Quality-enhanced AlN epitaxial films grown on Al substrates by two-step growth

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Abstract: Quality-enhanced AlN epitaxial films have been grown on Al substrates by pulsed laser deposition with the two-step growth by the combination of low-temperature (LT) and high-temperature (HT) growth. The effect of HT growth temperature on the interfacial property, surface morphology and crystalline quality of as-grown AlN epitaxial films is studied in detail. It is found that as the HT growth temperature increases from 450 to 650 ºC, the AlN/Al hetero-interfaces of ~300 nm-thick AlN epitaxial films keep sharp and clear, and the surface morphology and crystalline quality of ~300 nm-thick AlN epitaxial films are improved gradually. Especially, the ~300 nm-thick AlN epitaxial films grown at HT growth temperature of 650 ºC show sharp and abrupt AlN/Al hetero-interfaces, very smooth surface with the root-mean-square surface roughness of 1.1 nm, and high crystalline quality with the full-width at half-maximums for AlN(0002) and AlN($10\bar{1}2$) X-ray rocking curves of 0.45° and 0.72°, respectively. The quality-enhanced AlN epitaxial films on Al substrates are of paramount importance for the fabrication of highly-efficient AlN-based devices.

Keywords: pulsed laser deposition; two-step growth; interfacial property; surface morphology; crystalline quality.

1. Introduction

Group III-nitride semiconductor materials, such as AlN, GaN, InN, *etc.*, are excellent semiconductors, with unique properties very suitable for the application of micro-electronic and optoelectronic devices [1-3]. Among these group III-nitride semiconductors, AlN has direct band, high thermal conductivity, high surface acoustic wave (SAW) velocity, *etc* [4-6]. These properties make AlN possible for the application in the SAW devices, thin film bulk acoustic wave resonators (FBARs), *etc* [2-4].

To date, AlN-based devices based on the AlN epitaxial films grown on sapphire substrates by metal-organic chemical vapor deposition (MOCVD) have been commercialized [7-8]. However, the performance for these AlN-based devices on sapphire substrates is usually restricted by the poor thermal conductivity of sapphire substrate of 25 W m^{-1} K^{-1} in heat dissipating and the large lattice mismatch between AlN and sapphire of ~13.3% [9-10]. In this regard, researchers have tried hard to grow AlN epitaxial films on higher thermal conductivity substrates with smaller lattice mismatch between AlN and substrates [11-14]. Among these substrates [11-14], Al seems to be one of the most suitable substrates. On the one hand, the thermal conductivity of Al is 237 W m^{-1} K^{-1}, which is more than 9 times of that of sapphire [8]. On the other hand, the lattice mismatch between Al and AlN is 8.7%, which is smaller than that between AlN and sapphire [8]. Therefore, it is very promising to the fabrication of highly-efficient AlN-based devices on Al substrates. So far, AlN epitaxial films have been grown on Al substrates by various methods [13-14]. T. Honda, et al. used molecular beam epitaxy (MBE) to grow AlN epitaxial films on Al substrates at ~660 ºC. In their work, the as-grown AlN epitaxial films are polycrystalline [13]. These results may be ascribed to the serious interfacial reactions between AlN epitaxial films and Al substrates during the high temperature (HT) growth, where high-density dislocations are formed during the initial growth, and eventually leads to the nucleation difficult [8-9]. W. Wang et al, deployed pulsed laser deposition (PLD) to grow AlN epitaxial films on Al substrates at low temperature (LT) of 450 ºC with one-step growth [14-15]. In their work, the single-crystalline AlN epitaxial films grown on Al substrates are obtained [14]. These results may be ascribed to the LT growth by PLD, which can effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates [11-12]. The pulsed laser can supply enough energy for the migration of precursors on the substrates, and therefore make the films growth at LT possible [15-17]. Nevertheless, the properties, especially, the surface morphology and crystalline quality, of AlN epitaxial films grown on Al substrate by PLD at LT with one-step growth are still not good enough [14-15], and still have space to be further improved to meet the recent requirements in the fabrication of highly-efficient AlN-based devices on Al substrates.

In this work, we report on the growth of AlN epitaxial films on Al substrates by PLD with two-step growth by the combination of LT and HT growth. The LT AlN buffer layer is firstly grown by PLD to effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates, and then HT growth is carried out to further improve the quality of AlN epitaxial films. The properties of as-grown AlN epitaxial films on Al substrates by two-step growth are investigated in detail with various measurements, such as high-resolution transmission electron microscopy (HRTEM), polarized light microscopy (PLM), scanning electron microscopy (SEM), atomic force microscopy (AFM), high-resolution X-ray diffraction (HRXRD), *etc*. This work of achieving quality-enhanced AlN epitaxial films is of significant importance for the fabrication of highly-efficient AlN-based devices which require abrupt hetero-interfaces and smooth surfaces.

2. Experimental

The as-received Al substrates with the dimension of 1×1 cm^2 were taken a degassing treatment in an ultra-high vacuum (UHV) load-lock chamber with a background pressure of 1.0×10^{-8} Torr at 200 ºC for 30 min. Subsequently, the as-degassed Al substrates were transferred into an UHV PLD growth chamber with a background pressure of 3.0×10^{-10} Torr, followed by an annealing process was performed to the as-degassed Al substrates to remove residual surface contaminants at 550 ºC for 60 min. After the annealing, the as-annealed Al substrates were nitrided in a plasma ambient produced by a radio-frequently plasma generator attached to the PLD growth chamber at 550 ºC for 60 min. During the growth, a KrF excimer laser light (λ=248 nm, t=20 ns) placed at 8 cm away from the AlN target with the energy density and the pulse repetition of 3.0 $J.cm^{-2}$ and 30 Hz was used to ablate the high-purity AlN target. The ~50 nm-thick AlN buffer layer was firstly grown on Al substrates at LT of 450 ºC in a 4 mTorr nitrogen ambient to effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates. Subsequently, ~250 nm-thick AlN epitaxial films were grown on this LT buffer layer at various temperatures ranging from 450 to 650 ºC in a 4 mTorr nitrogen ambient to improve the quality of films, as shown in Fig. 1. Since the temperature of Al melting point is ~660 ºC [13], the highest growth temperature for AlN epitaxial growth used in this study is 650 ºC. To fully understand the effect of two-step growth, ~300 nm-thick AlN epitaxial films grown on Al substrates with one-step growth under the growth temperature of 650 ^{o}C were carried out. The interfacial property, structural property and surface morphology of as-grown ~300 nm-thick AlN epitaxial films were studied by HRTEM (JEOL 3000F), PLM (OLYMPUS, BX51M), SEM (Nova Nano SEM 430 Holland), AFM (Bruker Dimension Edge, American), and HRXRD (Bruker D8 X-ray diffractometer with Cu Kα1 X-ray source λ=1.5406 Å). As for TEM characterization, the as-grown ~300 nm-thick AlN epitaxial films on Al(111) substrates were firstly mechanically polished to be ~50 μm, and then a low-energy and low-angle ion milling (Fischione 1010 Low Angle Ion Milling & Polishing System) process was carried out. At first, the low-energy and low-angle for ion milling were set as 5 keV and 10°, respectively; and then were reduced to 4 keV and 4-5°, respectively, when the hole appeared. After finishing the ion milling, the sample edge thickness was about 20 nm. The cross-sectional samples were then transferred into a JEOL 3000F field emission gun TEM, which works at a voltage of 300 kV and gives a point to point resolution of 0.17 nm.

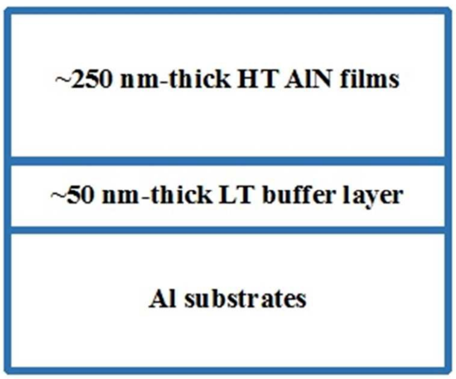


Fig. 1. Schematic diagram of AlN epitaxial films grown on Al substrates by PLD.

3. Results and discussion

It is known that the interfacial property between AlN epitaxial films and substrates is of paramount importance in the fabrication of AlN-based devices [4-6, 14], and thereby the cross-sectional TEM is adopted to study the interfacial property of ~300 nm-thick AlN epitaxial films with two-step growth. Figs. 2a and 2b show the cross-sectional TEM images for ~300 nm-thick AlN epitaxial films grown with HT growth temperature of 650 ºC at low magnification and high magnification, respectively. From Fig. 2a, we can find the clear hetero-interface between AlN epitaxial films and Al substrates, and ~301 nm-thick AlN epitaxial films are measured in this case. Moreover, to investigate the interfacial property of AlN/Al hetero-interface after the HT growth, cross-sectional TEM at high magnification is carried out. Due to fact that the Al melting point temperature is ~660 ºC, the highest temperature for AlN epitaxial films for this study is 650 ºC [13]. It is found that the AlN/Al hetero-interface keep sharp and abrupt in the HT growth temperature ranging from 450 to 650 ºC. Fig. 2b is the cross-sectional TEM image for AlN/Al hetero-interface in ~300 nm-thick AlN epitaxial films grown with HT growth temperature of 650 ºC, from which sharp and abrupt hetero-interface still can be clearly identified and is consistent with the result reported at growth temperature of 450 ºC [14-15]. These results confirm that subsequent HT growth does not influence the interfacial properties of AlN/Al hetero-interface during the initial LT growth.

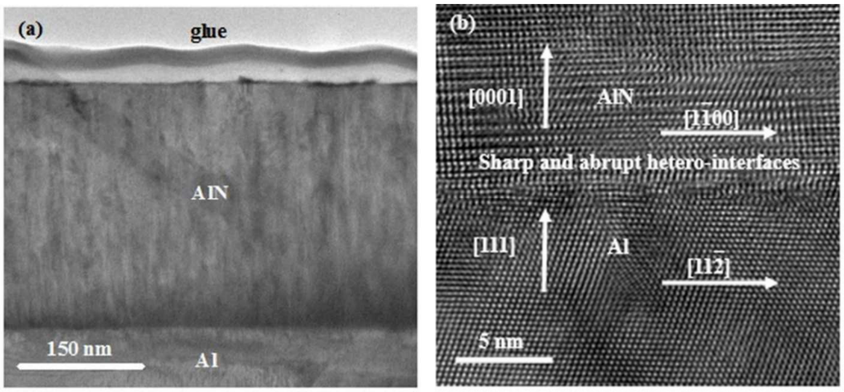


Fig. 2. Cross-sectional TEM images for ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature of 650 ºC at (a) low magnification and (b) high magnification.

The effect of coefficient of thermal expansion (CTE) on the properties of as-grown ~300 nm-thick AlN films is carefully studied. It is known that the large CTE mismatch between epitaxial films and substrates would lead to the formation of large stress in epitaxial films, which may result in the cracks [18-19]. Figs. 3a and b reveal the PLM images for AlN films grown on Al substrates with different cooling rates. One can find the cracks on the surface of ~300 nm-thick AlN epitaxial films with the cooling rate of 15 ºC/min shown in Fig. 3a, while there are no cracks on the surface of ~300 nm-thick AlN epitaxial films with the cooling rate of 2 ºC/min shown in Fig. 3b. These results may be ascribed to the large CTE mismatch between AlN and Al of 85.22%, which may lead to the formation of cracks under the high cooling rate [14]. At the cooling rate of 15 ºC/min, the large stress is formed in the AlN epitaxial films rapidly and then releases with high-density dislocations. In this case, the formed dislocation accumulates and then results in the cracks. However, at the cooling rate of 2 ºC/min, the stress is also formed in the AlN epitaxial films but is released slowly by the formation of dislocation. In this case, the dislocation does not cause the cracks in the AlN epitaxial films [18-19]. Therefore, the cooling rate plays an importance role in obtaining crack-free thin films in the case of the large CTE mismatch between epitaxial films and substrates.

The effect of HT growth temperature on the RMS surface roughness of ~300 nm-thick AlN epitaxial films grown on Al substrates is also studied by AFM and SEM. Figs. 4a-e show the surface morphology of ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature ranging from 450 to 650 ºC. It can be noted that the RMS surface roughness of 2.4 nm in 5×5 μm^2 for ~300 nm-thick AlN epitaxial film grown on Al substrate with HT growth temperature of 450 ºC is provided in Fig. 4a. As the HT growth temperature is gradually increased from 450 to 650 ºC, as shown in Figs. 4b-e, one can find that the RMS surface roughness measured by AFM in 5×5 μm^2 for ~300 nm-thick AlN epitaxial films grown at 500, 550, 600, and 650 ^{o}C is 2.1, 1.7, 1.5, and 1.1 nm, respectively. These results confirm the improvement of surface morphology for AlN epitaxial films with the increase of HT growth temperature from 450 to 650 ºC. However, the ~300 nm-thick AlN epitaxial films with one-step growth under the growth temperature of 650 ºC show very poor surface morphology with the RMS surface roughness of 17.7 nm shown in Fig. 4f. We attribute these results to the two-step growth by PLD. On the one hand, the LT growth by PLD not only can effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates [20-21], but also benefit to the nucleation of AlN epitaxial films during the initial growth [22]. On the other hand, the subsequent HT growth can promote the migration of AlN precursors and enhance the coalescence of AlN epitaxial films [23]. Both of these two aspects lead to the improvement of AlN surface smoothness.

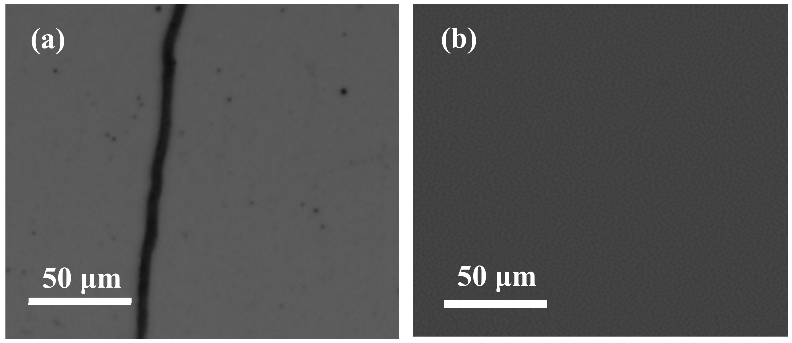
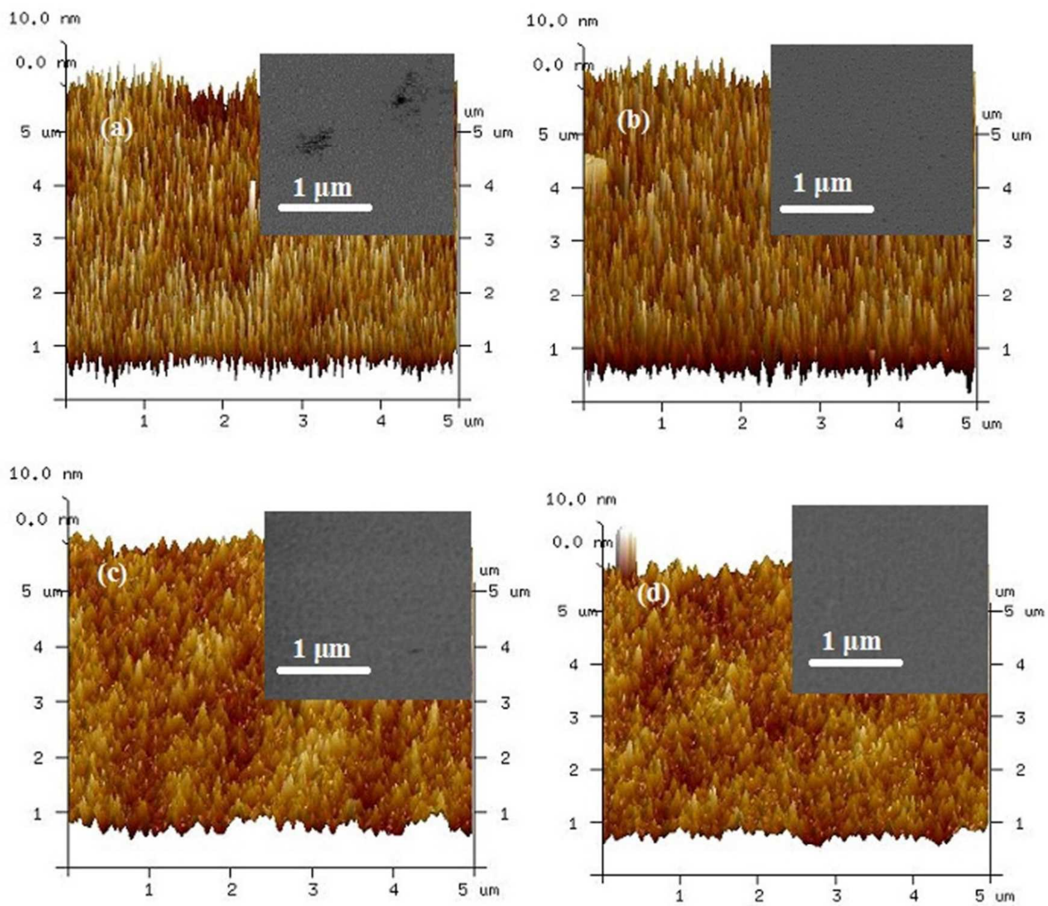


Fig. 3. PLM images for AlN films grown on Al substrates with HT of 650 ºC under the cooling rates of (a) 15 and (b) 2 ºC/min, respectively.



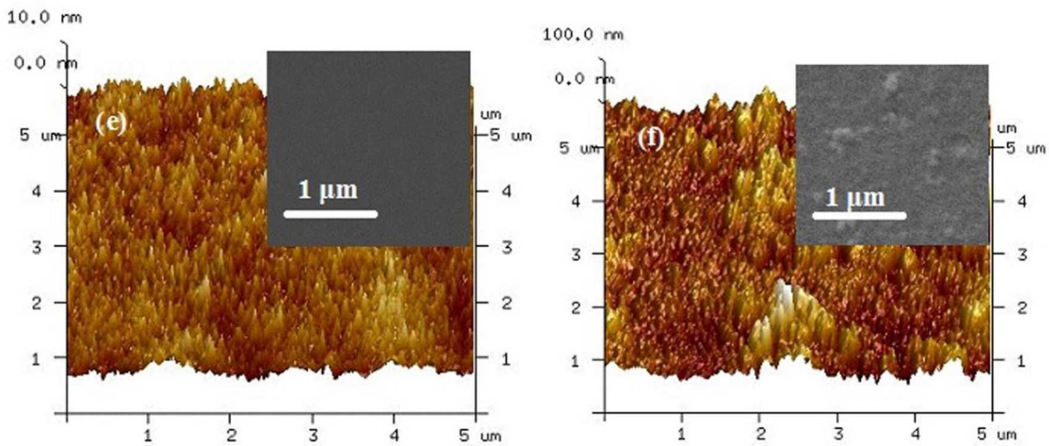


Fig. 4. AFM and SEM images for ~300 nm-thick AlN epitaxial films grown on Al substrates with two-step growth under the HT growth temperature of (a) 450, (b) 500, (c) 550, (d) 600, and (d) 650 ºC, respectively; and (f) AFM and SEM images for ~300 nm-thick AlN epitaxial films grown on Al substrates with one-step growth under the growth temperature of 650 ºC.

The structural properties of AlN epitaxial films grown with two-step growth and one-step growth are studied by XRD. Typical 2θ-ω for~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature ranging from 450 to 650 ºC is provided in Fig. 5a. The peak observed at 2θ=36.02° is ascribed to the diffraction from AlN(0002) and the peak identified at 2θ=38.56° is attributed to the diffraction from Al(111) [14, 24]. Moreover, It can be noted that the intensity of AlN(0002) peak is monotonously raised as the HT growth temperature is increased from 450 to 650 ºC. These results confirm the increase in crystalline quality of AlN epitaxial films to some extent [25]. Furthermore, AlN($10\bar{1}2$) φ scan is measured, and six-fold rotational peaks with 60° interval are identified in Fig. 5b. Based on the 2θ-ω and φ scans, we can conclude that single-crystalline AlN epitaxial films have been grown in this work [23]. However, as for ~300 nm-thick AlN epitaxial films grown on Al substrates with one-step growth under the growth temperature of 650 ºC, as shown in Fig. 5c, no AlN peak can be found except the diffraction peak of Al substrate. This result confirms the poor-quality of AlN epitaxial films grown at high temperature with one step growth [14-15].

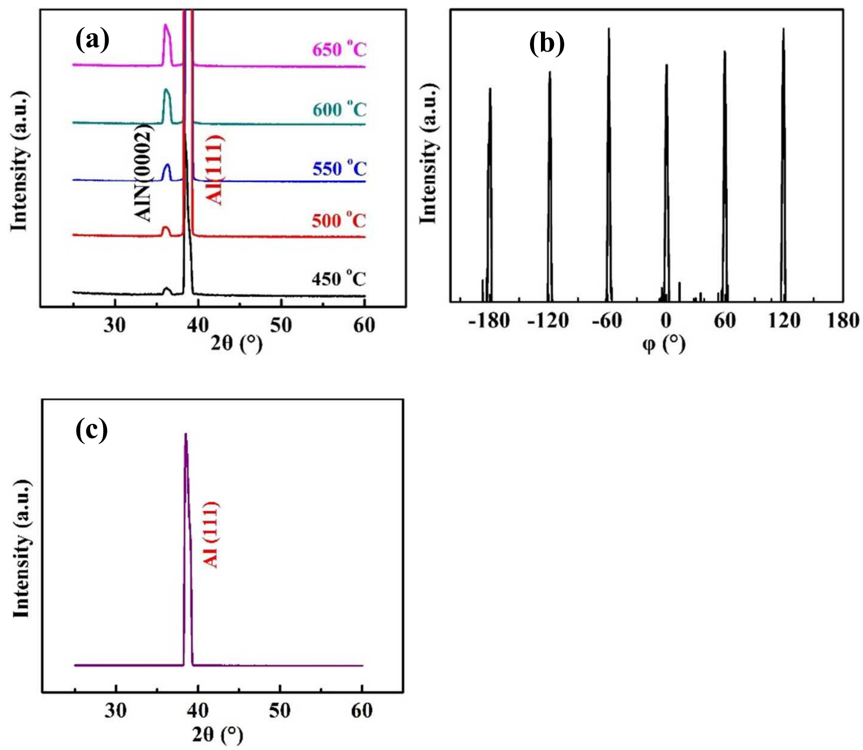


Fig. 5. (a) XRD 2θ-ω scan for ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature ranging from 450 to 650 ºC, and (b) AlN($10\bar{1}2$) φ scan for ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature of 650 ºC. (c) XRD 2θ-ω scan for ~300 nm-thick AlN epitaxial films grown on Al substrates with one-step growth under the growth temperature of 650 ºC

To further study the crystalline quality, the X-ray rocking curve (XRC) measurement is deployed. Figs. 6a and b reveal the typical AlN(0002) and AlN($10\bar{1}2$) XRCs for ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature of 650 ºC, where the full-width at half-maximums (FWHMs) for AlN(0002) and AlN($10\bar{1}2$) XRCs are measured to be 0.45° and 0.72°, respectively. These results are smaller than those of AlN epitaxial films grown by PLD with one-step growth [14], and are in striking contrast to the polycrystalline AlN epitaxial films grown on Al substrates by MBE [13].

Apparetently, the effect of HT growth temperature in the two-step growth on the crystalline quality of AlN epitaxial films on Al substrates is also investigated in depth. At HT growth temperature of 450 ºC, the FWHMs of AlN(0002) and AlN($10\bar{1}2$) XRCs for ~300 nm-thick AlN epiatxial films are 0.6° and 0.9°, respectively, as shown in Fig. 6c. One can note that when the HT growth temperature increases, the FWHMs of AlN(0002) and AlN($10\bar{1}2$) XRCs are gradually reduced, ending up with 0.45° and 0.72°for ~300 nm-thick AlN epiatxial films grown with HT growth temperature of 650 ºC, respetively, as shown in Fig. 6c. It is known that the FWHMs of AlN(0002) and AlN($10\bar{1}2$) XRCs are related to the srew disloaction generated from the different step heights of the substrate and the pure edge and mixed dislocations mainly formed during the coalescence process among the mis-oriented individual islands [26-28], respectively. Furthermore, it is also found that the dislocation density shows a positive correlation with FWHM of as-grown AlN films [26]. In this regard, based on the results as shown in Fig. 6c, as the HT growth temperature increases, the crystalline quality of AlN epitaxial films is improved siganificantly. The reason for achiveing high-quality AlN epitaxial films is the introducing of two-step growth. On the one hand, the LT buffer growth by PLD can effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates, where dislocation density is greatly reduced [20-21]. On the other hand, the subsequent HT growth by PLD can enhance the coherence of AlN epitaxial films, and thereby the formation of dislocations is eventually reduced [22, 26, 29-35]. Moreover, It is found that within the range of HT growth temperature from 450 to 650 ºC, the higher the HT temperature is, the higher crystalline quality of AlN epitaxial films will be.

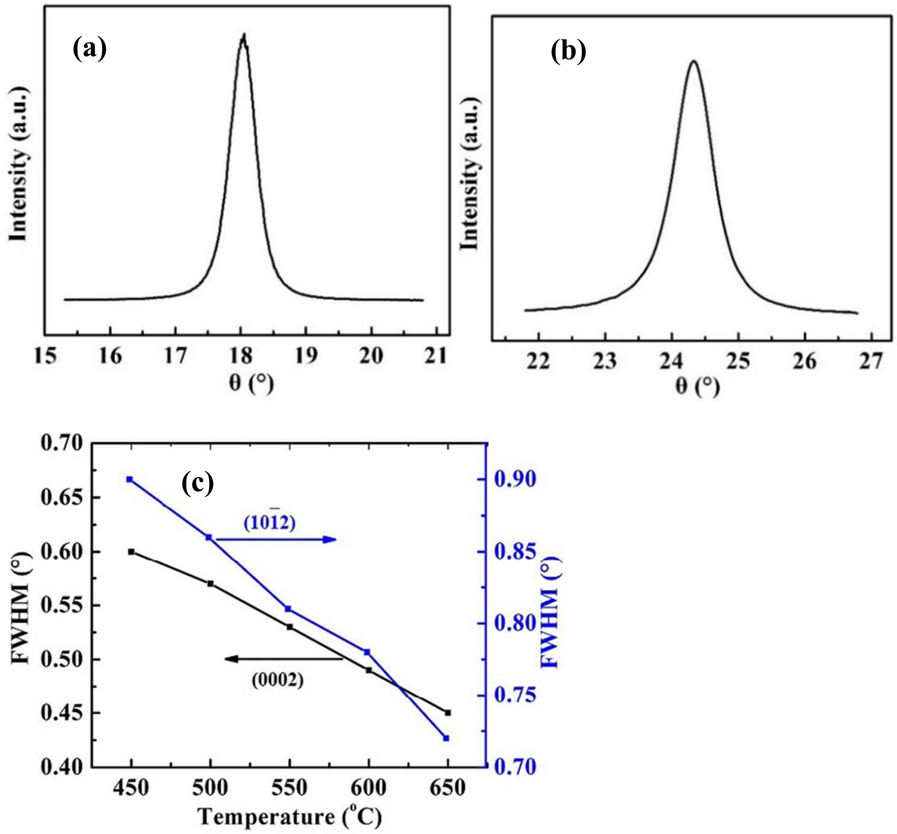


Fig. 6. XRC of (a) AlN(0002) and (b) AlN(10$\bar{1}$2) for ~300 nm-thick AlN epitaxial films grown on Al substrates with HT growth temperature of 650 ºC. (c) Temperature dependence of HT growth of FWHM for AlN(0002) and AlN(10$\bar{1}$2) XRCs in ~300 nm-thick AlN epitaxial films grown on Al substrates.

4. Conclusions

Quality-enhanced AlN epitaxial films have been grown on Al substrates by PLD with two-step growth. The effect of HT growth temperature on the properties of AlN epitaxial films on Al substrates are studied by various measurements, such as HRTEM, AFM, XRD, *etc.* It is found that the AlN/Al hetero-interfaces keep sharp and abrupt under HT growth from 450 to 650 ºC. It is clear that the RMS surface roughness is 2.4 nm, and the FWHMs of AlN(0002) and AlN(10$\bar{1}$2) XRCs are 0.6° and 0.9°, respectively, for ~300 nm-thick AlN epitaxial films grown at HT growth temperature of 450 ºC. As the HT growth temperature increases, the surface morphology and crystalline quality of AlN epitaxial films are gradually improved. The RMS surface roughness is 1.1 nm, and the FWHMs of AlN(0002) and AlN($10\bar{1}2$) XRCs are 0.45° and 0.72°, respectively, for ~300 nm-thick AlN epitaxial films grown at HT growth temperature of 650 ºC. We attribute these results to the two-step growth by PLD. On the one hand, the LT growth by PLD not only can effectively suppress the interfacial reactions between AlN epitaxial films and Al substrates, but also benefit to the nucleation of AlN epitaxial films during initial growth. On the other hand, the subsequent HT growth can promote the migration of AlN precursors and enhance the coalescence of AlN epitaxial films. Both of these two aspects improve the quality of AlN epitaxial films. This work of achieving quality-enhanced AlN epitaxial films is of paramount importance for the application of highly-efficient AlN-based devices on Al substrates. Moreover, this two-step growth by PLD opens up an abroad prospect for the growth of AlN epitaxial films on thermally active substrates.

Acknowledgements

This work is supported by National Science Fund for Excellent Young Scholars of China (No. 51422203), National Natural Science Foundation of China (Nos. 51572091 and 51372001), Distinguished Young Scientist Foundation of Guangdong Scientific Committee (No. S2013050013882), Key Project in Science and Technology of Guangdong Province (Nos. 2014B010119001 and 2011A080801018), and Strategic Special Funds for LEDs of Guangdong Province (Nos. 2011A081301010, 2011A081301012 and 2012A080302002). Wenliang Wang and Weijia Yang contributed equally to this work.

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