detak3 surface profilometer was employed to measure the films thickness. The crystallographic structure of AlN films was studied by X-ray diffraction (XRD, PW1800, Philips). The Fourier transform infrared spectroscopy (FTIR; Perkin Elmer spectrum 100) was used to identify the chemical composition of the films deposited on silicon substrates. AFM analysis (AFM, Park Scientific Instrument Auto probe CP) was performed to study the surface morphology of deposited films.

Table 1. The deposition parameters.

Deposition method	Single ion beam sputtering
Target	Al
Substrate	silicon
Sputtering gas	Ar
Reactive gas	N_2
Ar flow rate (sccm)	25
N_2 flow rate (sccm)	25
Base pressure (Torr)	$6\,u\,10^{-6}$
Working pressure (Torr)	$2\,u\,10^{-4}$
Substrate temperature (ºC)	400
Ion beam energy for sputtering (keV)	2.2
Ion beam current (mA)	20
Deposition time (min)	10 For Sample S1 20 For Sample S2 50 For Sample S3 70 For Sample S4

Results and discussion

The film thicknesses were in the range of 50-300 nm and the plot of film thickness versus the deposition time is shown in Figure 1. The surface morphology of films is examined with an atomic force microscopy in contact mode (scan area: 2×2 μm^2) and the results are shown in Figure 2.

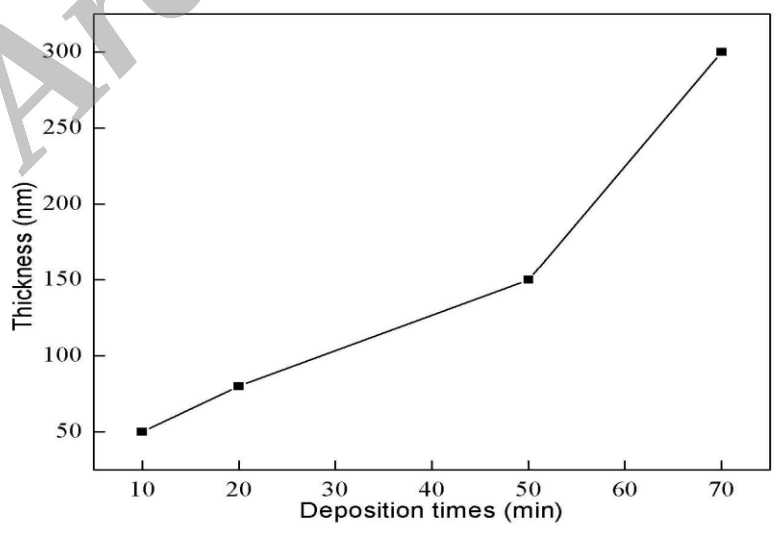
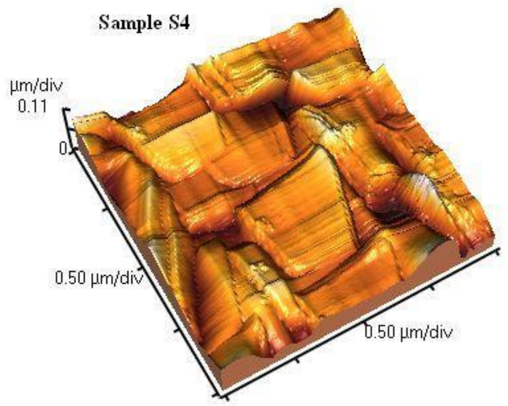


Figure 1. The plot of AlN films thickness versus deposition time.


Sample S1
μm/div
0.00073
0
0.50 μm/div
0.50 μm/div
Sample S1
0.40 nm/Div
0.00
1.00
2.00 μm
Sample S2
μm/div
0.0055
0
0.50 μm/div
0.50 μm/div
Sample S2
4.0 nm/Div
0.00
1.00
2.00 μm
Sample S3
μm/div
0.0037
0
0.50 μm/div
0.50 μm/div
Sample S3
2.0 nm/Div
0.00
1.00
2.00 μm



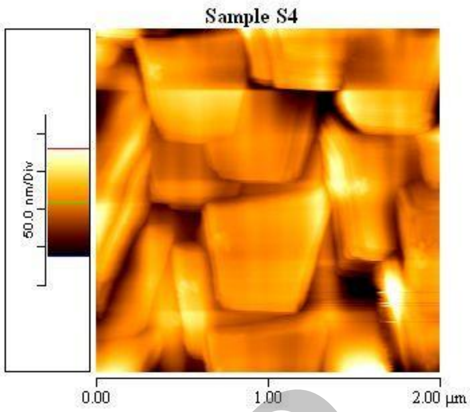


Figure 2. 3D and 2D AFM images of AlN films with various thicknesses deposited on Si substrates.

It is observed that, the films deposited in smaller deposition times are smooth and dense and by increasing the deposition time up to 70 min the pyramidal types of grains grow perpendicular to the substrate surface. When the deposition time increased to 70 min the morphology of films has completely changed to separate hillock grains and showed a highly agglomerated growth mode with high surface roughness. The AFM analysis indicated that, the root mean square (RMS) surface roughness increases from 0.11 to 0.93 nm when the film thickness varies from 50 to 150 nm. However, the RMS roughness increased drastically to 23.57nm for thickness of 300 nm that indicates of rough surface resulting from 3D island growth mode. The roughness values are shown in Table 2.

Table2. The deposition time, thickness, RMS and average roughness of AlN films.

Sample	S1	S2	S3	S4
Deposition time (min)	10	20	50	70
Thickness (nm)	50	80	150	300
RMS Roughness (nm)	0.11	0.81	0.93	23.57
Average Roughness (nm)	0.07	0.63	0.73	17.91

Also, the average grain size evaluated from AFM measurements varied in the range of 65 to 350 nm and the results are shown in Figure 3. Therefore, it is apparent that the average grain size and RMS roughness of the films increases with increasing film thickness. A similar increase of surface roughness with the increase of grain size was also reported by Z. Xin etal [18] in dc magnetron sputtered Au films.

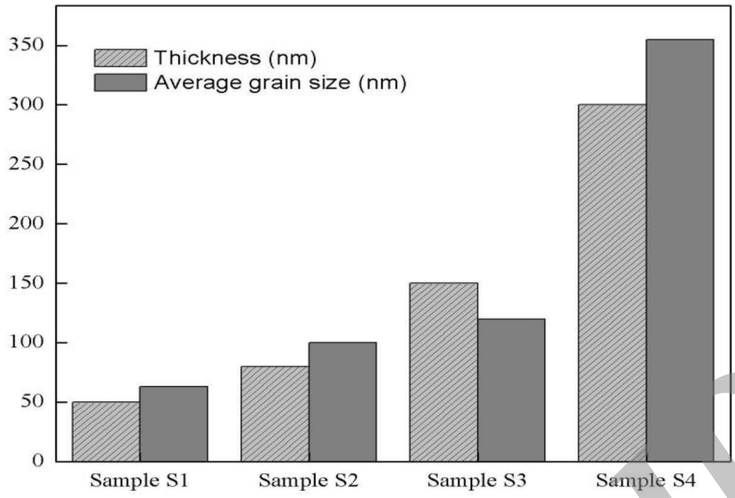


Figure 3. The plot of thickness and grain size for Samples (S1-S4).

The XRD patterns of deposited films on silicon substrates for different thicknesses in the range of 50-300 nm are depicted in Figure 4. It is clear that the films with thickness lower than 300 nm show any crystalline phase of AlN film and only one peak belonging to Si at about 70 degree is observed. The film of 300 nm thickness shows two characteristic peaks of hexagonal AlN at 2θ =33.36º diffracted from the (100) plane and at 2θ =58.68º diffracted from the (110) plane, as well as a peak at 2θ=38.89º belonging to cubic Al (111) plane. This indicates that the film is polycrystalline. we can say that for films with thickness of 50-150nm the deposition times varied between 10-50 min and the atoms have insufficient time to diffuse and grow, causing the amorphous structure and then diffusion has begun cause the grain growth after 70 min. These XRD patterns imply that the crystallization can be improved by films thickness. This improvement of crystalline quality has been confirmed by AFM results.

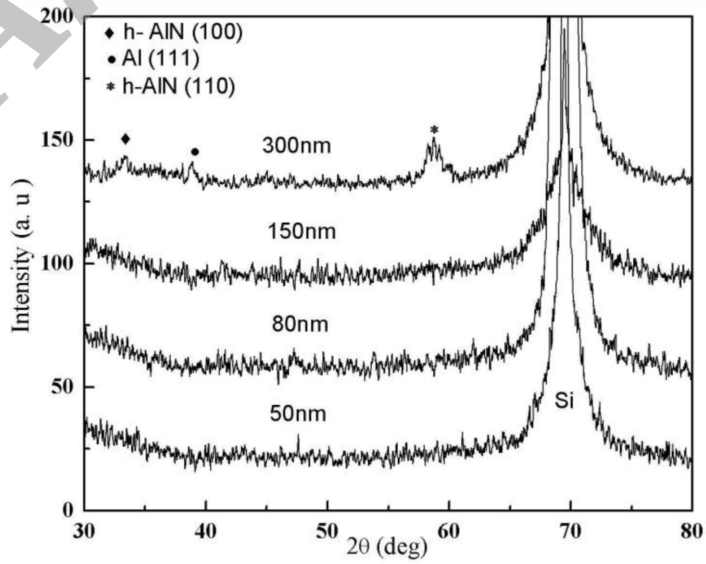


Figure 4. X-ray diffraction patterns of AlN films with various thicknesses deposited on Si substrate.

For the amorphous films, the Al-N bond information is recorded by FTIR spectroscopy at room temperature in the transmission mode with spectral resolution of 4cm-1. Figure 5 shows the FTIR spectra of the amorphous films deposited on Si substrate at different times of deposition. The absorption peak at 611cm-1 is related to the silicon substrate and two other absorption peaks at 668 and 739cm-1 are assigned to the Al-N bond, which is very close to the characteristic value of aluminum nitride [19-22]. It is observed that the intensity of absorption peak of Al-N bond increases with increase of deposition time due to an increase of film thickness.

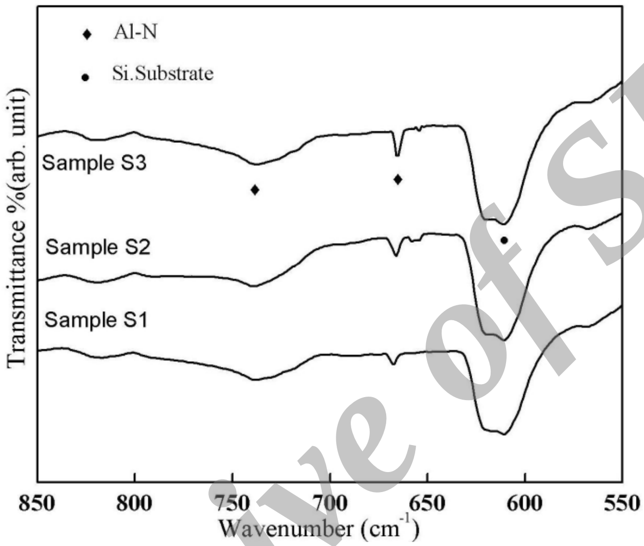


Figure 5. FTIR transmittance spectra (T) of AlN films deposited on Si substrates versus wavenumber (k).

Conclusion

The single ion beam sputtering method has been used to prepare of AlN films with different thicknesses in the range of 50-300 nm onto silicon and quartz substrates. The influence of film thickness on the structural and morphological properties of AlN films was studied. It has been found that an increase in films thickness leads to change of films structure from amorphous to crystalline. FTIR transmittance spectra confirmed the results obtained from the XRD. The surface roughness of films, were found to increase with increasing film thickness, which is associated with the phase change, agglomeration of grains, and growth of grain size.

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References

[1] F. Jose, R. Ramaseshan, S. Bera, A. K. Tyagi, B. Raj, *J. Phys. D Appl. Phys.*, 43, 075304 (2010).
[2] T. S. Pan, Y. Zhang, J. Huang, B. Zeng,

D. H. Hong, S. L. Wang, H. Z. Zeng, M.Gao, W. Huang, Y. Lin, *J. Appl. Phys.*, 112, 044905 (2012).

[3] Z. R. Song, Y. H. Yu, S. C. Zou, Z. H. Zheng, D. S. Shen, E. Z. Luo, Z. Xie, B. Sundaravel, S. P. Wong, I. H. Wilson, *Thin Solid Film*, 459, 41 (2004).

[4] S. Bakalova, A. Szekeres, G. Huhn, K. Havancsak, S. Grigorescu, G. Socol, C. Ristoscu, I. N. Mihailescu, *Vacuum*, 84, 155 (2009). .

[5] A. Artieda, M. Barbieri, C. S. Sandu, P. Muralt, *J. Appl. Phys*., 105, 02450 (2009).

[6] M. Clement, E. Iborra, J. Sangrador, A. Sanz-Hervas, L. Vergara, *J. Appl. Phys*., 94, 1495 (2003).

[7] R. Yousefi, B. Kamaluddin, M. Ghoranneviss, F. Hajakbari, *Appl. Surf. Sci*., 255, 6985 (2009).

[8] Y. Z. You, D. Kim, *Thin Solid Film*, 515, 2860 (2007).

[9] S. Venkataraj, D. Severin, R. Drese, F. Koerfer, M. Wuttig, *Thin Solid Film*, 502, 235 (2006).

[10] B. Abdallah, C. Duquenne, M. P. Besland, E. Gautron, P. Y. Jouan, P.Y. Tessier, J. Brault, Y. Cordier, M. A. Djouadi, *Eur. Phys. J. Appl. Phys*., 43, 309 (2008).

[11] A. Szekeres, Zs. Fogarassy, P.Petrik, E. Vlaikova, A. Cziraki, G. Socol, C. Ristoscu, S. Grigorescu, I. N. Mihailescu, *Appl. Surf. Sci*., 257, 5370 (2011).

[12] B. Liu, J.Gao, K. M. Wu, C. Liu, *Solid state Commun*., 149, 715 (2009).

[13] M. Sadiq, S. Ahmad, A. Waheed and M. Zakaullah, *Plasma Sources Sci. Technol*., 15, 295 (2006).

[14] S. Nasser, F. U. Khan, N. U. Rehman, A. Qayyum, F. Rahman, M. Zakaullah, *Eur. Phys. J. App. Phys*., 49, 21001 (2010).

[15] H. Y. Chen, Sh. Han, H. C. Shih, *Surf. Coat. Technol.*, 200, 3326 (2006).

[16] F. Hajakbari, M. M. Larijani, M. Ghoranneviss, M. Aslaninejad, A.Hojabri, *Jpn. J. Appl. Phys*., 49, 095802 (2010).

[17] R. Dhunna, Ch. Lal, V. Sisodia, D. K. Avasthi, V. Ganesan, I. P. Jain, *Mater. Sci. Semicond. Process.*, 11, 126 (2008).

[18] Z. Xin, S. Xiao-Hui, Z. Dian-Lin, *Chin. Phys. B*., 19, 086802 (2010).

[19] L. Shen, R. k. Y. Fu, P. K. Chu, *Rev. Sci. Instrum*., 75, 719 (2004).

[20] Y. Fu, X. Li, Y. Wang, H. He, X. Shen, *Appl. Phys. A*., 106, 937 (2012).

[21] L. Lei, L. Q. Shan, L. Li, Z. L. Chun, W. C. Feng, Q. H. Xia, *Chin. Phys. Lett*., 24, 552 (2007).

[22] Z. M. Ren, Y. F. Lu, H. Q. Ni, T. Y. F. Liew, B. A. Cheong, S. K. Chow, M. L. Ng, J. P. Wang, *J. Appl. Phys.*, 88, 7346 (2000).