Sputtering of aluminium nitride (002) film on cubic silicon carbide on silicon (100) substrate: influences of substrate temperature and deposition power

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Abstract

We perform pulsed DC sputtering of aluminium nitride (002) thin films on top of cubic silicon carbide-on-silicon (100) substrates at different substrate temperatures and deposition powers. The films are characterised using the following parameters: FWHM of diffraction peak, FWHM of the rocking curve, residual stress, thickness, deposition rate, grain size, and surface roughness. The overall quality of the films improve at escalated temperature and power. However, they have hillocks on the surface, which is caused by high amount of tensile stress.

Keywords: aluminium nitride; silicon carbide; sputtering; substrate temperature; deposition power

1. Introduction

Aluminium Nitride (AlN) has great potential for use in power electronics because of its wide bandgap (6.2 eV), high thermal conductivity ($3.3 \text{ WK}^{-1}\text{cm}^{-1}$), and high electrical resistivity ($10^{15} \text{ }\Omega\cdot\text{cm}$) [1-3]. It could also be used to fabricate RF MEMS, piezoelectric sensors and actuators because of high acoustic velocity (12,000 m/s) [4, 5]. In comparison to other

piezoelectric materials such as zinc oxide (ZnO) and lead zirconate titanate (PZT), AlN is preferred for high-temperature applications in the automotive, aerospace, and energy industries because of its high Curie temperature (1200 °C) [6], whereas performance of ZnO and PZT degrade significantly [6, 7]. The piezoelectric properties of AlN largely depend on its crystallographic orientation. C-axis oriented AlN i.e. (002) plane has higher piezoelectric property d_{33} (5.0961 pm/V) compared to the other crystal orientations [8, 9]. Also, AlN electromechanical coupling coefficient (kr^2) is closely related to the film's orientation [10]. Based on the above motivations, deposition of AlN (002) had been investigated by many researchers using fabrication techniques such as molecular beam epitaxy, chemical vapor deposition, reactive sputtering, and pulsed laser deposition. Among these, sputtering is preferred for its low cost and low temperature process. Recently, the authors of this paper published a review paper that summarizes reactive sputtering of AlN (002) films [11] from over 80 articles, of which, many groups investigated the influence of sputtering parameters. They observed the effect of power, gas ratio, process pressure, substrate temperature etc. on the orientation and quality of the films [11, 12].

The kinetic energy of the adatoms on the surface of the deposited films is proportional to the deposition power. Specific amount of energy is needed to tune the mobility of the adatoms to arrange themselves in (002) orientation. Several groups have documented this influence. For example, Kusaka et al. [13] observed the increase in the grain size and the enhancement of c-axis oriented AlN films deposited on glass substrate with increasing DC power. Similarly, Vashaei et al. [14] noted an improvement in the growth rate and crystal quality of AlN (002) films deposited on C-sapphire substrate with increasing RF power. More importantly, both groups reported the adverse effect of exceeding the optimum value of sputtering power. At this point, the role of

secondary adatoms has become dominant. These high-energy atoms attack substrate surface, causing morphological damage and deterioration in the crystal quality of the film [13].

In addition to the sputtering power, the kinetic energy and the mobility of the adatoms could also be controlled by the substrate temperature [15, 16]. However, further increment after the optimum value deteriorates the quality of the films due to the thermal stresses and absorption of the impurities into the deposited film [10]. Several research groups investigated the influence of the substrate temperature on their AlN films. Kuang et al. [9] reported the optimum temperature of 300 °C to produce the highly c-axis oriented AlN films on top of the sapphire substrate. Hao et al. [15] studied the role of substrate temperature in the range of 60 °C to 520 °C on the deposition of the AlN film on the Si (100) substrates. They reported 430 °C as the optimal substrate temperature, achieving FWHM of a rocking curve value of 2.25° for the AlN (002) films.

The sputtered films have both the extrinsic and the intrinsic stresses. The extrinsic (or thermal) stress results from the difference in the coefficient of thermal expansion (CTE) between the substrate and the deposited film, which is induced by the deposition temperature. The intrinsic stress arises from the lattice mismatch and the structural ordering processes that occurs during the film growth [17]. Kusaka et al. [13] studied the influence of the sputtering power to the residual stress of the AlN films on the glass substrate using the DC magnetron sputtering. They observed a tensile stress at a low input power, which transformed to a compressive stresses at a high power. Zhong et al. [18] investigated the effect of sputtering parameters on the residual stress of the AlN films deposited on the Si (111) substrates using the RF sputtering. They observed that the stress changed from the compressive to tensile with a decrease in N₂

concentration and an increase in the sputtering pressure. In contrary to Kusaka et al. however, the substrate temperature and the RF power have a minimum effect.

One method to reduce the residual stress of the AlN films is by reducing the CTE difference between the film and the substrate. AlN is typically deposited on the Si substrates due to a lower cost per surface unit area and the latter's compatibility with the CMOS process. However, the difference in CTE between both materials is 47 %. As an alternative, a cubic silicon carbide (3C-SiC) film has been epitaxially grown on a Si substrate as a buffer layer. This solution manages to reduce the lattice mismatch and the CTE difference between both AlN (002) and 3C-SiC (100) films to 28.6% and 18%, respectively. The most well-known work on AlN (002)/3C-SiC (100) structure was by Lin et al. [19] in 2010. They employed a commercial 3C-SiC/Si (100) substrate from NOVASiC. In order to achieve a high quality crystal with the full width half maximum (FWHM) rocking curve of 1.73° , they performed a two-step deposition processes. First, a 50 nm seed layer was sputtered using a 3 kW power at the substrate temperature of 450°C . Then, the remaining 1 μm thick film was produced at the deposition power of 5.5 kW and the substrate temperature of 350°C . Since 2013, our group attempted to simplify the process using a lower deposition power, using 3C-SiC/Si substrates that we custom-fabricated at Queensland Micro- and Nano-technology Centre of Griffith University. In the first work in 2014, we reported the deposition of polycrystal AlN thin films with (002), (100) and (101) orientations [20]. In 2016, we were able to control the AlN's orientation by tuning the nitrogen/argon ratio [21]. We produced (002) and (100) orientations at low and high nitrogen concentrations, respectively. Although we did not manage to produce highly c-axis AlN film at that time, we gained a lot of lessons from those works.

In this latest work, we performed the parametric studies on the AlN (002) films by varying the substrate temperature from 250 to 450⁰ C and the deposition power from 1200 to 2400 W. To the best of our knowledge, the influences of both parameters on the 3C-SiC/Si (100) substrate have not been reported in the literature [11]. In all the experiments, we achieved the highly c-axis AlN films with the FWHM rocking curve ranging from 3.38⁰ down to 2.12⁰. Based on the collected data, we elucidated the role of sputtering power and substrate temperature on the film's crystal quality, morphology, deposition rate, and residual stress. Finally, we propose a growth model as the function of adatom's kinetic energy and surface mobility to explain these results.

2. Experimental Details:

Prior to the AlN sputtering, a 80 nm thick 3C-SiC film was epitaxially grown on top of an on-axis Si (100) wafer [22], which was 150 mm in diameter using an epitaxial reactor (SPT Microtechnologies, San Jose, CA, USA). The surface roughness of the 3C-SiC (100) film was measured to be about 2 nm (rms). The 3C-SiC/Si wafer was cut to the size of 15 mm by 15 mm samples using a Disco dicing saw (Disco Corp., Tokyo, Japan). The samples were then cleaned using a standard piranha process to remove the organic substances, followed by submersion in a diluted 1% HF acid for 5 minutes to remove the oxides.

The pulsed DC sputtering of the AlN thin films on top of the 3C-SiC-on-Si (100) substrates was performed using a magnetron sputtering system (Gamma 1000, Surrey Nano Systems, Newhaven, UK). The system as shown in Figure 1 consists of a high vacuum chamber with an ultimate base pressure of 10⁻⁹ Torr, 4 x 4 inch sputtering targets, shutters, gas feeds, and

susceptor that can be heated and RF biased. The types of deposition power are either DC, pulsed DC or RF. Prior to 2017, we found from the previous deposition works [23, 24] that the nitrogen (N_2) gas formed a coating on the target that would lead to the arcing and instabilities in the DC power. Starting in 2017, pulsed DC power supply (Pinnacle Plus, Advanced Energy, Fort Collins, USA) was utilized at 250 kHz with the time off of 0.4 μs to prevent the electrical arcing. The negative bias was applied to the aluminum (Al) target, which served as the cathode, while the positive bias was applied to the susceptor (substrate), which acted as the anode. The pulsed DC voltage ionized the argon (Ar) atoms into a plasma state. The Ar^- ions accelerated to the positively-charged susceptor, while the Ar^+ ions accelerated and collided with the negatively-biased Al target. As a result of the collision, the Al atoms were ejected out from the target's surface and get ionized (Al^+ and Al^-). Then, the N_2 gas was introduced to the chamber and get ionized (N_2^+ , N_2^-) by the pulsating voltage. Under Poison condition, they reacted with the Al ions to form AlN. The negatively-charged AlN molecules were attracted towards the positively-charged susceptor. They eventually changed state from plasma to solid (called adatoms) on the surface of the 3C-SiC/Si (100) substrate.

Figure 1: Illustration of the reactive sputtering process inside the chamber to produce AlN film

We employed an Al target of 100 mm in diameter with 99.999 % purity, while the target to substrate spacing was set to 20 cm. Prior to the sputtering, an in-situ RF reactive ion etching was carried out on the 3C-SiC/Si substrate to aid adhesion to the sputtered AlN film. All the sputtering was performed at a process pressure of 2 mTorr and 58 % of N_2/Ar ratio in reactive mode, at a total flow rate of 60 sccm. This was determined as the “poison” mode [21].

In the first part of the experiment, we increased the substrate temperature from 250 °C to 450 °C in steps of 50 °C, at the sputtering power of 1800 W, while keeping all other sputtering parameters constant. In the second part of the experiment, we set the substrate temperature at 350 °C, while increasing the sputtering power from 1200 W to 2400 W in step of 300 W, while

keeping all other sputtering parameters constant. The values and ranges of the substrate temperature and deposition power were selected based on the previous works [11].

The AlN films were characterized by the following parameters and equipment. The X-ray diffraction (XRD) 2theta-omega and omega rocking curve measurements were performed using Empyrean (Malvern Panalytical Ltd., Royston, UK). This equipment used monochromatized Cu K α 1 beam with $\lambda=1.5405980\text{\AA}$ and a fixed anti-scatter slit with PIXcel-3D detector. The detector was in “scanning mode” for 2theta-omega scan, and was in “open mode” for rocking curve scan. We measured surface roughness using NX20 AFM (Park Systems, Suwon, Korea), deposition rate using Nanospec AFT 210 (Nanometrics Inc., Milpitas, CA, USA), residual stress using Flexus 2320 system (KLA-Tencor Corp, Milpitas, CA, USA), and cross-sectional SEM using Raith150 SEM and E-Beam Litography System (Raith GmbH, Dortmund, Germany).

3. Results and discussions:

Table I. Characterization results for AlN films

Sample ID	Substrate temperature (°C)	Sputtering power (W)	Peaks position (36° <002>)*	FWHM of diffraction peak (°)	FWHM of rocking curve (°)	Thickness (nm)	Deposition rate (nm/min)	Stress (tensile) (MPa)***	Grain size (nm)**	Surface roughness (nm)
X1	250	1800	36.18	0.18	3.38	665	15.1	380.1	48.51	3.40
X2	300	1800	36.18	0.18	2.85	680	15.4	313.6	48.5	3.109
X3	350	1800	36.16	0.13	2.37	657	14.9	322.6	67.1	2.899
X4	400	1800	36.19	0.12	2.38	686	15.6	315	72.7	2.57
X5	450	1800	35.6	0.11	2.12	680	15.4	308	79.3	2.67
X6	350	1200	36.17	0.11	2.6	620	10.3	432.7	79.3	3.99
X7	350	1500	36.18	0.15	2.7	623	12.4	333.5	58.2	3.7
X3	350	1800	36.16	0.13	2.37	657	14.9	322.6	67.1	2.89
X8	350	2100	36.19	0.18	2.51	620	15.9	300	48.2	2.30
X9	350	2400	35.7	0.15	2.22	504	18	230	58.2	2.63

*The observed XRD peaks can be assigned to wurtzite AlN phase (Card # 01-070-2545) with AlN (002) peak at $2\theta = 36.023^\circ$ [25]

** The grain size is calculated using the Scherrer equation.

*** Residual stresses are measured without applying RF bias voltage on the substrate.

Table I summarizes the characterization results for all films. It represents two set of experiments. The top half shows the characterized samples (X1, X2, X3, X4, X5) as a function of the substrate temperature. The bottom half of Table I shows the characterized samples (X3, X6, X7, X8, X9) as a function of the deposition power. If one glances at the data, the overall quality of the films improve with increasing substrate temperature and deposition power, in agreement to many previous works that sputtered AlN (002) on different substrates [11].

Figure 2(a) shows the 2theta-omega XRD scan (sometimes referred to as the normal angle XRD scan) of the samples that were deposited at substrate temperatures of 250 °C to 450 °C. Two major diffraction peaks are observed at 36.2° and 69.2° that correspond to AlN (002) and Si (400) crystal orientations, respectively. One minor diffraction peak can be seen at 41.5° for SiC (200) orientation. The values of FWHM of the diffraction peak for (002) orientation is extracted using a Gaussian fitting, as shown in Table I. Figure 2 (b) shows the FWHM of the

rocking curve plot for (002) orientation. The rocking curve values are recorded in Table I. In general, both FWHM parameters indicate improvement in the crystal quality of the AlN (002) film at increasing substrate temperature. This trend is in agreement with Kusaka et al. [13] and Vashaei et al. [14], which sputtered AlN on glass and Si (111) substrates, respectively.

FIG. 2(a) 2theta-omega XRD scan of AlN film on 3C-SiC/Si (100) substrate at five substrate temperatures.

FIG. 2(b) Rocking curve measurement of AlN film on 3C-SiC/Si (100) substrate at five substrate temperatures.

The 2theta-omega XRD scan of the samples that were prepared at five deposition powers are shown in Figure 3 (a). The XRD plot is very similar to Figure 2 (a). Figure 3 (b) shows the FWHM of the rocking curve plot for (002) orientation. The values of the FWHM of diffraction peak and FWHM of rocking curve as a function of deposition power are provided in Table 1. There is no recognizable trend for FWHM of diffraction peak. However, FWHM of rocking curves indicate improvement in the crystal quality of the AlN (002) film at increasing deposition power. This trend is in agreement with Kuang et al. [9] and Hao et al. [15], which sputtered AlN

films on sapphire and Si (100) substrates, respectively. In contrast to their results however, the smallest value of 2.22° at 2400 W indicates that the optimized deposition power has not been reached in this work.

FIG. 3 (a) 2theta-omega XRD scan of AlN film on 3C-SiC/Si (100) substrate at five deposition powers.

Fig. 3 (b) Rocking curve measurement of AlN film on 3C-SiC/Si (100) substrate at five deposition powers.

The thicknesses of all AlN films were measured using Nanospec AFT 210. We used the wavelength of 632 nm and assumed the theoretical value of 2.14 for the AlN refractive index. In order to accurately measure the reflection between the AlN film and the Si substrate without interference from the 3C-SiC buffer layer, we had to separately deposit AlN films on top of the p-type doped Si (100) wafers. These AlN/Si (100) samples were batch-sputtered with the ones that were sputtered on top of 3C-SiC/Si (100) substrates. The data for the thickness of the sputtered AlN films are provided in Table I. For samples X1, X2, X3, X4 and X5, all depositions

lasted for 44 minutes, resulting in AlN thicknesses between 657 nm to 686 nm. For samples X6, X7, X8 and X9, the sputtering time was between 28 to 44 minutes, resulting in thicknesses between 504 nm to 657 nm. We need the thickness data and deposition time to calculate the deposition rate.

Sputtering power plays a major role in the deposition rate. It is evident from Table 1 that the latter goes up with an increase in deposition power. According to Kusaka et al. [13], this is due to an increase in Al atoms being sputtered from the target to the substrate. It is important to note that the upward trend could not been observed for substrate temperature. On average, the deposition rate hovers around 15.3 nm/min as the temperature rose from 250 °C to 450 °C.

In order to measure the residual stress, AlN thin films were sputtered on top of full wafer at different substrate temperatures and deposition powers. A Tencor Flexus 2320 system measured the change in the wafer's curvature before and after the AlN film deposition. This value is then inserted into Stoney's equation to calculate the stress (σ) using the following equation:

$$\sigma = \frac{E}{6(1-\nu)} \frac{t_s^2}{t_f} \left(\frac{1}{r_c} - \frac{1}{r_u} \right) \quad (1)$$

where E, ν , t_s are the Young's modulus, Poisson's ratio, thickness of the substrate (wafer), respectively. t_f is the thickness of the film, and r_u and r_c are the radii of curvature of the substrate before and after film's deposition, respectively.

The values of measured stress for all samples are recorded in Table I. The theory states that the residual stress of AlN film increased with escalating substrate temperature due to CTE difference [17]. For example, Iriarte et al. reported that trend for AlN film on top of Si (100). We

observed an inverse trend on top of 3C-SiC (100) substrate. In comparison with that work, ours have lower CTE differences. Furthermore, the FWHM data from Table I indicate that we have not reached optimized temperature. Chason et al. [26] offered plausible explanation for the inversing trend. In their theory, the non-equilibrium nature of the surface during the sputtering could drive the adatoms into the grain boundary, which in turn induces compressive (negative) stress. As we escalate the substrate temperature, the surface mobility of the adatoms increases as well. The ones that impinge the grain boundary would ultimately decrease the residual stress further. The same theory could be applied to explain the inverse relationship between stress and deposition power.

Figure 4 shows the surface of AlN films at five substrate temperatures. All figures have the same amplification factor to enable fair comparison. The presence of hillocks i.e. rough surface structure make it a bit harder for the readers to see the grain patterns. It could be seen that the grains for all samples have pebble shape. If one compares Figure 4 (a) to 9e) closely, the grain size is getting bigger at higher substrate temperature. In order to support this qualitative assessment, we use Scherrer equation to calculate the grain size from the XRD data, as provided in Table 1. Fang et al. observed the same trend when they annealed ZnO film [27]. They explained that as temperature increased, small crystals coalesce, resulting in bigger grain size.

It is noteworthy to investigate the reason for the existence of these hillocks. From literature, Lee et al. [28] reported similar condition on the surface of their AlN film on top of Si (100) substrate. Only on film with tensile stress of 0.67 GPa possessed hillocks (readers are referred to Figure 4 in that article). The other three films at compressive stress of -1.1 GPa and -0.75 GPa, and at tensile stress of 0.03 do not have hillocks. Thornton and Hoffman [29] pioneered the study of stress in thin film in 1989. They state a relationship between adatoms'

surface mobility and stress i.e. low mobility contribute to higher stress. As the substrate temperature goes up, the stress decreases. The tensile stress data from Table I corroborate this theory nicely. In addition, it could be seen from Figure 4 that there are less number of hillocks at higher substrate temperature i.e. higher surface mobility.

FIG. 4 SEM image of AlN film at different substrate temperatures: (a) sample X1 at 250 °C (b) sample X2 at 300 °C (c) sample X3 at 350 °C (d) sample X4 at 400 °C (e) sample X5 at 450 °C

The surface roughness of AlN films was measured in a $5\ \mu\text{m} \times 5\ \mu\text{m}$ area, using contact mode Atomic Force Microscopy with a silicon cantilever. Figure 5 shows the AFM images at five substrate temperatures. The data (in root mean square) are recorded in Table 1. In essence, the surface of AlN film becomes smoother at higher substrate temperature. It is quite obvious

that the hillocks contribute to rougher surface. As their presence decreases at higher substrate temperature, the surface becomes smoother.

FIG. 5 AFM image of AlN film at different substrate temperatures: (a) sample X1 at 250 °C (b) sample X2 at 300 °C (c) sample X3 at 350 °C (d) sample X4 at 400 °C (e) sample X5 at 450 °C

Figure 6 shows the surface of AlN film at five deposition powers. The surface is very similar to Figure 4 i.e. pebble shape grains with the presence of hillocks. If one performs a closer comparison between both figures, there are two subtle differences. First, there are lesser number of hillocks at higher deposition power. In other words, the influence of deposition power to surface roughness is bigger than substrate temperature. Second, we could not see a trend in term

of the size of the pebble-shaped grains, as shown in Table 1. Our observation is in contrast with the trend that is reported in literature. Liu [30] and Kee et al. [31] found that the grain size increases at higher sputtering power. They contribute it to higher deposition rate of the film.

FIG. 6 SEM image of AlN film at different sputtering powers: (a) sample X6 at 1200 W (b) sample X7 at 1500 W (c) sample X3 at 1800 W (d) sample X8 at 2100 W (e) sample X9 at 2400 W

Figure 7 shows the AFM image of the AlN film at different deposition powers. We follow the same scan setting as in Figure 5, and the surface roughness is recorded in Table I. Similar trend is observed i.e. the film becomes smoother due to decreasing number of hillocks, as illustrated in Figures 6 and 7. The lowest surface roughness of 2.3 nm amongst all samples is recorded at deposition power of 2100 W.

FIG. 7 AFM image of AlN film at different sputtering powers: (a) sample X6 at 1200 W (b) sample X7 at 1500 W (c) sample X3 at 1800 W (d) sample X8 at 2100 W (e) sample X9 at 2400 W

In order to provide additional evidence on the successful deposition of c-axis AlN film, we randomly pick sample X2 and took its cross-sectional image using Raith150 System. Figure 8 clearly differentiates the Si substrate, SiC film, and most importantly AlN film. The columnar growth of the latter, perpendicular to the substrate attests to successful fabrication along c-axis. We also took the opportunity to measure the thickness of AlN film. The value of 690 nm is very close to the one recorded in Table I i.e. 680 nm being obtained using Nanospec AFT 210.

Fig.8 Cross-sectional SEM image of sample X2 at 50,000 magnification factor.

Figure 9 show the simple growth model, which is based on the flow chart that we originally propose in our review paper [11]. Fig 9 (a)-(c) illustrate the first scenario. At low deposition power and substrate temperature, the adatoms have low kinetic energy. Hence, they do not have sufficient surface mobility to fill all available vacancy along the c-axis. As a result, the AlN films has lower crystal quality, higher surface roughness, and higher tensile stress. Fig 9 (d)-(f) illustrate the opposite scenario.

Fig 9 (a) to (c): Growth model at lower substrate temperature and/or deposition power.

Fig 9 (d) to (f): Growth model at higher substrate temperature and/or deposition power.

4. Conclusion

This paper presents the pulsed DC sputtering of c-axis oriented AlN films on 3C-SiC/Si (100) substrates. We investigate the effect of substrate temperature and deposition power on the crystal orientation, deposition rate, film's stress, and film's morphology. Our observations are listed herein. First, the crystal quality of the AlN (002) film improves with an increase in the substrate temperature and the sputtering power. This trend has been reported widely in the literature. Second, the film's residual stress decreases with the escalating temperature and power,

which is in contrary to the trend being in literature. We offer alternative theory from Chason et al. [26] to explain our result. Third, the deposition rate increases with sputtering power, but remains constant at increasing temperature. The former is widely reported in the literature [11]. Fourth, there are presence of hillocks on the surface, which was also reported by Lee et al. [28], and is credited to tensile stress by Thornton and Hoffman [29]. Our residual stress data support corroborate that theory. Fifth, the grain size increases at higher substrate temperature. This is credited to coalescence of smaller grains, as observed by Fang et al. [27] when they annealed ZnO film. Sixth, the film's top morphology becomes smoother at increasing temperature and power as the hillocks disappear. Seventh, a simple growth model is proposed to explain the dependant of film's quality to substrate temperature and deposition power. Finally and more importantly, it should be emphasized that despite the presence of hillocks on the surface, we have produce a highly c-axis AlN film, albeit at high tensile stress. This statement is supported by the FWHM of rocking curve data, as well as cross-sectional SEM image of one of the samples.

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