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Fabrication of *w*-AlN Thin Films using Tilted Sputter Target and Unrotated Substrate Holder

Anis Suhaili Bakri[1], Nafarizal Nayan[2*], Mohd Khairul Ahmad[2], Soon Chin Fhong[2], Mohd Zainizan Sahdan[2], Ahmad Shuhaimi Abu Bakar[3], Wan Haliza Abdul Majid[3], Muliana Tahan[1], Nur Amaliyana Raship[4], Mohd Yazid Ahmad[5]

[1]Faculty of Electrical and Electronic Engineering, Universiti Tun Hussein Onn Malaysia (UTHM), 86400 Parit Raja, Batu Pahat, Johor, MALAYSIA

[2]Microelectronic and Nanotechnology- Shamsuddin Research Centre (MiNT-SRC), Universiti Tun Hussein Onn Malaysia (UTHM), 86400 Parit Raja, Batu Pahat, Johor, MALAYSIA

[3]Low Dimensional Materials Research Centre, Department of Physics, Faculty of Science, Universiti Malaya, 50603 Kuala Lumpur, MALAYSIA

[4]Department of Electrical and Electronic Engineering, National Defense University of Malaysia, 57000 Kem Sungai Besi Kuala Lumpur, MALAYSIA

[5]Nanorian Technologies Sdn Bhd, 40 Jln Kajang Perdana 3/2, Taman Kajang Perdana, 43000 Kajang, Selangor, MALAYSIA

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Abstract: Aluminium nitride (AlN) thin film is deposited by RF magnetron sputtering using Al sputtering target at room temperature. The sputter source was tilted 45° and the substrate holder was unrotated. The deposited AlN films were in the thickness range between 200 to 280 nm. The structural, elemental composition, morphological and topological properties of AlN with different thickness has been investigated. XRD analysis results revealed that all the AlN films deposited crystallize in hexagonal wurtzite phase (*w*-AlN). The crystal orientation of AlN (002) plane start to appear when the thickness of the AlN film increases and the thickest AlN film has a highest peak intensity of (002) plane with smallest FWHM indicated a good crystal quality of c-axis structure. The chemical composition of AlN analyze using EDS shows that all the films have AlN composition nearest to the stoichiometric and have a rice-like morphology regardless of the film thickness. The AFM analysis revealed that the surface roughness of the AlN films is increases along with the grain size as the thickness of the AlN films increased.

Keywords: c-axis AlN, hexagonal wurtzite phase AlN, substrate rotation, magnetron sputtering

1. Introduction

Aluminium nitride (AlN) is one of the III-nitride based semiconductor that have an imerging interest nowadays due to its attractive features such as direct wide band gap of 6.2 eV [1] and high thermal conductivity [2]. This wide band gap makes AlN is a suitable candidate for optoelectronic devices such as light emitting diodes. An ultraviolet range of 210 nm AlN light-emitting diode were successfully fabricated by Taniyasu and Kasu [3]. On the other hand, AlN also

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*Corresponding author: nafa@uthm.edu.my

useful for high surface acoustic wave (SAW) velocity [4]. This feature makes AlN is a promising materials for SAW devices by the use of piezoelectric materials as reported by Zaghloul and Piazza [5].

In addition, AlN also have been widely used as a nucleation layer for the growth of heteroepitaxial layer of GaN on the silicon wafer. This is due to that the large lattice mismatch between the GaN and silicon makes the presence of defects when GaN is directly grown on the silicon [6][7]. Generally, AlN is crystallize in hexagonal wurtzite (*w*-AlN) structure with P63mc space group as shown in Fig. 1 (a) [8]. It have several planes such as a-axis (100) (101) and c-axis (002) plane, where the latter is very important for AlN thin film applied for UV-LED and HEMT applications. Another crytalline AlN also can be found in cubic zincblende (z-AlN) structure as shown in Fig. 1 (b) [9].

AlN can be deposited using various techniques such as hydride vapour phase epitaxy (HVPE), metal organic chemical vapour deposition (MOCVD) or also known as metal oxide vapour phase epitaxy (MOVPE), metal beam epitaxy (MBE), hydride vapour phase epitaxy (HVPE) and sputtering. HVPE is a process where the crystallization of the III-nitride are formed when the reaction of the III-chloride and ammonia takes place [10]. MOCVD is the technique that use a high temperature to make a chemical reaction between reactant gases while MBE is a process where the atom or molecules are heated up so they are in the formed of gas to be fired on the heated substrate from the effusion cells in an ultrahigh vacuum environment [11].

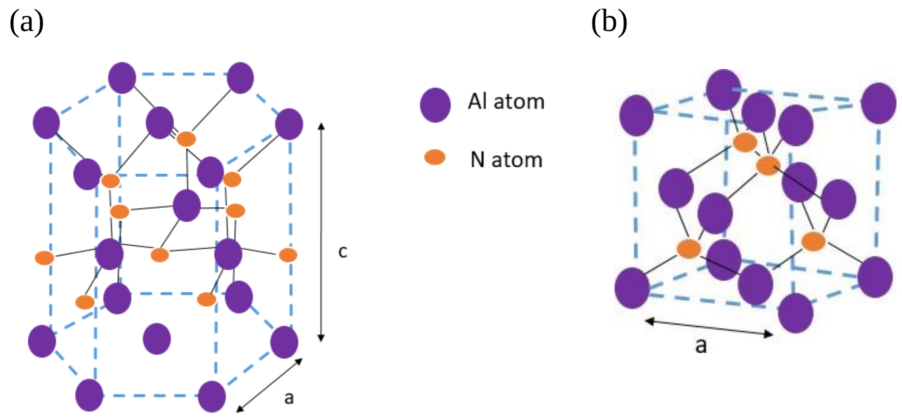


Fig. 1 - Crystal structure of (a) wurtzite; (b) zincblende AlN

In this paper, the AlN thin films were deposited via RF magnetron sputtering as this technique is a low cost technique with low deposition temperature, have good reproducibility and produce no toxic material. It is a process where the atom on the surface of the target is remove by ion bombardment in a high vacuum chamber [12] and deposited onto the substrate. One of the parameters that effect the properties of the film deposited is a thickness. Thus, in this study, the influence of the AlN film thickness towards its crystal structure, surface morphology, topology and elemental composition were presented.

2. Experimental details

AlN films were deposited using tilted sputter source, radio frequency (RF) power supply and unrotated stage holder. The silicon (100) substrate was placed on the stage as shown in Fig. 2. At first the stage was designed to be rotatable for uniform and homogenous thin film fabrication. In order to obtain several thickness of AlN thin film at one time, the stage was purposely unrotated. The AlN films were deposited without any external heating and the base pressure of the sputtering chamber was below than 1 x 10^{-6} Torr. The target to substrate distance was 5-inch and the gas mixture of Argon (Ar) and Nitrogen (N_2) mixture (100 sccm Ar + 50 sccm N_2) with the working pressure of 5 mTorr were controlled using the mass flow controller. The high purity target of Al (99.999 %) with 3-inch diameter was used as the target. The RF sputtering power was set to 200 W and the films were deposited with 2 hours deposition time.

Before the deposition, the silicon substrate was cleaned using the dilute hydrofluoric acid (HF) solution to remove the native oxide and the films were put into the chamber using the load lock system. After deposition, three points of the films were taken to be analyzed. To study the properties of the AlN films, ellipsometry, x-ray diffraction (XRD), field emission scanning electron microscope (FESEM) equipped with energy dispersive spectroscopy (EDS), JEOL JSM7600F and atomic force microscope (AFM) were used. The films thicknesses were measured using ellipsometry (Filmetrics, F20) while the crystal quality and growth direction of the AlN films were analyzed using the X-ray diffraction (XRD, Panalytical: Xpert3 Powder). The fixed diveregence slit (FDS) used was 1/4̊ with Cu-Kα radiation source at 40 mA and 40 kV. FESEM equipped with EDS was used to observe the surface morphology and estimate the elemental composition presence on the films. The surface topology of these samples were observed by AFM, Bruker to evaluate the surface roughness and grain size.

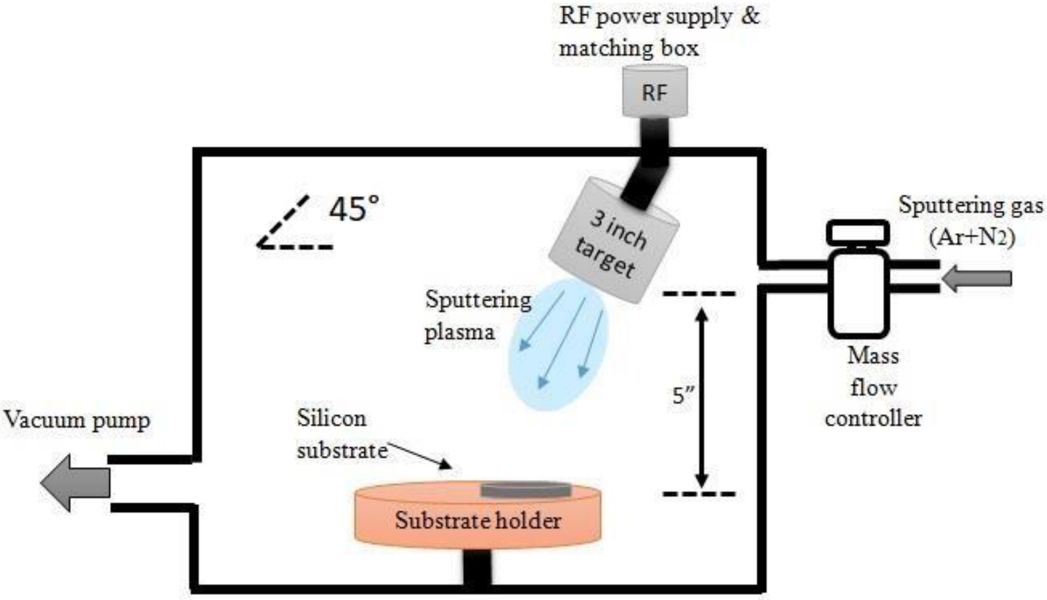


Fig. 2 - Schematic diagram of RF magnetron sputtering with 45° tilted sputter target

3. Results and Discussion

Fig. 3 shows the photo image of AlN films deposited on the Si (100) substrates without any rotation given to the substrate holder during deposition. It is clearly understand from the color that the AlN thin film is not uniform. Different colour gives different thickness of the AlN films. In order to analyze the films, three main points were taken. The thickness was measured using ellipsometry technique. Fig. 4 shows the snapshot of ellipsometry measurement where the goodness fitting was above 99%. The thickness measurement was repeated three times for each point and the average of the film thickness was calculated. Fig. 5 shows the AlN films with the thickness of 201 nm, 235 nm and 280 nm for point 1, 2 and 3, respectively.

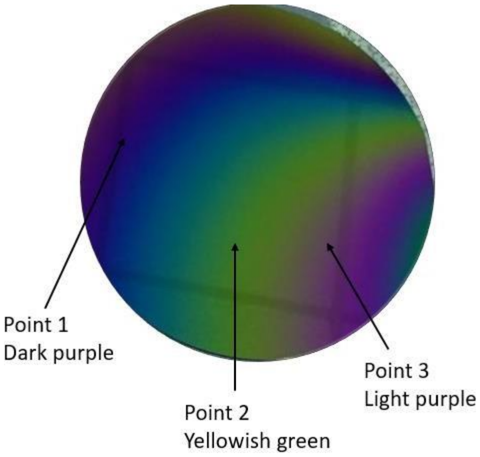
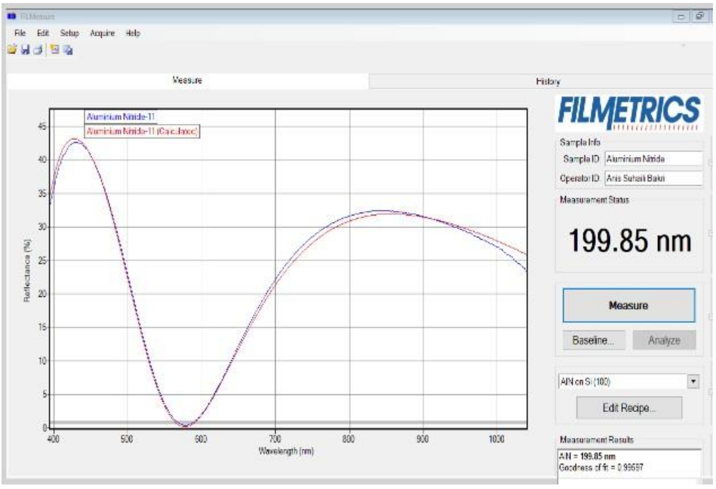


Fig. 3 - AlN films deposited using sputtering without any substrate rotation



Sample Info
Sample ID: Aluminium Nitride
Operator ID: Aris Suhaili Bakri
Measurement Status
199.85 nm
AlN on Si (100)
Measurement Results
AlN = 199.85 nm
Goodness of fit = 0.99697

Fig. 4 - Snapshot of AlN thin film thickness measured by ellipsometry at point 1. The goodness of fitting is above 99

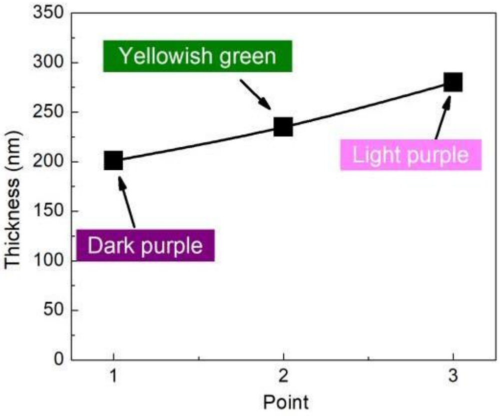


Fig. 5 - Average thickness of deposited AlN thin films at different point

3.1 Structural properties

In this paper, the study is to focus on the crystal quality of (002) plane/c -axis of AlN films at 2 theta of 36.0. The presence of the (002) plane is desired in most applications due to its properties [13] and the highly crystalline of c-axis AlN films is recognized to have a good performance when applied to the electronic devices [14]. Fig. 6 (a) shows XRD patterns of AlN films deposited with different thickness. From the XRD results, all the samples exhibit AlN-wurtzite type with hexagonal crystal structure (P63mc). The peaks of 2 theta 51-56° is observed in all samples indicate the silicon substrate. From the XRD pattern, it can be observed that the AlN films is a polycrystalline film with mix of AlN crystal planes. The peaks present at 2 theta of 32, 36, 37, 49, 58, 65, 69, 70, 72° were belong to AlN (100), (002), (101), (102), (110), (103), (200), (112) and (201) planes respectively. All peaks match well with Bragg reflections of the standard aluminium nitride structure Inorganic Crystal Structure Database (ICSD) file no 98-060-2459. The observed dominant peak in this XRD pattern was (100) plane. This is due to that the formation of the (100) plane need lower energy compared to the (002) plane [15].

Fig. 6 (b) shows the zoom-in patterns of the (002) plane. From the figure, it can be observed that the intensity of c-axis peaks of AlN corresponding to (002) planes is increasing as the thickness of the AlN films increases. This may be due to that at higher film thickness, the energy is enough to nucleate the (002) planes [13]. The properties of the structural (002) planes were presented in Table 1. The FWHM of (002) planes were measured using X'pert highscore software analysis while the crystallite size were calculated from the Scherrer's equation [16]

$$D = \frac{0.9\lambda}{\beta\cos\theta} \tag{1}$$

where D is the crystallite size, λ is the wavelength of x-ray radiation (0.154060 nm) and β is the full width at the half maximum (FWHM) of the observed peak. From the calculation, the thickest AlN film has biggest crystallite size due to its smallest FWHM. The smallest FWHM and the highest intensity of the (002) plane shows that the film with the thickest film has a good c-axis crystal structure. Furthermore, the microstrain value of the thickest film also shows the smallest value, indicating the presence of low dislocation density [17].

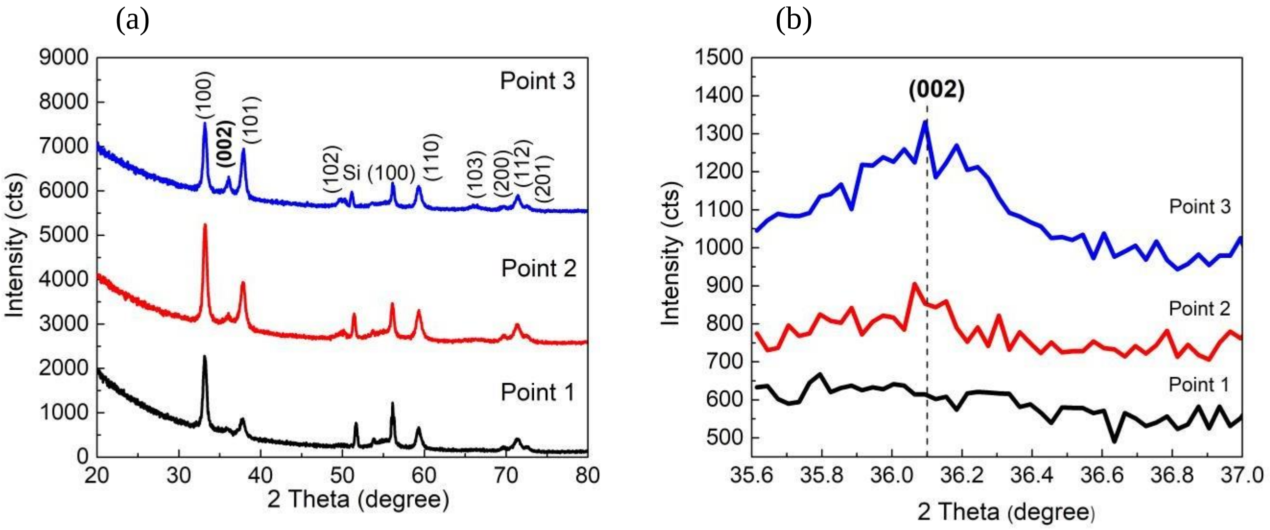


Fig. 6 - XRD pattern of the (a) AlN films (b) AlN (002) plane as a function of different point

Table 1 - Structural parameters calculated from XRD data as a function of different point

Samples	Intensity (cts)	Position 2 Theta (deg)	FWHM (deg)	Crystallite size (nm)	Microstrain (%)	Plane orientation (hkl)
Point 1	6.03	35.99	-	-	-	(002)
Point 2	60.63	36.02	0.4723	17.69	0.62	(002)
Point 3	192.34	36.11	0.3542	23.59	0.46	(002)

3.2 Surface morphology

The surface morphology analyzed using FESEM are presented in Fig. 7. From the FESEM images, it can be observed that all the AlN films have rice-like structure. All the AlN films show almost the similar morphology regardless of its thickness. This results show that the thickness does not effect on the formation of the AlN structure.

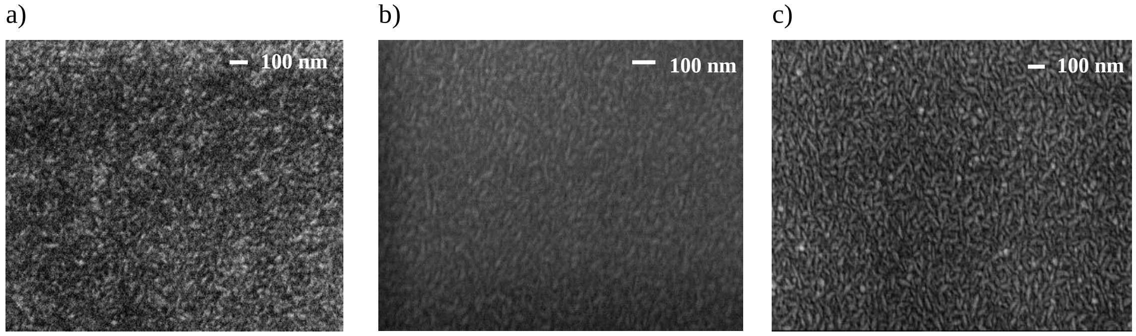


Fig. 7 - Surface morphology of AlN films (a) Point 1; (b) Point 2; (c) Point 3

The elemental composition of AlN calculated using the atomic percentage were presented in Table 2. From the calculation, all the films have the elemental composition close to the stoichiometric of AlN. The result shows that the different thickness of the film does not give effect on the composition of the AlN films.

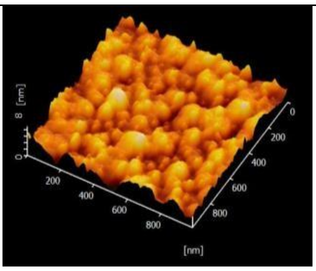
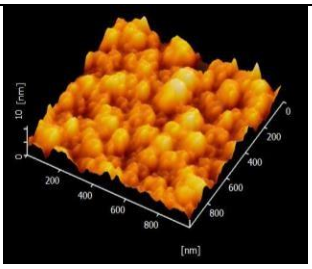
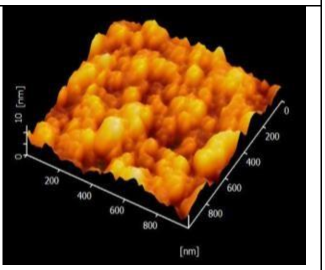
Table 2 - Chemical composition (at% of Al and N) as a function of different point

Samples	Atomic (%)		Al : N
	Al	N	
Point 1	39.22	60.78	1 : 1.55
Point 2	40.29	59.71	1 : 1.48
Point 3	39.88	60.12	1 : 1.51

3.3 Surface topology

The AFM analysis were done to study further on the effect of different thickness of AlN films on the surface topology. The details of the AFM analysis are presented in Table 3. From the analysis, the average roughness of the AlN films were increases as the thickness increases. The grain size of the AlN films were also increases. Therefore, thicker AlN thin film results in larger grain size and surface roughness.

Table 3 - The AFM images, average roughness and grain size of AlN thin films

Sample	**Point 1**	**Point 2**	**Point 3**
AFM 3-Dimensional Image			
Average roughness (nm)	**0.90**	**1.16**	**1.48**
Grain size (nm)	**65.63**	**72.78**	**114.20**

4. Summary

AlN films were succesfully fabricated by RF magnetron sputtering at room temperature on Si (100) substrates. The crystal structure, surface morphology, topology and elemental composition of different thickness of AlN were studied. XRD analysis of AlN with different thickness revealed that the films have hexagonal wurtzite phase crystal structure. The XRD results also show that the peak of AlN (002) plane increased as the thickness increased. Apart from that, the observation from the XRD pattern also show that the thinnest film of about 200 nm, AlN (002) plane was absent. However, the crystal quality of the (002) plane increases with thicker film. Results from the FESEM images show that all the AlN films have rice-like morphology regardless of its thickness while the elemental composition revealed that all the films composition of Al and N were close to the stoichiometry. These findings show that, the thickness of the film does not affect much on the surface morphology and elemental composition of the film. The average roughness was increased as well as the grain size as the thickness of AlN film increased.

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