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Highly c-axis oriented growth of GaN film on sapphire (0001) by laser molecular beam epitaxy using HVPE grown GaN bulk target

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Growth temperature dependant surface morphology and crystalline properties of the epitaxial GaN layers grown on pre-nitridated sapphire (0001) substrates by laser molecular beam epitaxy (LMBE) were investigated in the range of 500–750 °C. The grown GaN films were characterized using high resolution x-ray diffraction, atomic force microscopy (AFM), micro-Raman spectroscopy, and secondary ion mass spectroscopy (SIMS). The x-ray rocking curve full width at a half maximum (FWHM) value for (0002) reflection dramatically decreased from 1582 arc sec to 153 arc sec when the growth temperature was increased from 500 °C to 600 °C and the value further decreased with increase of growth temperature up to 720 °C. A highly c-axis oriented GaN epitaxial film was obtained at 720 °C with a (0002) plane rocking curve FWHM value as low as 102 arc sec. From AFM studies, it is observed that the GaN grain size also increased with increasing growth temperature and flat, large lateral grains of size 200-300 nm was obtained for the film grown at 720 °C. The micro-Raman spectroscopy studies also exhibited the high-quality wurtzite nature of GaN film grown on sapphire at 720 °C. The SIMS measurements revealed a non-traceable amount of background oxygen impurity in the grown GaN films. The results show that the growth temperature strongly influences the surface morphology and crystalline quality of the epitaxial GaN films on sapphire grown by LMBE. *© 2013 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License.* [http://dx.doi.org/10.1063/1.4821276]

I. INTRODUCTION

Group III-nitride compounds have attracted much attention for their emerging applications in optoelectronic devices such as light-emitting diode (LED), laser diodes (LD) and ultra-violet detectors, due to their direct band gap semiconducting nature and good thermal conductivity.[1–3] The surface morphology and crystalline quality of GaN templates are the key factors for the realization of high performance GaN based devices. GaN is heteroepitaxially grown on different substrates like SiC,[4,5] Si (111),[6,7] sapphire (0001),[8–12] etc.[13–15] Among these substrates, sapphire is commonly used for fabricating GaN based LED and LD devices due to its low cost, superior material quality and availability in large-sized wafers. Due to the large lattice mismatch between sapphire and GaN, a rough surface of GaN film with high density of threading dislocations is obtained on sapphire (0001).[11,12] However, initial nitridation (pre-nitridation) of sapphire substrates by nitrogen radicals acts as an intermediate or buffer layer for growing a high quality smooth surface GaN film.[16]

The GaN layers are mainly grown by epitaxial techniques such as hydride vapor phase epitaxy (HVPE),[8] metal organic chemical vapor deposition (MOCVD),[9,10] plasma assisted molecular beam epitaxy (PA-MBE)[11] and laser molecular beam epitaxy (LMBE).[12,17,18] Generally, the HVPE and

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MOCVD techniques employ a high deposition temperature for the growth of GaN layers. The high growth temperature may lead to formation of unwanted interfacial compounds or alloys, which may degrade the functionalities of the substrate, film and device performance. For most of the device applications, a sharp or abrupt interface at the substrate-overlayer is essential. In this respect, a low temperature growth will minimize the formation of thick interfacial compounds or alloys. The LMBE has advantage over MOCVD and PA-MBE techniques in that the growth of GaN film can occur at a low deposition temperature with moderate deposition rate due to high-kinetic energy film precursors produced by laser ablation.[4,18] The LMBE technique has been previously used to grow epitaxial GaN films on nearly lattice-matched substrates such as 6H-SiC (0001), ZrB_2 and (Mn,Zn)Fe_2O_4 (111) substrates at room temperature.[4,14,19] However, the reports on high quality growth of GaN epilayers on sapphire substrates are very limited.

In this paper, we report on the growth of high crystalline quality GaN films on pre-nitridated sapphire (0001) substrates by employing a HVPE grown high pure GaN bulk target in an ultra-high vacuum (UHV) LMBE system and discuss the effect of growth temperature on the structural properties of the grown GaN layers. A lowest x-ray rocking curve full width at half maximum of 102 arc sec has been achieved for GaN film grown at 720 °C. Micro-Raman studies also complimented the high crystalline quality with sharp characteristic peaks related to c-plane wurtzite GaN for film grown by LMBE.

II. EXPERIMENTAL DETAILS

The growth of epitaxial GaN films on pre-nitridated sapphire (0001) substrates were carried out in an UHV-LMBE system (SVT Associates, Inc. US) equipped with reflection high energy electron diffraction (RHEED), laser ablation targets, and a radio-frequency (RF) nitrogen plasma source to supply nitrogen radicals to the surface. The base pressure in the GaN growth chamber was better than 2×10^{-10} Torr. The back side of the sapphire substrate was coated with a layer of molybdenum of about 1 μm thickness to increase the absorption of heat radiation and uniform heat distribution. The substrates were cleaned using standard organic solvents and de-ionized water. The Mo-coated sapphire substrate was heated directly by radiation from a resistive heater. Prior to growth, the substrate was degassed for several hours at 250 °C in the preparation chamber of the LMBE system, followed by thermal cleaning at 850 °C for 10 min in the growth chamber. The sapphire surface nitridation was performed at 700 °C with RF plasma power of 400 W and N_2 gas flow of 1.2 sccm. A high purity polycrystalline HVPE grown GaN target (99.9999%) was ablated using a KrF excimer laser (248-nm wavelength, 25 ns pulse) with an energy density of ~5 J/cm^2 and a frequency of 10 Hz. The laser ablated GaN plume consists the energetic species such as GaN, GaN_{1-x} and Ga.[12] The GaN film was previously grown on sapphire substrates using solid GaN target in the presence of either NH_3 atmosphere or without any nitrogen source.[12,17] The nitrogen deficiency can be expected in LMBE grown GaN samples without the presence of adequate nitrogen flux during ablation of GaN target. In this work, the nitrogen radicals were supplied through the RF nitrogen plasma cell for stoichiometric GaN growth. Due to the low background pressure ($< 2 \times 10^{-10}$ Torr), the presence of any oxygen impurity or other residual gas contamination will also be minimized. We grew GaN for 2 hours on nitridated sapphire substrates at different growth temperatures ranging between 500 and 750 °C keeping all other parameters constant. The epitaxial GaN growth was monitored *in-situ* using RHEED. The thickness of grown GaN films on sapphires are measured using a stylus profilometer as 280 nm, 280 nm, 240 nm, 160 nm and 40 nm for growth temperatures 500 °C, 600 °C, 700 °C, 720 °C and 750 °C, respectively.

A PANalytical X'Pert PRO MRD HR-XRD system, with CuKα1 radiation, was employed to characterize the crystalline quality of GaN films grown on sapphire substrate. The rocking curve of the GaN layers for (0002) diffraction planes was recorded using a hybrid monochromator (multilayer graded mirror with channel-cut 2-bounce Ge (220) monochromator) in incident beam configuration. A scintillation detector was used to record the diffracted beam from the specimen during rocking curve and phi scan measurements. A Multimode-V Veeco AFM was employed in tapping mode to characterize the surface morphology of GaN films on sapphire using silicon tips of curvature radii less than 10 nm. The micro-Raman spectra were collected with a triple monochromator spectrometer

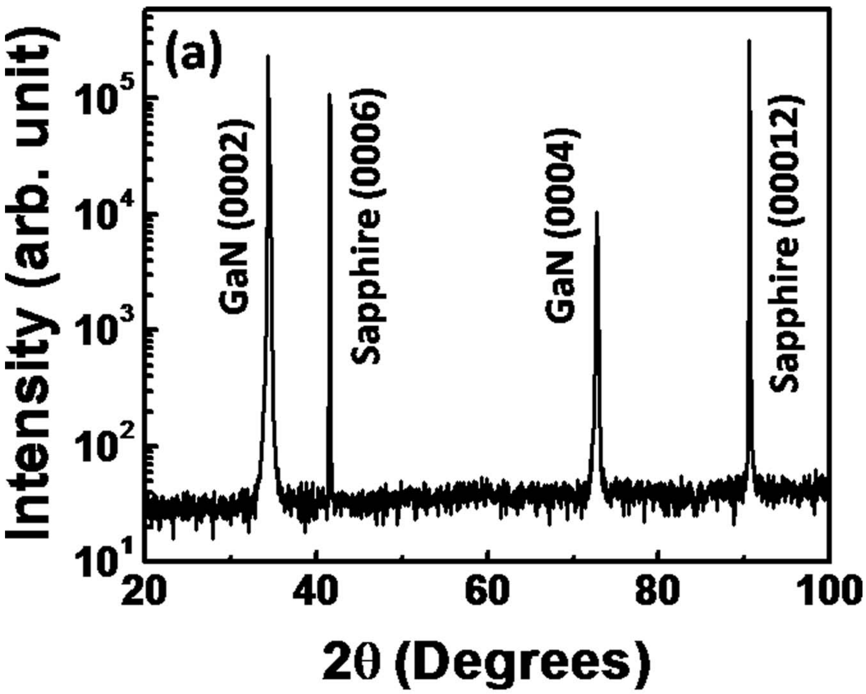
(T-64000, Jobin-Yvon/Horiba group) at room temperature using an excitation source of 514.5 nm wavelength with resolution of wave number 0.1 cm^{-1}. The secondary ion mass spectroscopy (SIMS) data was acquired by using time of flight (TOF)-SIMS 5 (ION-TOF GmbH Germany). The pulsed primary ion beam of Bi^+ with 25 KeV energy was incident on the sample to produce secondary ions. The Bi current was kept at 1.1 pA with a dose density of 8.72×10^{13} ions/cm^2. On the other hand, the depth profile data was obtained by sputtering the sample by 1 KeV Cs^+ ions. Cs current was 86.1 nA with a dose density of 4.81×10^{17} ions/cm^2.

III. RESULTS AND DISCUSSION

The XRD 2θ scans of all GaN films grown on pre-nitridated sapphire at various temperature ranging from 500 °C to 750 °C showed only {000l} family of planes of wurtzite GaN and sapphire. Figure 1(a) shows a typical XRD 2θ scan of LMBE grown GaN film on sapphire (0001) substrate. The XRD pattern clearly shows that the LMBE grown GaN film on sapphire has a high degree of texturing along [0001] normal to the sapphire. This indicates the heteroepitaxial relationship between $(0001)_{GaN}$ and $(0001)_{sapphire}$. The degree of in-plane alignment of GaN relative to the sapphire (0001) substrate is determined by ϕ-scan curve as shown in Fig. 1(b). The scanning planes used in ϕ-scan were (10-1 -10) for sapphire and (10–12) for GaN. The diffraction peaks from the (10–12) plane of GaN film were observed at intervals of 60° as seen in Fig. 1(b), which confirms the hexagonal structure of the epitaxial GaN film grown on sapphire. From Fig. 1(b), it is observed that there is a 30° rotation of the GaN unit cell with respect to sapphire due to the large lattice mismatch between GaN and sapphire (∼14%). This indicates the in-plane epitaxial relationship of GaN grown on sapphire is $(10\text{–}10)_{GaN}$ / $(11\text{–}20)_{sapphire}$.

The x-ray rocking curves of GaN films grown on sapphire at different temperatures were measured for (0002) GaN plane. The full width at half maximum (FWHM) of the GaN (0002) peak as a function of growth temperature is shown in Fig. 2(a). The (0002) plane x-ray rocking curve FWHM of GaN layer grown at 500 °C is 1582 arc sec. However, the value dramatically decreased to 153 arc sec when the growth temperature was increased from 500 °C to 600 °C indicating the high crystalline quality. The FWHM value further decreased with the increase of growth temperature and a lowest value of 102 arc sec has been achieved for the GaN layers grown at 720 ºC. The previous best reported value for LMBE GaN film on sapphire was 420 arc sec grown by laser ablating a polycrystalline GaN solid target under NH_3 ambient at growth temperature of 950 °C.[12] The LMBE GaN films on sapphire grown using laser ablation of Ga liquid target in the presence of nitrogen flux showed a FWHM of ∼1260 arc sec for GaN (0002) reflection.[20] The improvement in crystalline quality of GaN films with increase in growth temperature is due to the thermally activated surface processes such as reduced diffusion barrier, relaxed surface stress of the films and fast diffusion rate for GaN, Ga and nitrogen atoms or clusters on substrates. The x-ray rocking curves in Fig. 2(b) indicate that the LMBE grown GaN films on sapphire above 600 °C have a high crystallinity with an extremely low orientational spread in the direction normal to the surface. The high quality GaN crystalline film can be attributed mainly to the ablation of high purity HVPE grown polycrystalline GaN solid target under additionally supplied nitrogen radicals during the GaN growth. However, the FWHM for the GaN film grown at 750 °C was as large as ∼610 arc sec, where the layer thickness was ∼40 nm. As also shown in Fig. 2(a), the GaN growth rate decreased with increasing substrate temperature and might be due to increased desorption of Ga from the growing GaN surface at higher temperatures.[12]

It has been observed that the epitaxial GaN films grown on sapphire exhibit a high dislocation density.[10–12,21–24] The x-ray rocking curves have been widely used indirectly to quantify the presence of dislocation in the GaN epitaxial film.[25] It has been reported that the FWHM of the (0002) plane reflects lattice distortion from screw dislocations and mixed dislocations, while that of the asymmetric (10–12) plane FWHM is associated with lattice distortion from edge dislocations and mixed dislocations.[21–25] Since both symmetric and asymmetric planes are sensitive for mixed dislocation densities, the overestimation of total dislocation density can not be ignored because one mixed dislocation combines the displacement of one screw plus one edge dislocation. Further modeling and careful simulation required for accurate estimation of dislocation densities using x-ray rocking



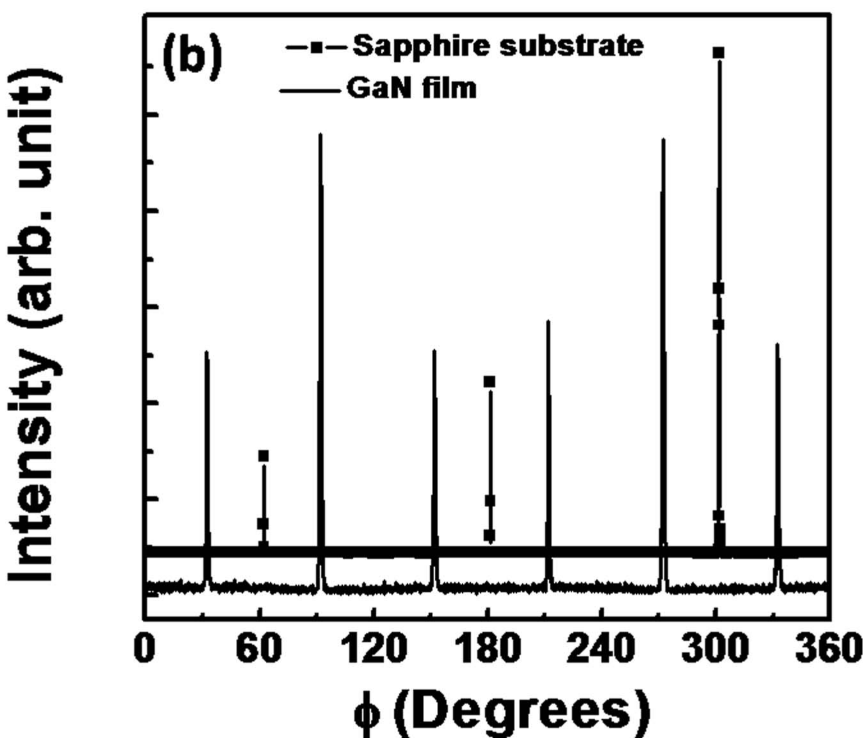
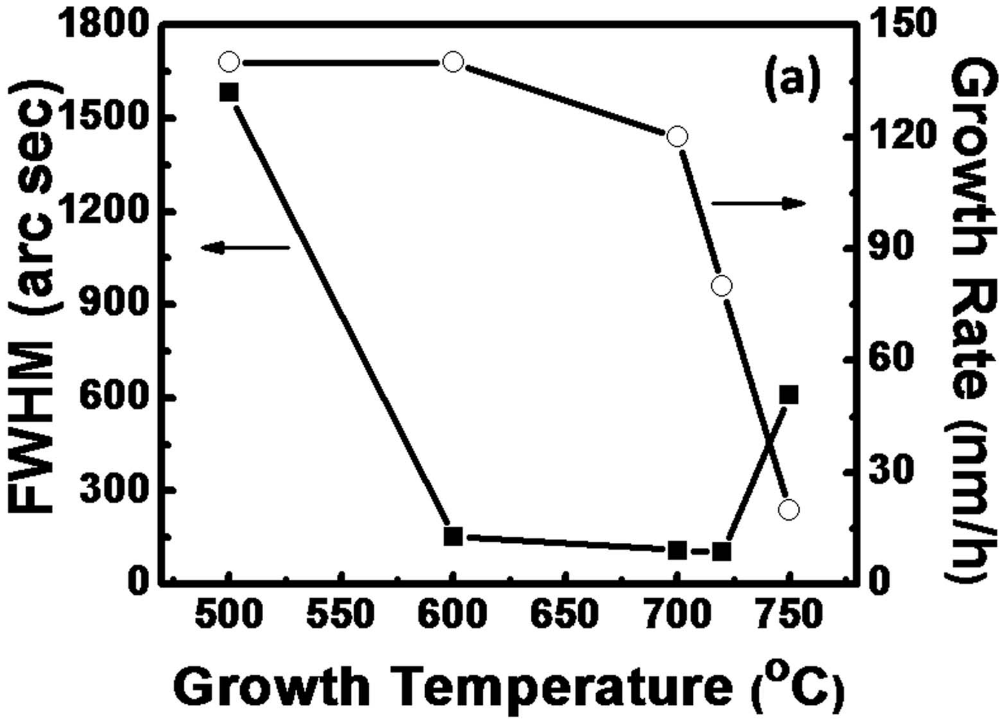


FIG. 1. (a) XRD 2θ scan of GaN grown on sapphire (0001), (b) XRD in-plane ϕ-scan of (10-1 -10) plane of sapphire (0001) substrate and (10–12) plane of grown GaN layer.



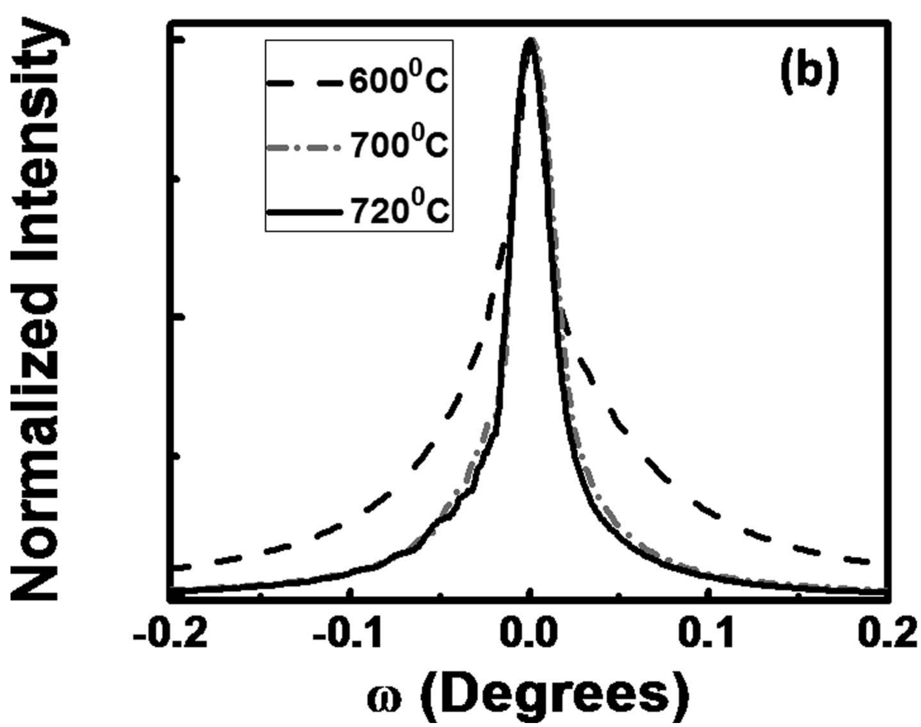


FIG. 2. (a) X-ray rocking curve FWHM of the GaN (0002) plane and GaN growth rate as a function of growth temperature; (b) Normalized x-ray rocking curves of GaN (0002) plane grown at 600 °C, 700 °C and 720 °C.

curves. Recently many researchers used FWHM value of (0002) plane for screw dislocation density estimation and (10–12) plane FWHM value to quantify the edge dislocation density.[21,23,25] We have estimated the dislocation density in LMBE grown GaN (0001) film on sapphire at 720 °C using the following equations:[21–23]

$$D_{screw} = \frac{\beta^2_{(0002)}}{9b^2_{screw}}, \quad D_{edge} = \frac{\beta^2_{(10-12)}}{9b^2_{edge}} \tag{1}$$

$$D_{dis} = D_{screw} + D_{edge} \tag{2}$$

where D_{screw} is the screw dislocation density, D_{edge} is the edge dislocation density, β is the FWHM values measured for (0002) and (10–12) planes by HR-XRD rocking curves and b is the Burgers vector length ($b_{screw} = 0.5185$ nm, $b_{edge} = 0.3189$ nm). The values for $\beta_{(0002)}$ and $\beta_{(10\text{–}12)}$ obtained from HR-XRD rocking curves for GaN grown at 720 °C are 0.028° and 0.4°, respectively. The dislocation densities calculated from (0002) and (10–12) planes are 1×10^7 and 5.31×10^9 cm^{-2}, respectively. The two order higher value of dislocation density calculated from (10–12) plane indicates that the dislocations present in our GaN layer are predominantly edge type.

Surface morphologies of the GaN film grown on sapphire at different growth temperatures were investigated using AFM in tapping mode and are presented in Fig. 3. For the GaN film grown on sapphire at 500 °C in Fig. 3(a), mostly small grains of size about 75–85 nm with hazy features were obtained. Fig. 3(b) shows the surface morphology of the GaN film grown at 600 °C on sapphire and shows very uniform grains of size 80–85 nm. It has been observed from HR-XRD and AFM characterizations that good crystalline film quality is only obtained at growth temperature higher than 500 °C. The lateral size of the grain is large (∼200 nm–280 nm) as shown in Fig. 3(c) for GaN film grown at 700 °C. With further increase of the growth temperature, the lateral size of the grain increased and hexagonal crystalline GaN structures with flat facets were obtained at 720 °C with a grain size of 200–300 nm as shown in Fig. 2(d). It should be noted that these grain features with a height of 15–20 nm are present on the surface of 160 nm thick GaN layer. It is well known that the high temperature growth promotes a two dimensional growth improving the surface flatness and the coalescence of crystallite islands due to enhanced migration of surface adatoms. The differences in the surface morphologies of GaN on sapphire at different growth temperatures are related with the diffusivity and characteristic coalescence time of GaN clusters on sapphire. Surface diffusion is a thermally activated process and normally depends on the growth temperature as:[26]

$$D(T) = D_0 \exp\left[{}^{-E_a}\!/_{kT}\right] \tag{3}$$

where $D(T)$ is surface diffusion coefficient, D_0 is the attempt frequency, E_a is an activation energy, k is the Boltzmann constant and T is the growth temperature. The surface diffusion coefficient increases with growth temperature. For a given island or grain size, the characteristic coalescence time is inversely proportional to the diffusion coefficient.[27] The shorter coalescence time is the reason why bigger GaN grains were obtained on sapphire at higher growth temperature in comparison to low growth temperature in the same growth conditions. However, a further increase in growth temperature to 750 °C, showed grains of random sizes varying from 180 to 300 nm, as shown in Fig. 3(e). It is likely that, at 750 ºC, GaN strongly desorbs from the growing surface as indicated by the lower growth rate thus, resulting in non-uniform growth. This indicates that the growth of GaN film on sapphire using LMBE at growth temperature of 750 °C or higher is not favorable due to lack of reasonable growth rate.

The HR-XRD and AFM results on the sample grown at 700 °C and 720 °C are very similar. In order to identify the better structural quality of GaN films at these growth temperatures, we performed micro-Raman spectroscopy on these samples. Fig. 4 shows the room temperature micro-Raman spectra of the GaN films on sapphire substrates grown at 700 °C and 720 °C. The Raman peaks of GaN films at 568.6 cm^{-1} and 736 cm^{-1} correspond to E_2 (high) and A_1(LO) phonon modes, respectively. These modes are the allowed vibrational states for the wurtzite phase GaN (0001) epitaxial layer when measured in the back scattering geometry.[28] The shoulder peaks at 576 and 751 cm^{-1} arise from the sapphire substrate.[29] The FWHM of the E_2 (high) signal for GaN film

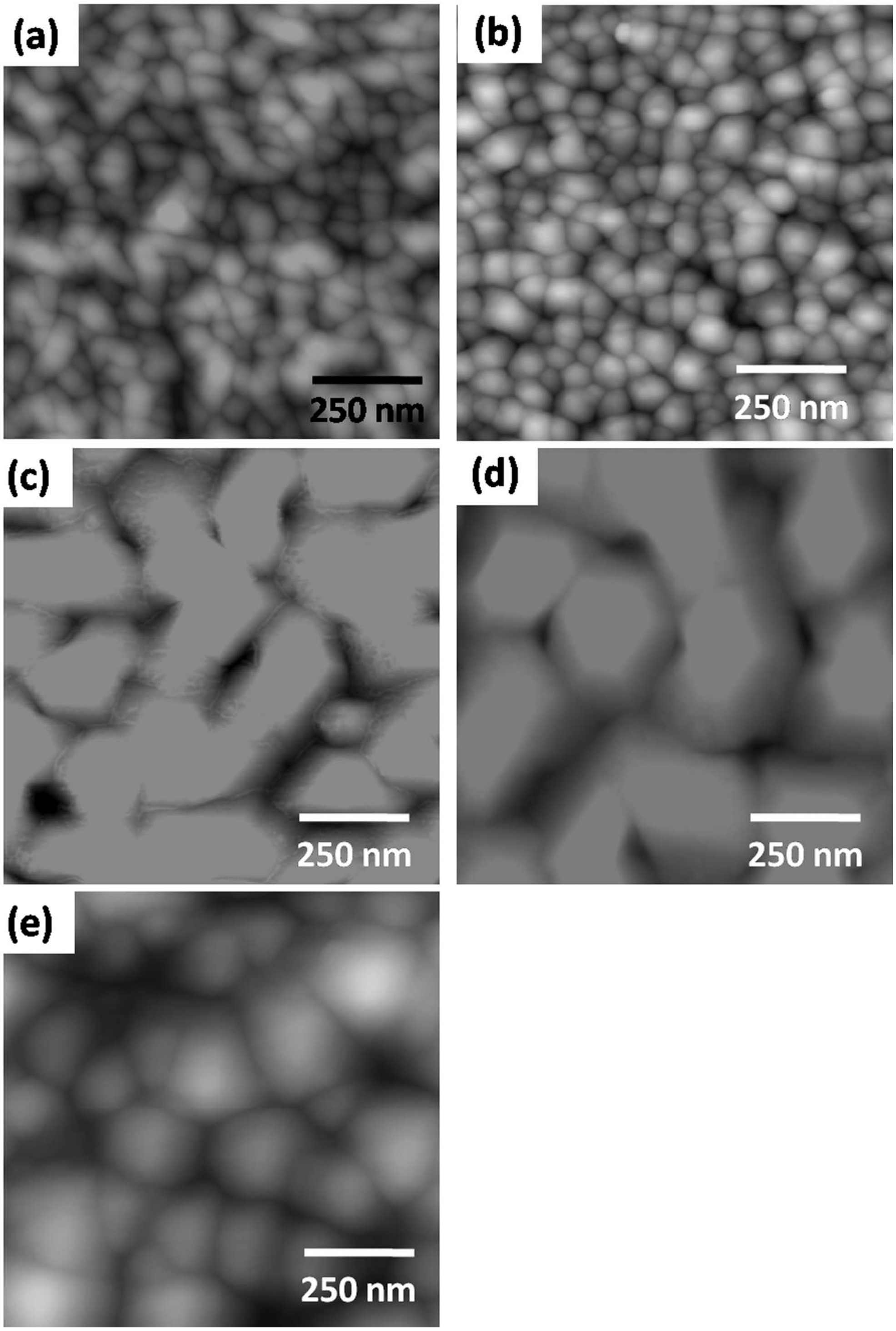


FIG. 3. AFM images of GaN films grown on sapphire at different growth temperatures: (a) 500 °C, (b) 600 °C, (c) 700 °C, (d) 720 °C and (e) 750 °C. Scan area: 1 μm × 1 μm.

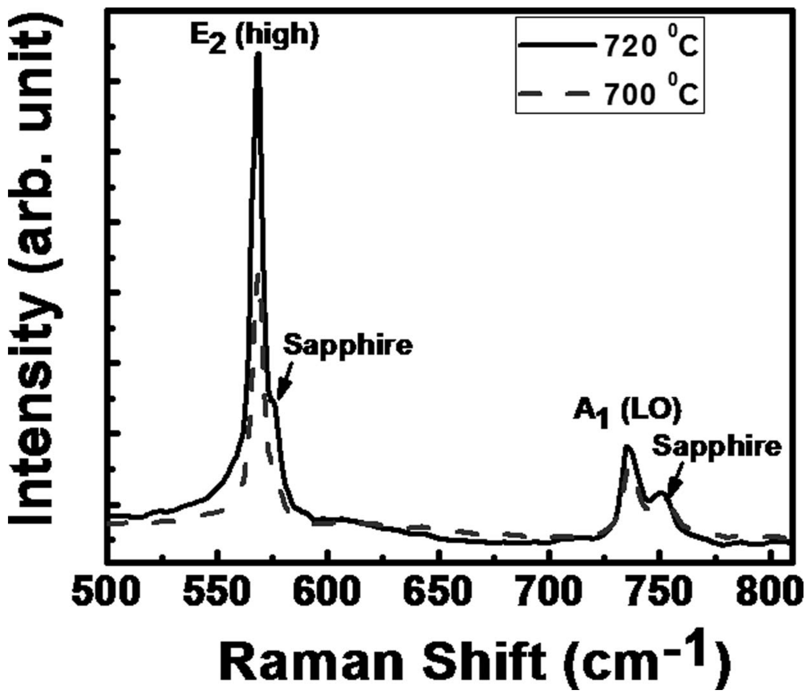


FIG. 4. Room temperature Raman spectra of GaN films grown on sapphire at 700 and 720 °C.

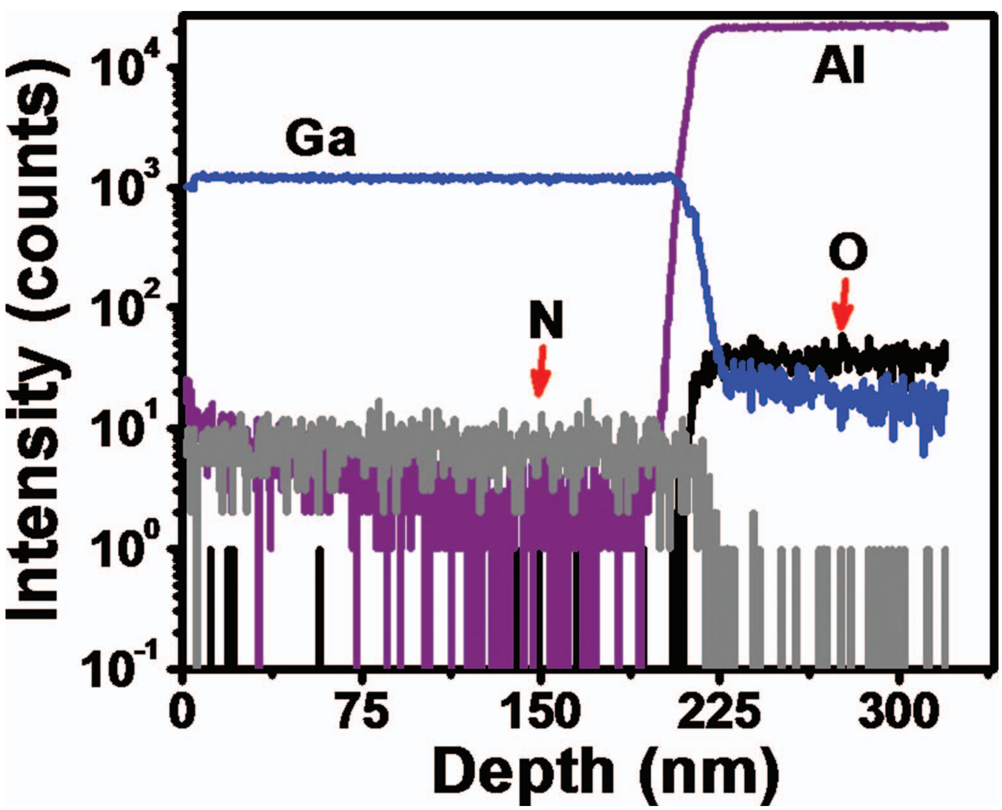


FIG. 5. The SIMS depth profile on GaN film grown on sapphire substrate.

grown at 700 °C is 5.4 cm^{-1} whereas that for GaN film grown at 720 °C is 5.1 cm^{-1}. The E_2 (high) peak for the sample grown at 720 °C is narrower than the sample grown at 700 °C. The HR-XRD, AFM and Raman spectroscopy results show that the GaN film grown at 720 °C has the highest quality crystalline properties.

Pulsed laser deposited GaN layers are generally found with substantial levels of oxygen contamination as the sintered GaN targets contain trapped oxygen.[30] We have performed TOF-SIMS depth profile analysis to find the impurity levels present in our grown GaN film. Figure 5 shows the SIMS depth profile of a typical GaN film grown on sapphire using LMBE. In the GaN film region, only Ga and N species were present. The low intensity of N is because of its low sensitivity factor for detection by Bi^+ ion. The presence of oxygen impurity is below the detectable limit in the GaN film. It has also been observed that the carbon incorporation level in the GaN film is below the detection limit (not shown). The lack of oxygen impurity or other residual gas contamination is achieved due to the low background pressure in the growth chamber as well as the use of high purity HVPE grown GaN solid target.

IV. CONCLUSIONS

We have grown epitaxial GaN films on pre-nitridated sapphire (0001) substrates at various growth temperatures ranging from 500 to 750 °C using a LMBE system. The FWHM of the x-ray diffraction rocking curve of GaN film decreases with growth temperature from 1582 arc sec (500 °C) to 102 arc sec (720 °C), indicating high crystalline quality growth at higher temperatures. AFM studies also showed that the average grain size of the GaN film increases with growth temperature and flat hexagonal islands of size 200–300 nm are observed for the GaN layer grown 720 °C. Raman spectroscopy studies also complimented the growth of high quality wurtzite GaN films at 720 °C. The SIMS measurement revealed no oxygen background impurity in the GaN film as a result of using high purity HVPE grown GaN bulk target and UHV environment. These results demonstrate the capability of LMBE to produce device quality GaN epilayers at a relatively lower temperature compared to other conventional epitaxial techniques.

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