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T. Tavsanoğlu

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# Synthesis of c-axis oriented AlN thin films at room temperature

T. Tavsanoglu\*

In this study, aluminium nitride (AlN) thin films were synthesised by plasma-enhanced reactive DC magnetron sputtering on glass and Si (100) substrates and the effect of bias voltages on the structural, optical and morphological properties of the coatings has been studied. According to the grazing angle XRD studies, AlN in hexagonal (wurtzite) structure has been obtained for all the coatings. (002) plane c-axis oriented films have been achieved at 150 and 175 V bias voltages. AFM analyses demonstrated that with the increase in the bias voltages, a transformation from coarse to fine granular morphology has been occurred and smoother surfaces with a decrease in the surface roughness from 3.40 nm to about 1.90 nm were obtained. The results also demonstrated that the high transmittance values of AlN films were not affected by the change in the bias voltages and about 80% of transmittance obtained for all AlN films deposited in this study.

**Keywords:** c-axis oriented AlN thin films, Plasma-enhanced DC magnetron sputtering, Bias voltage, Surface morphology, Microstructure, Crystallinity, Optical properties, Transmittance

## Introduction

Aluminium nitride (AlN) has a considerable standing among nitride-based materials with its superior mechanical, electrical, optical and thermal properties.<sup>1–10</sup> AlN thin films are widely used in microelectronic and optoelectronic devices such as ultraviolet detector, light emitting diodes, thermal interface materials, III–V semiconductors and insulators.<sup>1–8</sup> AlN films are also used as protective coatings, especially with Ti incorporation into the structure.<sup>11–15</sup> AlN has good piezoelectric properties and has been used in surface acoustic wave (SAW) devices.<sup>6,9,10</sup> In addition, AlN has a large optical band gap of about 5.9–6.02 eV and high transparency properties in UV and visible light range which makes it a good candidate for transparent electronics and optoelectronics.<sup>6,16,17</sup> Furthermore, AlN has been found applications in electronic structures with Ga- and In-based materials as insulator components and as alloying material for AlGaIn-based optical and electronic devices.<sup>5,6,18</sup> The deposition of AlN films with controlled morphology and high crystalline quality is mandatory to fabricate high performance AlN-based devices.<sup>6</sup> Especially low roughness is the prime requisite of a thin film for such applications.<sup>19</sup> To achieve these qualities, it is essential to understand the effect of the deposition parameters.<sup>19</sup>

AlN thin films have been obtained by different techniques such as molecular beam epitaxy,<sup>20,21</sup> plasma focus deposition,<sup>22</sup> chemical vapour deposition,<sup>23</sup> pulsed DC sputtering,<sup>24</sup> rf sputtering,<sup>18,25,26</sup> high power impulse

magnetron sputtering<sup>27,28</sup> and reactive DC magnetron sputtering.<sup>29–32</sup>

It is known from previous studies that c-axis (002) oriented AlN films have better piezoelectric properties,<sup>26,33</sup> but their deposition generally necessities high temperatures (300–1200°C).<sup>9</sup> Moreover, for most of the microelectronic and optoelectronic applications, piezoelectric AlN films need to be grown on a polycrystalline electrode film surface and the deposition temperature may not exceed 500°C so as to ensure compatibility with the standard integrated circuit technology.<sup>34</sup> Hence, expensive equipment and high deposition temperatures for preparing c-axis oriented AlN films have limited the wide applications of these films in microelectronic and optoelectronic fields.<sup>9,30</sup> In this study, the depositions were realised without any external heating, at room temperature and necessary energy for the transformation from (100) plane to c-axis (002) plane orientation was supplied by the bias voltages applied to the substrates that are in contact with the high-density plasma created by plasma-enhanced configuration. The effect of the bias voltages on the structural, morphological and optical properties of AlN thin films was investigated.

## Experimental

AlN thin films were deposited by plasma-enhanced reactive DC magnetron sputtering (HEF TSD350) of a planar and water cooled Al target (99.99% purity). Glass and Si (100) substrates were used in each deposition. The distance of the insulated substrate holder from the target was 65 mm. The base pressure of  $5 \times 10^{-5}$  Pa was obtained by a combination of a rotary and turbomolecular pump system. High-purity (99.999%) Ar and N<sub>2</sub> were

Faculty of Engineering, Department of Metallurgical and Materials Engineering, Mugla Sıtkı Kocman University, 48000, Kotekli, Mugla, Turkey

\*Corresponding author, email [tolgatavsanoglu@mu.edu.tr](mailto:tolgatavsanoglu@mu.edu.tr)

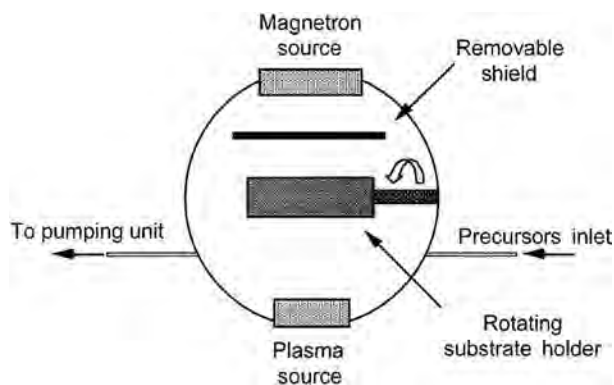
**Table 1. Deposition conditions of AlN films**

	AlN1	AlN2	AlN3	AlN4	AlN5	AlN6
Base pressure (Pa)	$5 \times 10^{-5}$	$5 \times 10^{-5}$	$5 \times 10^{-5}$	$5 \times 10^{-5}$	$5 \times 10^{-5}$	$5 \times 10^{-5}$
Working pressure (Pa)	0.3	0.3	0.3	0.3	0.3	0.3
Ar (sccm)	4	4	4	4	4	4
N <sub>2</sub> (sccm)	16	16	16	16	16	16
Power (W)	220	220	220	220	220	220
Voltage (V)	270	270	270	270	270	270
Current (A)	0.82	0.82	0.82	0.82	0.82	0.82
Bias (-V)	0	75	150	175	200	250
Temperature (°C)	40 ± 5	40 ± 5	40 ± 5	40 ± 5	40 ± 5	40 ± 5
Duration (min)	60	60	60	60	60	60

used as precursor and reactive gas respectively. N<sub>2</sub> fraction in the deposition gas was controlled by varying the flow rate through a mass flow controller. The working pressure was 0.3 Pa in all depositions. The cathode power was fixed at 220 W and Ar/N<sub>2</sub> ratio was kept constant at 20/80 for all the experiments. The substrates were cleaned in ultrasonic bath, in ethanol and were then blown dry with nitrogen. Before the deposition of each coating, Al target was pre-sputtered in the argon atmosphere for 30 minutes. Meanwhile, substrates were bias-etched with a gradually applied voltage (50–250 V). All depositions were realised without external heating at 40 ± 5 °C as a result of ion-matter interactions. The deposition time kept constant at 60 min for each deposition. Deposition conditions of AlN films are given in Table 1.

In the plasma-enhanced magnetron sputtering design shown in Fig. 1, the auxiliary plasma source is powered positively. Fast electrons produced by the ion bombardment of the target are trapped near the target by the magnetron design, whereas slow electrons are accelerated towards the positively powered plasma source, then they collide with argon atoms and create high-density plasma over the substrate holder.<sup>35</sup> In this configuration, while film growth by the negative magnetron source; the substrates were bombarded with highly energetic ions generated by the assistance of positive plasma source.<sup>35</sup>

Crystal structure of AlN films was characterised by XRD studies. The analyses were made by a grazing incidence X-ray diffractometer with a thin film attachment (Philips Model PW3710) using Cu-K $\alpha$  radiation over the 2 $\theta$  range of 30–80°. The  $\theta$  scan method with a fixed

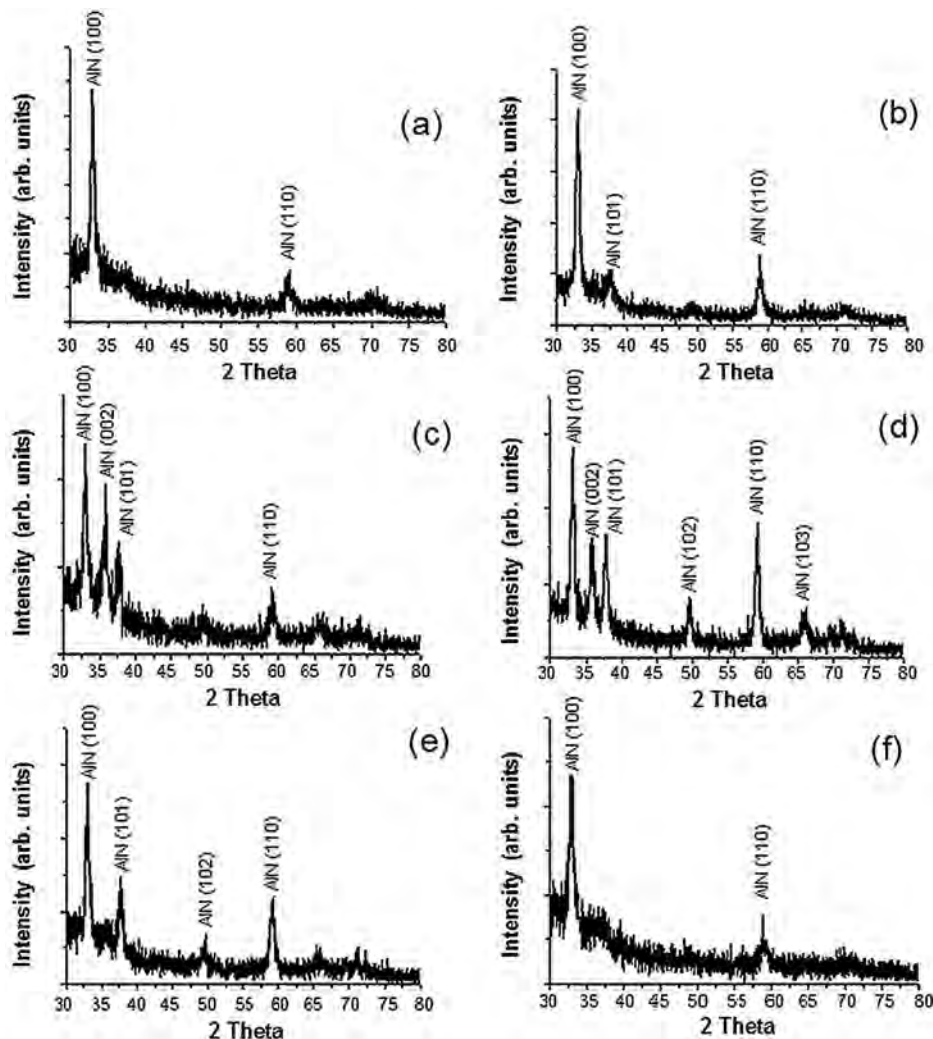
**1 Schematic of the deposition reactor**

incidence angle of 0.5° was used. Phases through the films were identified by matching the diffraction peaks with those of JCPDS database. Surface morphology of AlN thin films was characterised by AFM studies in contact mode (SPM-9500J3 Scanning Probe Microscope, Shimadzu). The elemental concentration of the films deposited on Si (100) substrates was obtained using a secondary ion mass spectrometer (SIMS-CAMECA IMS 6f) over their thicknesses. Optical measurements were made by an NKD-7000 V model spectrophotometer (Aquila Instruments, UK) over the spectral range from 300 to 1000 nm.

## Results and discussion

### Structural analyses

Crystallographic analyses of deposited films were carried out by grazing incidence XRD studies. According to the results, polycrystalline AlN in hexagonal (wurtzite) structure has been achieved for all the coatings (File No. 25–113 JCPDS-ICDD database). The effect of the bias voltages on the crystallographic orientation of AlN films is demonstrated in Fig. 2. As can be seen in Fig. 2, no strong textures were developed in any of the samples, but rather, a mixture with variable relative amounts of mainly (100), (110), (101) and (002) orientations was observed. For all deposited films, (100) plane orientation occurred in which c-axis is parallel to the substrate,<sup>29</sup> by increasing bias voltages films were also oriented in other planes. For AlN1 deposited at floating potential without applying any bias voltage, strong orientation in (100) plane was observed. For AlN2 deposited at 75 V bias voltage, no dramatic changes in orientation occurred. Orientation in (002) plane of AlN films, in which c-axis is perpendicular to the substrate<sup>36</sup> have been observed for AlN3 and AlN4 deposited at 150 and 175 V bias voltages respectively. (002) plane orientation for 150 V bias voltage was stronger and increasing bias voltage to 175 V resulted in a decrease in (002) peak. Further increase in the bias voltage to 200 V for AlN5 resulted with the disappearance of (002) peak and for AlN6 deposited at 250 V, again (100) plane became the only strong orientation. According to the literature, it is believed that AlN films deposited at 150 and 175 V bias voltages which have (002) orientation in their structure will have better piezoelectric properties.<sup>10,12,25,26,30,37</sup> For AlN films, the thermodynamically favourable plane is the (002) plane because it has the lowest surface energy and it is the most densely packed basal plane.<sup>38</sup> The intensive ion bombardment at 150 and 175 V bias voltages increased the kinetic energy and therefore enhanced the surface mobility of the atoms resulted in the formation of c-axis oriented (002) peak. The same phenomenon is observed by Yang *et al.*<sup>38</sup> while increasing the deposition temperature. In their case, the necessary energy is given to the atoms by the increase in the temperature, while in our case; ion bombardment is used without increasing the deposition temperature. The decrease in the intensity and the disappearance of (002) peak with further increase in the bias voltages is believed to be directly related to the crystalline lattice structure of AlN. Two kinds of Al–N bonds exist in wurtzite AlN, named B<sub>1</sub> and B<sub>2</sub>.<sup>9,38</sup> The (100) plane is composed only of B<sub>1</sub> bonds and (002) plane is composed both of B<sub>1</sub> and B<sub>2</sub> bonds. The nature of the B<sub>1</sub> bond is more covalent



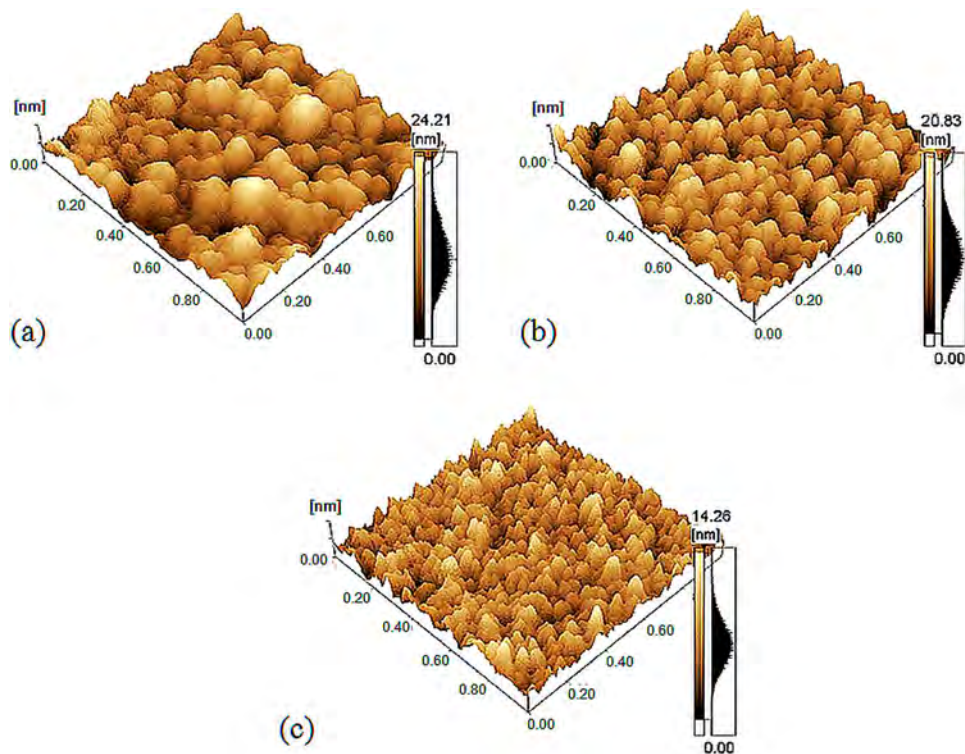
2 Effect of the bias voltages on the crystal orientation of AlN thin films a 0 V b 75 V c 150 V d 175 V e 200 V f 250 V

while that of the  $B_2$  is more ionic.<sup>38</sup> At floating potential and up to 150 V bias voltage, only  $B_1$  bonds can be observed and the films are strongly (100) plane oriented. (002) plane orientation with  $B_1$  and  $B_2$  bonds were observed at 150 and 175 V bias voltages with the increase in the kinetic energy and thus, surface mobility of the atoms. By further increasing the bias voltages, it is believed that  $B_2$  bonds of the Al–N which is weak compared to  $B_1$  bonds, dissociated by the excessive increase in the kinetic energies of the atoms, leading to the weakening of (002) peak and to the increase in (100) peak, as observed by Yang *et al.*<sup>38</sup> and Kuang *et al.*<sup>9</sup> while increasing the deposition temperature. The same approach can be considered for the appearance and the disappearance of (101), (102) and (103) peaks in Fig. 2. A few studies can be found in the literature attempted to deposit AlN films at room temperature; Kumari *et al.*<sup>16</sup> and Pessoa *et al.*<sup>39</sup> deposited AlN films by reactive DC magnetron sputtering at room temperature and no (002) orientation was observed in their studies, Moreira *et al.*<sup>30</sup> deposited AlN films by reactive DC magnetron sputtering at room temperature and according to the change in their deposition parameters, sputtering power and nitrogen flow, they found no (002) orientation and a mixture of (101) and (002) orientations and concluded with the fact that further arrangements and experiments should be done

to improve the atom mobility during the growth of the film, Wei *et al.*<sup>25</sup> deposited AlN films at room temperature by reactive RF sputtering, although their films had a mixture of (100) and (002) orientations a very large amount of oxygen up to 54.2% incorporated in the film structure.

### Surface morphological analyses

Surface morphology of deposited AlN films was investigated by AFM measurements. Evolution of the surface morphology, grain size and surface roughness of AlN films as a result of the change in the applied bias voltages has been determined. AFM images were taken in contact mode for all samples and the effect of bias voltage on the surface morphology is summarised in Fig. 3. As can be seen from Fig. 3a granular morphology with coarse grains has been achieved for AlN films deposited at floating potential. Increasing bias voltage decreased the grain size for AlN films deposited at 150 V bias voltage shown in Fig. 3b. A denser structure has been observed and grain size decreased apparently when bias voltage further increased for AlN films deposited at 250 V bias voltage as can be seen in Fig. 3c. According to AFM results, the maximum roughness ( $R_{max}$ ) decreased gradually from 24.21 to 20.83 nm and 14.26 nm whereas the root-mean-square roughness of the films decreased from



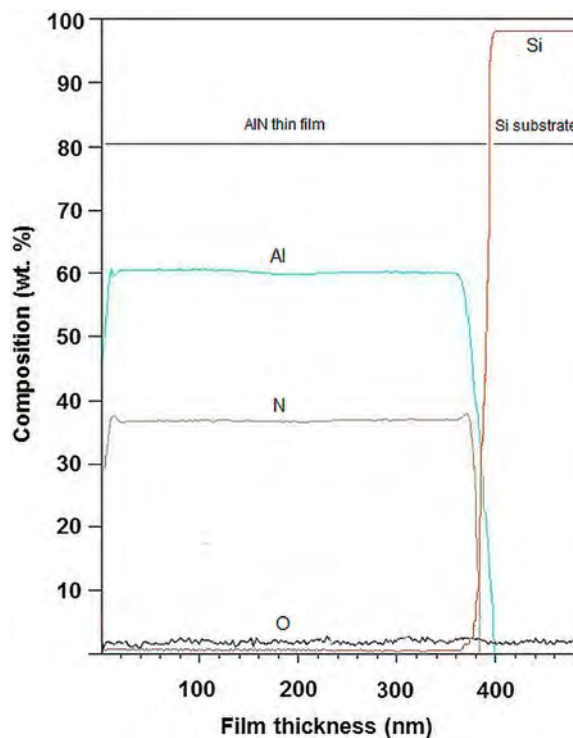
3 Three-dimensional AFM images of AlN films deposited at bias voltages of a 0 V b 150 V c 250 V

3.40 nm for AlN films deposited at floating potential to 2.82 nm for AlN film deposited at 150 V and to 1.90 nm for AlN film deposited at 250 V bias voltages respectively. We suppose that bias-induced extensive ion bombardment on the growing film by the plasma-enhanced configuration increased the surface mobility of the atoms and resulted with a denser and smoother film. When applying AlN films to SAW devices, its surface roughness has a critical impact on the quality of the device.<sup>9</sup> As the SAW is only propagated on the surface, all the energy is concentrated almost within the distance of a wavelength from the surface to the inside. Hence, the SAW is not able to pass through when the surface roughness is more than 30 nm.<sup>9</sup> The surface roughness as shown in Fig. 3 is less than 3.40 nm for all AlN films deposited in this study, which is much smaller than 30 nm to meet the requirement of SAW devices.<sup>9</sup>

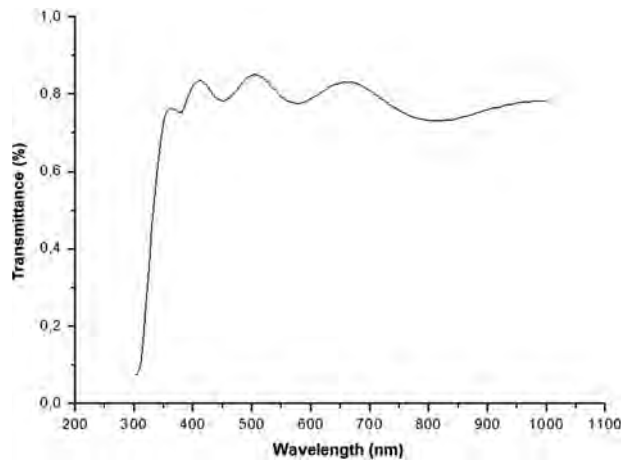
### Elemental concentration analyses

SIMS analyses were carried out for all the samples to determine the elemental concentration of the films over their thicknesses. All the analyses were realised on AlN films deposited on Si substrates. An  $O_2^+$  primary ion beam with 10 mm beam diameter was used at 15 keV acceleration voltage and a 200 nA primary beam current was used to scan a  $150 \times 150 \mu\text{m}$  area. For all the observations, the x axis was converted from time to thickness by measuring the depth of the craters obtained during analyses by a profilometer (Tester T 500eHommelwerke) and the y axis from counts/second to concentration (wt-%) by using a stoichiometric AlN as reference. Thus, for all the coatings deposited, the film thickness and the elemental composition were measured. Figure 4 demonstrates a representative SIMS concentration-thickness spectrum. All the films deposited have about 400 nm

thickness. Nearly stoichiometric AlN films with about  $60.2 \pm 2$  wt-% Al,  $37.3 \pm 2$  wt-% N and  $2.5 \pm 0.5$  wt-% O were deposited and the elemental film distribution is constant over the film thickness. The actual amount of O in the coating structure is believed to be lower than observed  $2.5 \pm 0.5$  wt-% as  $O_2^+$  primary ion beam should



4 Representative SIMS concentration-thickness profile of AlN films



5 Representative transmittance spectra of AlN films deposited

be used for the analyses because especially the detection of Al by SIMS necessitates positive secondary ions which enhanced by the use of  $O_2^+$  primary ion beam and the detected amount of O by using  $O_2^+$  primary ion beam is higher than the real value in the structure. It is also believed that the extensive ion bombardment on the growing film by the plasma-enhanced configuration, increased the surface mobility of the atoms and resulted with a denser film where O could not incorporate in excessive amounts in the structure.

### Optical analyses

Optical measurements were realised on AlN films deposited on glass substrates. Transmittance values of the coatings were elucidated by spectrophotometry in the wavelength ranges between 300 and 1000 nm. Figure 5 shows a representative spectrum of AlN films. The results demonstrated that the transmittance values of AlN films were not affected by the change in the bias voltages and about 80% of transmittance obtained for all AlN films deposited in this study in the UV and visible light range (380–750 nm), indicating that all the films were transparent.

### Conclusion

Nearly stoichiometric AlN films with about 400 nm thicknesses were successfully deposited by plasma-enhanced reactive DC magnetron sputtering of high-purity Al target in argon–nitrogen atmosphere. Following conclusions were drawn from our study;

- (i) Polycrystalline AlN films with hexagonal (wurtzite) structure were deposited on glass substrates, a mixture with variable relative amounts of mainly (100), (110), (101) and (002) orientations were observed according to grazing incidence XRD analyses.
- (ii) It is found that bias voltage has strong effect on the crystal orientation; c-axis (002) plane orientation has been achieved at 150 and 175 V bias voltages.
- (iii) AFM studies demonstrated that bias voltages have important effects on the microstructure and morphologies of AlN films; granular morphology with coarse grains has been observed for the film

deposited at floating potential which transformed to finer grains with the increase in the bias voltages.

- (iv) Roughness values decreased from 3.40 to 1.90 nm, smoother and denser films were obtained with the increase in the bias voltages.
- (v) About 80% of transmittance in the visible light range (380–750 nm) obtained for all AlN films deposited, indicating that all the films were transparent.
- (vi) It is concluded that c-axis oriented AlN films with very low surface roughness and high transparency could successfully be deposited by plasma-enhanced reactive DC magnetron sputtering configuration at room temperature for being used in microelectronic and optoelectronic applications.

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