



Study on c-axis orientation of AlN thin film on the influence Al buffer layer and Ar/N₂ gas flow ratio in reactive magnetron sputtering

M. Vanamoorthy^a, Bindu Salim^{b*}

^a Centre of Polymer Systems, University Institute, Tomas Bata University in Zlin, Trida Tomase Bati 5678, 760 01 Zlin, Czech Republic.

^b Director-Research, Sri Shakthi Institute of Engineering and Technology, Coimbatore, Tamil Nadu, India.

* Corresponding Author: bindusalim@ieec.org

Received: 17-10-2023, Revised: 23-11-2023, Accepted: 01-12-2023, Published: 12-12-2023

Abstract: AlN is a piezoelectric material suitable for high temperature dynamic pressure sensing applications. Its piezoelectric coefficient purely depends on its crystal structure and growth direction. Highly c-axis (002) orientation exhibits high piezoelectric coefficient. Deposition of highly (002) oriented AlN thin film poses a challenge since such a growth depends on multiple process parameters and substrate material. In this work, AlN thin film was deposited using reactive radio frequency (RF) magnetron sputtering to correlate the gas flow rate and crystal orientation. AlN deposition was carried out on Si (100) substrate with and without 220 nm Al buffer layer under different Ar/N₂ gas flow ratio. The samples were analyzed through X-ray diffraction technique. Results indicated that for the optimized value of 1:1 Ar/N₂, (002) AlN intensity at its maximum for both AlN/Si and AlN/Al/Si samples. It is also observed that the use of 220 nm Al buffer layer on Si substrate enhanced the (002) intensity compared to AlN/Si.

Keywords: AlN Thin Film, C-axis, Ar/N₂, AlN/Si, Magnetron Sputtering

1. Introduction

In the field of nano and micro electromechanical devices and system, Aluminum Nitride (AlN) is drawing importance due to its place in group III-nitride materials. Because of its compatibility to standard CMOS processes, chemical stability and piezoelectric property, AlN is commonly used in sensors and actuators as active layer [1-3]. Especially for high temperature piezoelectric pressure sensing applications, AlN turns to be a suitable material due to its superior thermal properties compared to other piezoelectric materials like PZT and ZnO. But to enhance the piezoelectric coefficient of AlN, one need to achieve highly c-axis oriented AlN

since its piezoelectric property rely on its crystal structure i.e., Hexagonal Wurtzite with high (002) or *c*-axis orientation [4]. There are various techniques to develop AlN thin films, of which reactive RF magnetron sputtering is the most commonly used method due to its easy process control and low deposition temperature. Many reported the impact of sputtering parameters like temperature, power, gas flow working pressure etc., on AlN growth orientation [4]. Among those, varying the Ar and N₂ flow ratio is a simple but effective way to influence the AlN growth. Cheng *et al.* [5] and Kar *et al.* [6] specifically studied the influence of N₂ concentration on AlN growth orientation. Both of them have grown AlN on Si (100) substrates and reported that (002) orientation has been enhanced with increase in N₂ concentration. Liu *et al.* [7] also reported the strong influence of the N₂ concentration on AlN film but differed with the end result as they observed decrease in FWHM of the (002) amplitude with decrease in nitrogen concentration. Here, AlN deposition was carried out on Si (100) substrates with and without Al buffer layer, controlling the Ar to N₂ ratio. X-ray diffraction study was performed to investigate the growth orientation of AlN.

2. Methodology

The substrates used for this experiment were Si (100) wafers. Degreasing of substrates was carried out by using RCA cleaning method. Deposition of AlN and 220 nm Al buffer layer thin films were carried out in a Multi-Source PVD system (HHV TF600) using RF magnetron sputtering (SEREN R601 power supply with MC2 automatic matching network controller) in a class 10000 clean room environment. For the deposition of 300 nm Al buffer layer on Si (100) substrate, the key sputter parameters used are given in table 1. The sputter parameters used to deposit AlN were taken from a previous work [8] which is also listed in table 1. AlN was deposited under different Ar/N₂ gas flow (i.e., 15/45, 20/40, 30/30, 40/20 sccm) on well cleaned Si (100) substrate with and without 300 nm Al buffer layer labeled as AlN/Si and AlN/Al/Si respectively. The gas flow was controlled using mass flow controller (Bronkhorst High-Tech, EL-FLOW). Prior to each deposition the pressure inside the sputtering chamber was pumped down to below 5×10^{-4} Pa before high purity (99.999%) Ar and N₂ were introduced then pre sputtering was done for 15 minutes.

Table 1. Key sputter parameters

Parameter	Al buffer layer	AlN
Target	Al (99.999%)	
Power (W)	100	400
Temperature (°C)	RT	300
Working Pressure (Pa)	0.93	0.66

Gas Flow Ar/N₂(sccm)	30/0	15/45,20/40, 30/30, 40/20
Duration (min.)	15	60

By using Empyrean PANalytical diffractometer with Cu-K α X-ray radiation, crystal structure and orientations of the films were identified in GIXRD mode with incident angle (ω) of 6.5°. “HighScore” Software was used for XRD data interpretations and calculations. Thickness of the Al buffer layer was measured as 220 nm by using a stylus profilometer (α -step D-600).

3. Result and Discussion

Figure 1a and 1b shows the XRD results of AlN deposited under different Ar/N₂ flow (i.e., 15/45, 20/40, 30/30 and 40/20 in sccm) on Si and Al/Si substrates. It is clear from figure 1 that all the samples are grown with high (002) orientation. Growth orientations of AlN and Al is indicated in figure 1 along with its respective ICDD file numbers where, AlN and Al have hexagonal wurtzite and simple cubic crystal structures respectively. With a fixed gas flow of 60 sccm, when we decreased the N₂ flow, it is observed that the intensity of (002) AlN is increasing up to the point where the flow of Ar and N₂ are equal (i.e., 30 sccm). When the N₂ flow is decreased further we observed decreasing nature in (002) intensity of AlN.

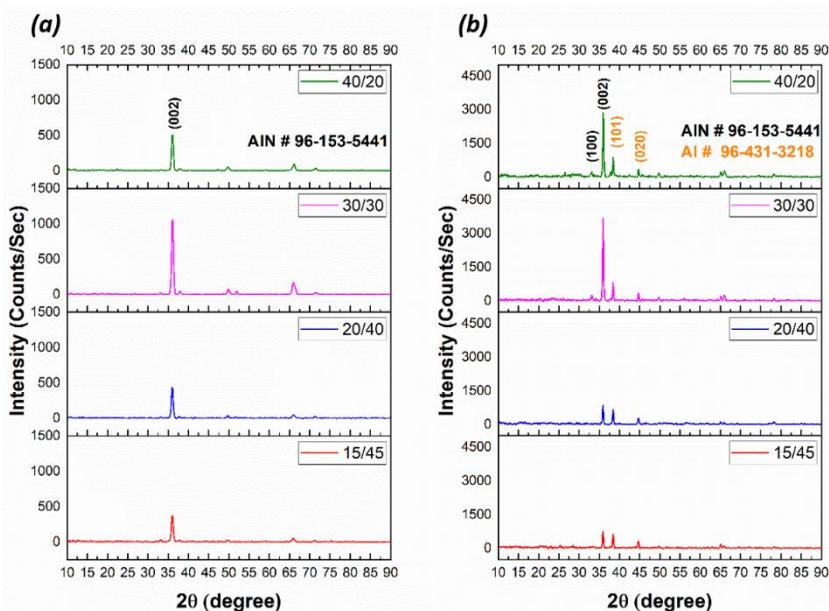


Figure 1. GIXRD Results of AlN samples prepared on Si substrates (a) without Al buffer layer (b) with Al buffer layer under different Ar/N₂ gas flow

This mechanism is observed irrespective of the substrates used. Smaller-mass N₂ particles and the bigger-mass Ar particles are playing a major role behind this mechanism by

impacting the adatoms energy. In the case of higher N_2 concentration, the rate of deposition is reduced due to lack of high energy Ar particles which lead to insufficient adatom energy and mobility. In the case of lower N_2 concentration, the sputtered particles lose its energy greatly by colliding with a greater number of heavy Ar particles which again led to low adatom energy. But for the optimized gas flow of 30 sccm Ar and N_2 , maximum (002) intensity is achieved irrespective of the substrates used (Figure 2). So, it is verified from the results that the optimized Ar/ N_2 gas flow is needed to develop highly c-axis oriented AlN. Figure 2 also reveals the comparison in (002) intensity between AlN deposited on Si (100) with and without 220 nm Al buffer layer. It is clear from the results that the use of buffer layer enhanced the (002) growth compared to AlN/Si. This is possible due to the presence of same mother element that is Al between AlN and the interface layer. This may promote the sputtered particles to more easily sit in the respective lattice sites on the surface of the Al buffer layer.

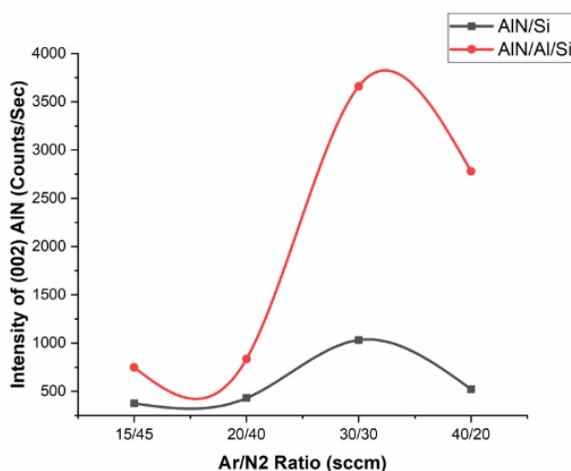


Figure 2. (002) intensity of AlN/Si and AlN/Al/Si deposited under different Ar/ N_2 gas flow

4. Conclusion

In this work, AlN was deposited on Si (100) substrates with and without Al buffer layer using reactive RF magnetron sputtering. Effect of nitrogen concentration on the (002) growth orientation of AlN is investigated with fixed gas flow of 60 sccm. It is verified from our results that for the optimized value of Ar/ N_2 ratio as 1:1 (i.e., 30 sccm each), (002) AlN intensity is at its maximum for both AlN/Si and AlN/Al/Si samples. It is also observed that introduction of 220 nm Al buffer layer enhanced the (002) intensity compared to AlN/Si. AlN being the most suitable material for high temperature piezoelectric applications, process optimization to achieve c-axis thin film is challenging. Also, the process parameters change from system to system. This study was done to add knowledge on controlling Ar/ N_2 ratio for optimum results.

References

- [1] M. Akiyama, Y. Morofuji, T. Kamohara, K. Nishikubo, M. Tsubai, O. Fukuda, N. Ueno., Flexible piezoelectric pressure sensors using oriented aluminum nitride thin films prepared on polyethylene terephthalate films, *Journal of Applied Physics*, 100(11) (2006) 114318. <https://doi.org/10.1063/1.2401312>
- [2] D. Chen, J. Wang, D. Li, L. Zhang, X. Wang, The c-axis oriented AlN solidly mounted resonator operated in thickness shear mode using lateral electric field excitation, *Applied Physics A: Materials Science and Processing*, 100(1), (2010) 239–244. <https://doi.org/10.1007/s00339-010-5591-6>
- [3] K. Kishi, Y. Ooishi, H. Noma, E. Ushijima, N. Ueno, M. Akiyama, T. Tabaru, Measurement of output voltage of aluminum nitride thin film as a pressure sensor at high temperature, *Journal of the European Ceramic Society*, 26(15), (2006) 3425–3430. <https://doi.org/10.1016/j.jeurceramsoc.2005.08.011>
- [4] A. Iqbal, F. Mohd-Yasin, (2018) Reactive sputtering of aluminum nitride (002) thin films for piezoelectric applications: A review, *Sensors*, 18(6), 1797. <https://doi.org/10.3390/s18061797>
- [5] H. Cheng, Y. Sun, J.X. Zhang, Y.B. Zhang, S. Yuan, P. Hing, AlN films deposited under various nitrogen concentrations by RF reactive sputtering, *Journal of Crystal Growth*, 254(1–2), (2003) 46–54. [https://doi.org/10.1016/S0022-0248\(03\)01176-X](https://doi.org/10.1016/S0022-0248(03)01176-X)
- [6] J.P. Kar, G. Bose, S. Tuli, Influence of nitrogen concentration on grain growth, structural and electrical properties of sputtered aluminum nitride films, *Scripta Materialia*, 54(10), (2006) 1755–1759. <https://doi.org/10.1016/j.scriptamat.2006.01.038>
- [7] H.Y. Liu, G.S.Tang, F. Zeng, F. Pan, Influence of sputtering parameters on structures and residual stress of AlN films deposited by DC reactive magnetron sputtering at room temperature. *Journal of Crystal Growth*, 363, (2013) 80–85. <https://doi.org/10.1016/j.jcrysgro.2012.10.008>
- [8] M. Vanamoorthy, B. Salim, K. Mohanta, Study on optimizing c-axis oriented AlN thin film for piezoelectric sensing applications controlling the sputtering process parameters, *Applied Physics A*, 128(48), (2022). <https://doi.org/10.1007/s00339-021-05166-5>

Funding

No funding was received for conducting this study.

Conflict of interest

The Authors have no conflicts of interest to declare that they are relevant to the content of this article.

About The License

© The Author(s) 2023. The text of this article is open access and licensed under a Creative Commons Attribution 4.0 International License