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ARTICLES

Four-inch high quality crack-free AlN layer grown on a high-temperature annealed AlN template by MOCVD

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Abstract: In this work, based on physical vapor deposition and high-temperature annealing (HTA), the 4-inch crack-free high-quality AlN template is initialized. Benefiting from the crystal recrystallization during the HTA process, the FWHMs of X-ray rocking curves for (002) and (102) planes are encouragingly decreased to 62 and 282 arcsec, respectively. On such an AlN template, an ultra-thin AlN with a thickness of ~700 nm grown by MOCVD shows good quality, thus avoiding the epitaxial lateral overgrowth (ELOG) process in which 3–4 μm AlN is essential to obtain the flat surface and high crystalline quality. The 4-inch scaled wafer provides an avenue to match UVC-LED with the fabrication process of traditional GaN-based blue LED, therefore significantly improving yields and decreasing cost.

Key words: AlN; high temperature annealing; MOCVD

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1. Introduction

The explosive spread of the coronavirus disease (COVID-19) in the 2019 pandemic intensively excites the requirement for high-efficiency environmental sterilization that interrupts the most important link in the chain of disease transmission. Although plenty of conventional methods have been used, e.g. alcohol immersion, high-temperature treatment, as well as high-energy irradiation, AlGaN-based ultraviolet-C (UVC, $\lambda \leqslant 280$ nm) light-emitting diode (LED) disinfection is emerging as one of the most promising and convincing avenues to confront COVID-19[1–3]. However, when compared with conventional GaN-based blue LED, the UVC-LED only exhibits external quantum efficiency (EQE) less than 10%[4]. One of the most crucial challenges is the difficulty of acquiring high-crystalline lattice-matched meanwhile a non-UV-absorbed substrate for upper UVC device epitaxy. Due to the short wavelength of UVC irradiation, the large band gap (>4.6 eV) is the prerequisite to screen the candidates of whom sapphire and aluminum nitride (AlN) substrates are both qualified. Unfortunately, by weighing the trade-off between large-wafer scale and lattice-match, neither of them is overall-competent: the large lattice-mismatch exhibits between sapphire and AlGaN epilayer while the inch-scale AlN wafer preparation is still a great challenge. Direct AlN epitaxy on sapphire substrate usually accompanies high dislocation density (10^{10} cm^{-2}), which murders the device performance. Nevertheless, the proposal and verification of the face-to-face high-temperature annealing (HTA) technique obviously relieves on the above embarrassment through achieving an excellent-crystalline AlN template on sapphire substrates[5, 6]. Subsequently, the successful regrowth of pronounced-crystalline AlN[5, 7, 8] and AlGaN[9, 10] epilayers as well as a full structure UVC-LED device[11, 12] by metal-organic chemical vapor deposition (MOCVD) unambiguously highlights the validation and prospective of such a solution.

From the viewpoint of industry and commercialization, the employment of economical AlN template, a 4-inch scaled wafer in particular would be more attractive if it exhibits the possibility to further reduce the cost of UVC-LED: (i) the cost would be intensively decreased by using crack-free 4-inch AlN template which has never been achieved to date; and (ii) the previously necessary epitaxial thickness of 3–4 μm (to acquire the qualified flat morphology and high crystalline quality for subsequent device epitaxy) would be avoided and largely save the expense of device epitaxy.

In this work, by combining the physical vapor deposition (PVD) and face-to-face HTA technique, for the first time, a 4-inch single-crystalline AlN template whose dislocation density is as low as 9.2×10^8 cm^{-2} level is achieved. On the basis of the HTA AlN template, the MOCVD regrowth of homo-epitaxial AlN layer at the 4-inch wafer scale is initialized, highlighting the prospective of wafer-sized HTA AlN templates in UVC

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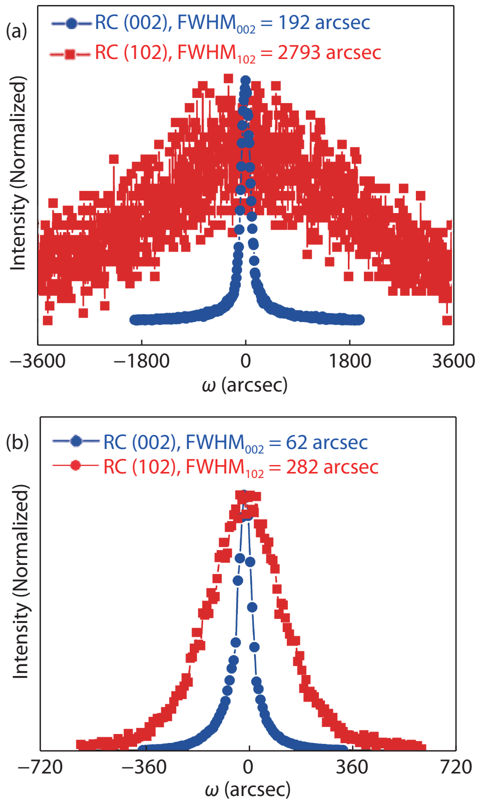

Fig. 1. (Color online) The XRD rocking curves (XRC) of (002) (circles) and (102) (squares) planes for (a) as-sputtered and (b) HTA AlN samples.

irradiation source revolution.

2. Experiment

Four-inch AlN wafers were prepared on *c*-plane sapphires by reaction magneto-sputtering technique using aluminum (purity ~ 99.999%) as the target, and the sputtering ambient was set as the mixture of argon and nitrogen as a ratio of 1 : 4. The AlN thickness was set as 500 nm by calibrating the growth speed and the sputtering power was 3000 W. Afterwards, as-sputtered AlN wafers were annealed by utilizing a tube furnace at 1700 °C for over 5 h, and the annealing ambient was nitrogen with a flow rate of 0.5 SLM. The MOCVD AlN regrowth was performed by a Prismo HiT3 MOCVD system at a temperature of 1200 °C. The regrown thickness is 200 nm. The rocking curves of AlN (002) and (102) planes were measured by X-ray diffraction (XRD, Brucker D8 Discovery) to evaluate the AlN crystallinity. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) was carried out in a Thermo fisher FEI Themis Z Cs probe-corrected STEM system operated at 300 kV, weak-beam dark-field (WBDF) TEM was observed using Tecnai F30 at 300 kV. The scanning electron microscopy (SEM, Hitachi Regulus 8100) and atomic force microscopy (AFM, Veeco Dimension$^{\rm TM}$ 3100) with a typing mode were used to explore the surface morphology of all AlN samples.

3. Results and discussion

Fig. 1 shows XRD rocking curves (XRC) of (002) and (102) planes of as-sputtered and HTA AlN. Before the HTA treatment, the as-sputtered AlN presents *c*-oriented characteristic. The full width at half maximum (FWHM) of (002) and (102) XRC are 192 and 2793 arcsec, respectively, which are comparable with the values of previously reported as-Al target-sputtered[12, 13], as-AlN target-sputtered[6, 14], as well as as-MOCVD-grown AlN films[15]. Notably, the HTA treatment intensively improves the crystallinity of as-sputtered sample, and the FWHMs of (002) and (102) planes decrease to 62 and 282 arcsec, respectively, indicating the highly ordered re-alignment of the crystalline lattice triggered by high temperature process[16]. According to the mosaic model, the densities of two-type dislocation are described as below[17–19]:

$$D_{\rm dis} = \frac{\beta^2}{4.36b^2}, \qquad (1)$$

where the $D_{\rm dis}$ represents the density of dislocation, b is the length of Burgers vector, with values of 0.4982 and 0.3112 nm for screw- and edge-type dislocations, respectively; β represents the tilt angle $\beta_{\rm tilt}$ or twist angle $\beta_{\rm twits}$ of the mosaic structure, which are obtained by analyzing the dependence of FWHM values of XRCs on different symmetric and asymmetric planes, respectively[17, 20–23]. It is worth noting that the HTA obviously reduces the total threading dislocation density (TDD) from 9.27×10^{10} to 9.20×10^{8} cm^{-2}, whereas the screw and edge dislocation densities decrease from 8.02×10^{7} and 9.26×10^{10} cm^{-2} down to 8.37×10^{6} and 9.19×10^{8} cm^{-2}. Such a reduction in dislocation density is mainly due to the recrystallization and lattice rearrangement process of AlN columnar crystals[6, 24], and during which it is partially assisted by the reduction of lattice mismatch between the AlN and sapphire substrate at ultra-high temperatures. Due to the larger thermal expansion coefficient of sapphire (α_a = 8.1 × 10^{-6} K^{-1}) over AlN (α_a = 4.2 × 10^{-6} K^{-1})[25], the lattice parameters along the *a*-axis are changed from 0.3111 and 0.4758 nm to 0.3133 and 0.4824 nm for AlN and sapphire substrate from room temperature to 1700 °C, respectively, thus the lattice mismatch is reduced from 13.3% to 12.5%, which partly contributes to the improvement of excellent crystalline quality of the AlN layer.

To capture more information of the HTA AlN, the dark field cross-sectional TEM measurement was adopted, with two beam conditions carried out to analyze dislocations in different types. As shown in Figs. 2(a) and 2(b), the screw-type dislocation is nonvisible while only two edge-type dislocation lines are seen in the scanned area. Such low dislocation density agrees with XRC results. To analyze the dislocation annihilation driven by HTA operation, high-resolution HAADF-STEM was performed, focusing on the interfacial region between HTA AlN and sapphire. From the Fig. 2(c), an atomically sharp interface between sapphire and HTA AlN is visible and demonstrates a nice epitaxial nature in the high temperature treated AlN/sapphire system. However, a color-contrast region around 10 nm away from the interface is detected as a boundary between sapphire-neared N-polar AlN and upper Al-polar AlN. Actually, such a depth has been observed and verified as inversion domain boundary which consists of compound (111) γ-AlON[13, 26, 27]. According to the Al_2O_3–AlN–AlON system phase diagram[26], the γ-AlON does not directly contribute to the crystalline optimization. However, as we know, the as-sputtered AlN film exhibits columnar-like AlN grains which perform lateral polarity distribution and the grain boundaries between these domains are important source of threading dislocations[28, 29]. As shown in Fig. 2, the

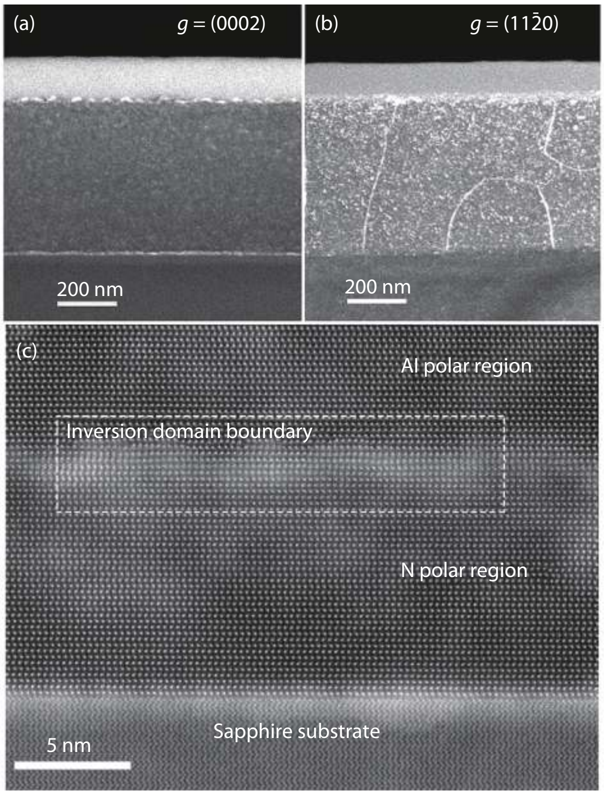

Fig. 2. Cross-sectional weak-beam dark-field (WBDF) TEM images of HTA AlN taken under diffraction conditions of (a) $g = (0002)$ and (b) $g = (11\bar{2}0)$. For $g = (0002)/(11\bar{2}0)$, the screw-type/edge-type dislocation is visible. (c) Cross-sectional high-resolution HAADF-STEM along the $[11\bar{2}0]$ direction by focusing on the interfacial region.

participation of γ-AlON successfully turns the N-polar phase into homogenous Al-polar one. Therefore, although the participation of such a γ-AlON region does not directly contribute to the crystalline improvement, it potentially suppresses the possible generation of threading dislocations from the grain boundaries between the N-polar and Al-polar regions by forming a uniform stable Al-polar epilayer.

In addition to the prospective from the application point of view, a series of fundamental experience, which possibly deepens the understand of high temperature AlN regrowth, particularly the contribution from oxides cooperation[14], curvature[30, 31], as well as defect evolution[10, 32, 33], are collected. Some methods reveal new avenues to intentionally improve the high-temperature annealing film, e.g. artificially involving the domain inverse region to release the interfacial stress by fully employing oxidation[14]. Therefore, our HTA AlN template acts as a great test bed to explore the optimization of single-crystal thin film.

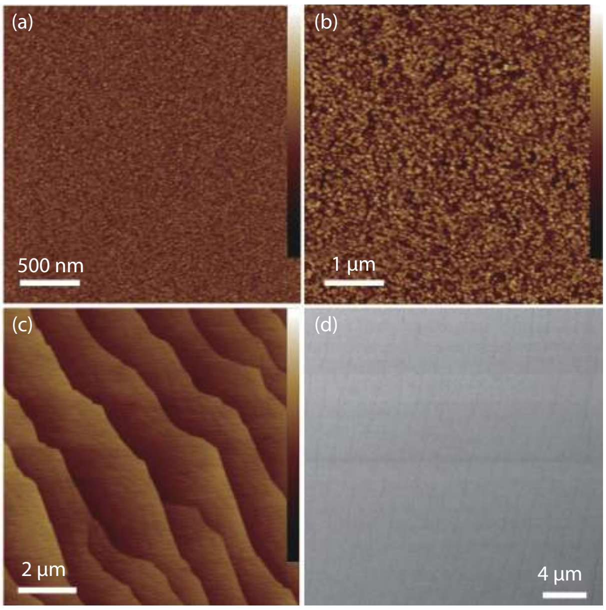

Fig. 3. (Color online) Atomic force microscopy images of (a) as-sputtered AlN, (b) HTA AlN as well as (c) MOCVD regrown AlN. (d) The SEM image of MOCVD regrown AlN on HTA AlN template. The height bar is 20 nm.

The morphologies of as-sputtered, post-annealed, as well as MOCVD-regrown, AlN were studied by AFM, and the results are shown in Fig. 3. The as-sputtered AlN presents the columnar-morphology which is the same as the previously reported AlN film prepared by magneto-sputtering[5, 6, 13], and the root mean square (RMS) is 2.62 nm. The HTA operation does not essentially change the morphology: the columnar- or particle-like instead of the reported step-bunching surface with an RMS of 0.86 nm is present. Such a distinction is possibly from the different target used in the sputter process, e.g. the step-bunching morphology is mostly observed in the AlN target sputtered system[5, 6, 9, 10, 14, 34–36], but the particle-like morphology is mostly detected in the Al target sputtered one[7], and this may affect the surficial decomposition during the annealing treatment. Nevertheless, it seems that such a rough surface does not negatively contribute to the subsequent AlN regrowth by MOCVD, and the AFM and SEM images of MOCVD-grown AlN layer are present in Figs. 3(c) and 3(d). It is seen that the as-annealed rough surface has been refreshed by the step-bunching morphology which results from the high enough diffusion length of Al atom exceeding a certain value in relation to the terrace width[37], indicative of the ideal platform of HTA AlN for subsequent AlN-based device fabrication. The obtained morphology and RMS are both comparable with the samples prepared by epitaxial lateral overgrowth (ELOG) at high temperatures[3]. Encouragingly, the successful epitaxy of regrown AlN fulfils the prerequisite of fabricating 4-inch UVC-LED.

When compared with a 2-inch wafer, the 4-inch AlN layer grown on sapphire by MOCVD generally exhibits larger bow and terrible cracks which is detrimental to upper epilayers and acts as the main obstacle to large-sized device epitaxy[38, 39]. As shown in Fig. 4(b), the regrown AlN layer on 4-inch HTA template clearly shows an advantage in this viewpoint that the cracks just appear at the region around 1.5 mm away from the edge. The wafer-scaled surface cracks are visibly measured by Candela, and the results are shown in Figs. 4(c) and 4(d). It is observed that the conventional 4-inch AlN/NPSS template obtained by the ELOG process presents lots of cracks on the surface, even in the central region of the wafer. Meanwhile, as shown in Figs. 4(b) and 4(c), only a little bit roughening appears in the edge region. The comparison emphasizes the advantage of the HTA technique in achieving 4-inch-sized high-quality single-crystalline AlN template. Moreover, the FWHM values of XRC (002) and (102) planes in selected five points [shown in Fig. 4(a)] on as-annealed and post-regrown samples indicate excellent homogeneity, as shown in Table 1. In addition to crystallinity, the strain of re-

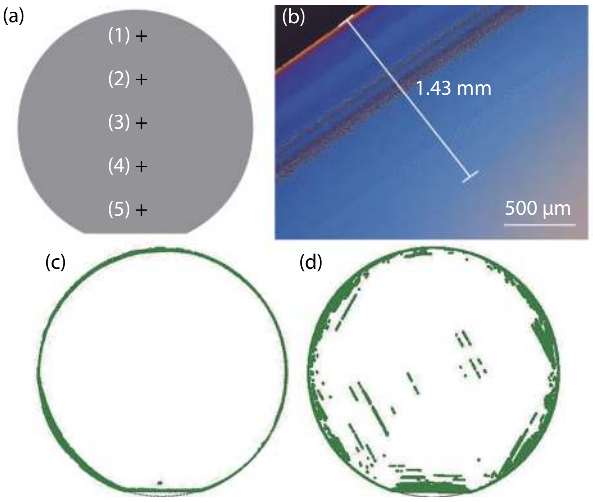

Fig. 4. (Color online) (a) Positions of five measured XRC points on 4-inch as-sputtered AlN and MOCVD regrown AlN wafers, and the results are shown in Table 1. (b) Optical microscopy image of the edge region in MOCVD regrown AlN wafer on 4-inch HTA AlN template. The images of surface cracks on (c) HTA AlN and (d) AlN/NPSS templates are measured by Candela.

Table 1. Five points XRC FWHMs and θ–ω calculated strains of 4-inch post-HTA AlN and MOCVD regrown AlN wafers.

Position	HTA AlN FWHM $_{(002)/(102)}$ (arcsec)	Regrown AlN FWHM $_{(002)/(102)}$ (arcsec)	Regrown AlN strain // c (%)
1	87 / 310	162 / 381	0.0869
2	64 / 288	95 / 372	0.0799
3	57 / 283	85 / 320	0.0795
4	61 / 310	78 / 312	0.0795
5	70 / 321	89 / 329	0.0807

grown AlN layer also presents a homogenous compressive feature with a value of about 0.08% as shown in Table 1.

4. Conclusion

In summary, the pronounced single-crystalline 4-inch AlN templates with dislocation density as low as 9.2×10^8 cm^{-2} on the *c*-plane sapphire are achieved. Thanks to such high-quality AlN template, the MOCVD regrown AlN with the thickness of only 700 nm shows comparative quality as the 3–4 μm AlN layer grown by ELOG. The exhibiting bunching-step morphology and low dislocation density in the regrown 4-inch AlN layer prove its qualification of being an ideal candidate substrate for low-cost UVC-LEDs.

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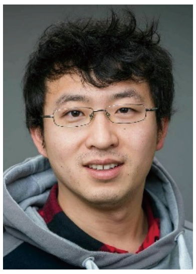

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