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Dislocation reduction in GaN film using Ga-lean GaN buffer layer and migration enhanced epitaxy

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A B S T R A C T

A GaN buffer layer grown under Ga-lean conditions by plasma-assisted molecular beam epitaxy (PAMBE) was used to reduce the dislocation density in a GaN film grown on a sapphire substrate. The Ga-lean buffer, with inclined trench walls on its surface, provided an effective way to bend the propagation direction of dislocations, and it reduced the dislocation density through recombination and annihilation processes. As a result, the edge dislocation density in the GaN film was reduced by approximately two orders of magnitude to $2\times10^{8}\ \mathrm{cm}^{-2}$. The rough surface of the Ga-lean buffer was recovered using migration enhanced epitaxy (MEE), a process of alternating deposition cycle of Ga atoms and N_2 radicals, during the PAMBE growth. By combining these two methods, a GaN film with high-crystalline-quality and atomically-flat surface can be achieved by PAMBE on a lattice mismatch substrate.

1. Introduction

GaN materials are usually grown on foreign substrates, such as sapphire, silicon or silicon carbide, because a large, low-cost, commercial-grade native substrate is still not available. The quality of the GaN film is therefore critically dependent on the ability of the transition layer used to accommodate the stress generated from large mismatches in the lattice constant and thermal expansion coefficient. Generally, GaN films grown using metal-organic chemical vapor deposition (MOCVD) have higher crystalline quality than do those grown by molecular beam epitaxy (MBE) processes, including plasma-assisted MBE (PAMBE) [1]. For MOCVD growth, the transition from a three-dimensional low-temperature (525–600 °C) GaN nucleation layer to a two-dimensional GaN growth on a sapphire substrate during the substrate temperate ramp-up to a higher process temperature (1060–1080 °C) can effectively reduce the stress and improve the crystalline quality of a GaN film [2]. On the other hand, the PAMBE growth process cannot achieve this transition due to the much lower growth temperature (700–800 °C) used for the growth of a GaN layer. Therefore, the high dislocation density generated as a result of a mismatch in lattice constant between the GaN film and the substrate will propagate into the active GaN layer. However, some of the best GaN devices have been grown by MBE on high-quality GaN templates [3,4]. Those results have proven the ability of an MBE growth process to achieve a GaN film and device structure suitable for high-performance devices if an intermediate buffer layer is available.

MBE is one of the major epitaxial growth techniques used to realize GaN materials and device structures. It provides several advantages, such as real-time monitoring of material growth using reflection high-energy electron diffraction (RHEED), a carbon- and hydrogen-free growth environment, a smooth surface and sharp hetero-interfaces. Furthermore, GaN films can also be grown under a wide growth regime using MBE through the careful control of growth parameters, i.e., temperature and Ga/N flux ratios. Recently, the GaN buffers grown using MBE under nitrogen stable conditions have attracted attention due to the improved crystalline quality of the GaN film. These GaN buffers were grown either in slightly nitrogen stable

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[5–7] or under nitrogen-rich [8–10] growth conditions. Here, we refer to the former as the “Ga-lean” condition to differentiate between the slightly nitrogen stable condition and the nitrogen-rich condition which is normally referred to as the highly nitrogen-rich condition.

The GaN buffer prepared under Ga-lean conditions by MBE exhibits a rough surface with a hill and valley topography. However, this rough surface promotes the bending of dislocations that tend to grow perpendicularly to the inclined material surface, and it also enhances their self-interactions [6]. The interaction reduces dislocations through recombination and annihilation processes. Therefore, a greater reduction of dislocations may be realized by using an intentionally created buffer with a rougher surface morphology. Nevertheless, recovering a GaN surface with an overly rough morphology is rather difficult due to the slow lateral growth rate of MBE, even when the growth conditions are shifted to a Ga-rich environment [5,6]. On the other hand, a GaN buffer with a nanocolumn structure can be grown under the nitrogen-rich condition. Studies have revealed that a very low dislocation density was generated in these individual GaN nanocolumns [9]. However, the use of this buffer structure has created other concerns. First, the surface morphology of a nanocolumn buffer is very rough. This makes it difficult, in the subsequent growth step, to achieve a material with a smooth surface for device fabrication, especially for MBE. Therefore, the MOCVD method, which is associated with a much faster lateral growth ability, is always used to recover the rough surface morphology of the nanocolumn buffer with a sufficiently thick (several micrometers) overgrown GaN layer [11]. In addition to the complications associated with this approach (using more than one growth chamber), the final quality of the overgrown layer is also somewhat inferior. The inferior quality is a consequence of individual nanocolumns growing on the substrate at slight disorientation with respect to each other. Studies have shown that these small disorientations produce a high density of threading dislocations (TDs), stacking faults, and grain rotations along the growth direction [10,11] in the overgrown layer.

In this work, a Ga-lean GaN buffer was used to reduce the dislocation density in GaN grown by MBE on a sapphire substrate to avoid the problems of using a nanocolumn buffer. To enhance the dislocation reduction effect, a prolonged growth period of the Ga-lean layer produced a rougher buffer surface. After that, migration enhanced epitaxy (MEE), which is composed of an alternative deposition of Ga atoms and N_2 radicals, was employed to recover the rough Ga-lean buffer surface. The MEE method is known to be able to enhance the migration distance of Ga atoms on GaAs surfaces to achieve layer-by-layer growth [12]. In this study, the usefulness of MEE to recover a rough GaN surface and the effectiveness of a Ga-lean buffer to reduce dislocation density were investigated.

2. Experimental procedures

The growth of all epitaxial layers was accomplished using PAMBE (ULVAC MBE System) on sapphire (0001) substrates. The initial growth procedure, namely the thermal cleaning of the substrate, the nitridation, and the growth of a thin AlN (15 nm) buffer layer, was previously described in detail [13]. After the growth of a thin AlN buffer, the substrate temperature was set to 740 °C for the growth of the Ga-lean GaN buffer. During the growth of this layer, the effective flux ratio for Ga/N of ~0.75 was used. Here, we define an effective flux ratio of one at the point where a rough to smooth transition is seen by RHEED. The Ga-lean buffer was grown to a thickness of ~700 nm followed by MEE to recover the rough Ga-lean buffer surface. During the MEE, Ga atoms and nitrogen radicals were impinged onto the substrate alternatively for 10 s in each half-cycle. This was achieved with a programmable logic control (PLC) that has been designed to control the operation of mechanical shutters in front of the growth sources (i.e., Ga and nitrogen radicals). The Ga/N ratio was set to that used to produce a smooth GaN surface, i.e., one during the MEE growth, and the growth temperature was also set at 740 °C.

To study the effect of MEE on a rough GaN surface, growth interruptions were introduced. The first growth stop was carried out after the Ga-lean buffer was deposited. The completion of this layer was indicated by the change in RHEED pattern from streaky lines (Fig. 1(a)) to broken, downward arrow lines (Fig. 1(b)). This change suggested that the GaN buffer surface was flat at the beginning and transformed gradually into a rough surface with a specific morphology that will be described later. The sample was removed from the chamber, and the GaN surface morphology was characterized using an atomic force microscope (AFM, Dimension 3100, Veeco) with a silicon tip that was operated in tapping mode. Additionally, a high resolution X-ray diffraction (HRXRD, Bede D1) system equipped with a high power (2 kW) X-ray source using the Cu K_α line was also used to determine the crystal quality of GaN film. The sample was then cleaned with organic solvents and diluted hydrochloric acid prior to the GaN re-growth using the MEE method. This procedure was repeated several times until the GaN surface was fully recovered. Finally, another GaN layer (~1.5 μm), grown with an effective Ga/N ratio of one, was deposited. The total thickness of the sample was approximately 2.5 μm. This sample was designated at Sample A. For comparison, a control sample (Sample B) with a similar thickness but without the Ga-lean buffer was also prepared. In this sample, the GaN layer was grown with an equal Ga/N ratio to one throughout the process. In addition to HRXRD, the crystal quality of these GaN samples was also determined using cross-sectional transmission electron microscopy (TEM, JOEL 2010 F) operated at 200 kV. The TEM samples were prepared by cleaving the GaN materials along the (11–20) plane, followed by conventional mechanical polishing and an Ar-ion milling process.

3. Results and discussion

The evolution of GaN surface morphology in Sample A at different MEE cycles was studied by atomic force microscopy. The AFM measurements (Fig. 2 (a)) show that the initial Ga-lean GaN buffer surface was decorated with flat, truncated mesas and deep trenches up to ~100 nm, which correspond with the RHEED pattern shown in

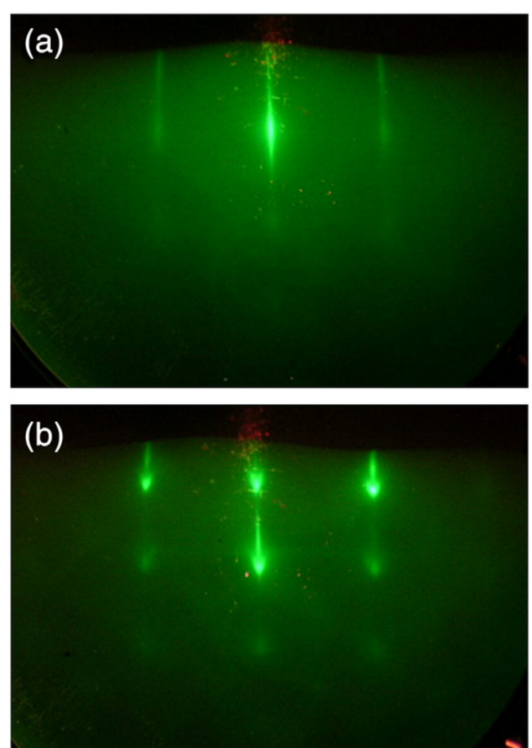

Fig. 1. RHEED patterns of (a) smooth GaN surface and (b) rough Ga-lean GaN buffer surface.

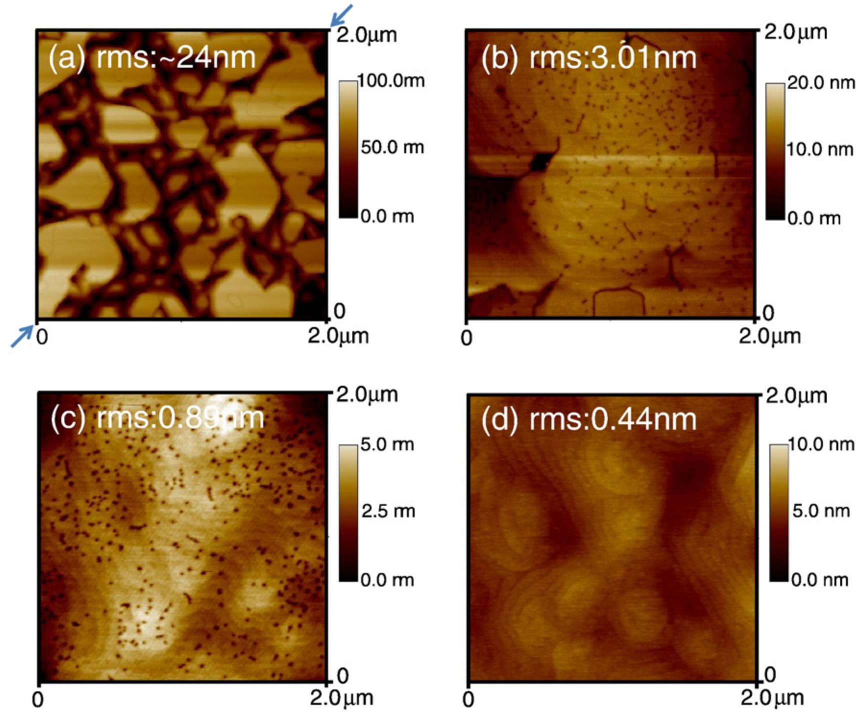

Fig. 2. AFM images of the Ga-lean GaN surface after (a) 0, (b) 300, (c) 600 and (d) 800 cycles of MEE growth. A cross-sectional profile across the diagonal in image (a) (indicated by the arrows) is shown in Fig. 6 (b).

Fig. 1(b). After the first 300 cycles of MEE, most of the deep trenches were recovered, and only short, shallow trenches and pits were left (Fig. 2 (b)). The RHEED pattern remained less streaky even though the arrow marks vanished. The trenches on the GaN surface were entirely eliminated by an additional 300 MEE cycles (Fig. 2 (c)), leaving only small pits on the sample surface. The RHEED pattern appeared to be streaky during the MEE process, but the pattern became less streaky again after the MEE process was stopped and the sample was annealed for a short duration. Finally, the GaN surface was fully recovered (Fig. 2 (d)) with an additional 200 MEE cycles. The corresponding RHEED pattern of this smooth GaN was streaky and similar to that shown in Fig. 1(a). The root-mean-square (*rms*) roughness was improved from 24 nm to 0.44 nm. This demonstrates the ability of MEE to recover a rough GaN surface. The roughening of a GaN surface grown under Ga-lean conditions was a result of the low surface diffusivity of adatoms. It has been calculated that the diffusion barrier for Ga adatoms on a highly N-rich surface is as high as 1.8 eV, whereas it is only 0.4 eV on a Ga-saturated surface [14]. We believed that the diffusion barrier for Ga adatoms on a Ga-lean surface is somewhere between these two values. The reduced mobility of adatoms was responsible for the GaN surface roughening, as shown in an early work by Tarsa et al. [15].

The results suggest one potential mechanism by which MEE recovers the Ga-lean buffer. As illustrated in Fig. 3 (a), the inclined trench surfaces on a rough Ga-lean buffer can be represented by a series of steps edges and kinks sites. Due to the abundance of unsaturated bonds, the Gibbs free energy of these sites was higher than that of the flat mesas, which favored adatom residence [16]. During the first MEE half-cycle in which only Ga atoms were deposited, new Ga-N bonds did not form, and the Ga atoms were relatively free to move. They had sufficient time to diffuse along the GaN surface, but most of them would be trapped at the kink sites where the surface energy was higher (Fig. 3 (b)). During the second MEE half-cycle, the trapped Ga atoms reacted with nitrogen radicals to form new GaN material (Fig. 3 (c)). On the other hand, nitrogen radicals arrived at the mesa areas without free Ga atoms would stay uncombined and desorbed from the mesa areas. Therefore, the growth rate at trench areas was significantly faster than at the flat mesas, and the GaN surface became smooth after a sufficient number of MEE cycles (Fig. 3 (d)).

In our experiments, the MEE half-cycle was relatively long (10 s), compared with those used by Horikoshi (1 s, to grow GaAs) [12] or by Lu et al. (2 s, to grow InN) [17]. This minimized the risk of mechanical failure for the shutters that might be caused by rapid operation. However, we found that this was sufficient to recover the rough GaN surface. The results indicate that, although most of the Ga adatoms had desorbed from flat mesa surface, those Ga adatoms trapped at step edges and kinks sites on the trenches survived until N_2 radicals arrived. In addition, the surface morphology of the GaN film exhibited step-flow growth (Fig. 2 (d)). The average surface step height of 0.25 nm, which is very close to the 0.26 nm step height of one (0001) GaN monolayer, was a result of enhanced Ga adatom migration

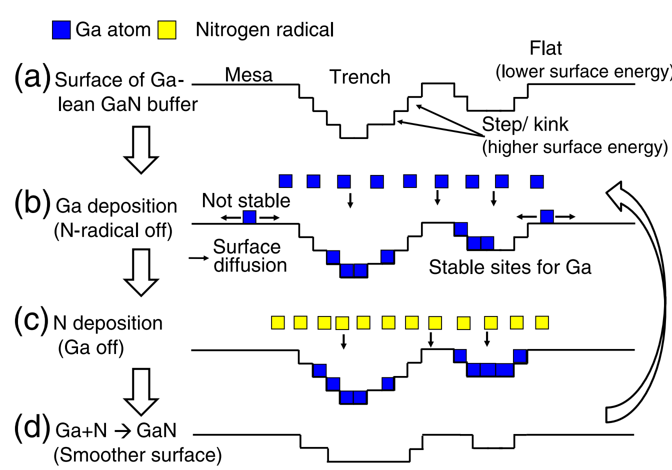

Fig. 3. The proposed mechanism of MEE to recover the rough GaN surface grown under Ga-lean condition. (a) A Ga-lean GaN buffer surface covered with truncated mesas and deep trenches, (b) deposition of Ga atoms during the first half-cycle of MEE, (c) deposition of nitrogen radicals during the second half-cycle of MEE, (d) a smoother GaN surface after the MEE process.

distances on the GaN surface [12]. These results demonstrate the use of MEE as a means to recover a rough GaN surface. The MEE method is commonly used during the growth of GaAs material, and it has been shown to enhance the migration distance of Ga atoms on the GaAs surface [12].

The GaN film's change in structural quality at different stages of growth was investigated using HRXRD. Both symmetric (0002) and asymmetric (10–12) X-ray rocking curves were measured from the GaN samples (Fig. 4 (a)) to estimate the threading dislocation (TD) density within each sample. Three types of TDs normally occur in GaN: screw TD (with Burgers vector $b = <0001>$, edge TD ($b = 1/3<11–20>$), and mixed TD ($b = 1/3<11–23>$). If the (0002) X-ray rocking curve is broadened by screw and mixed TDs, and the (10–12) X-ray rocking curve is broadened by all TDs (screw, edge and mixed) [18,19], then the dislocation density in GaN can be estimated from XRD results using the following equation [20]:

$$D_{dis} \sim \beta^2 / 9b^2$$

where D_{dis} is the TD density in the film, β is the full width at half-maximum (FHWM) of a given XRD peak, and b is the length of Burgers vector of the corresponding dislocation. Adopting methods from the literature [13,18], the screw and edge TD densities in GaN film can be calculated (Fig. 4 (b)). The horizontal axis in this figure represents GaN samples after different stages of growth. The displayed values represent the deposition cycles of MEE on Ga-lean buffer, whereas the rightmost one indicates the XRD results of Sample A, with a total GaN thickness of 2.5 μm. The XRD results for Sample B (without Ga-lean GaN buffer, leftmost in Fig. 4 (b)) with an FHWM of 113 and 2015″ for (0002) and (10–12) diffractions, respectively, are also included in the figure.

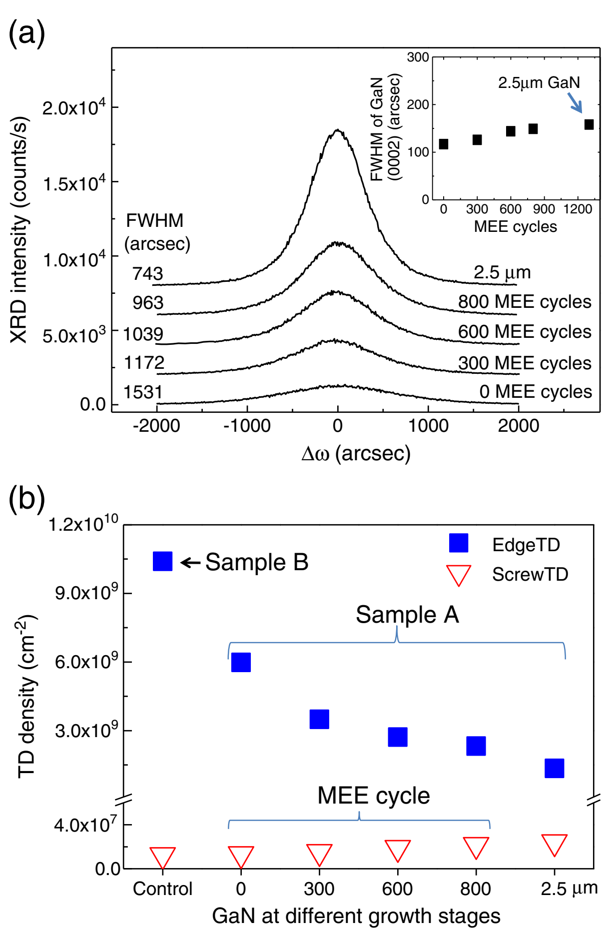

Fig. 4. (a) Asymmetric (10–12) rocking curves of the GaN film at different stages of growth. Inset shows the FWHMs of GaN symmetric (0002) deflection peaks after different MEE cycles. (b) Estimation of dislocation densities in GaN samples from the XRD results.

The reduction of TDs, in particular edge TDs, in the GaN material was clearly observed after using the Ga-lean buffer layer, and the TD number continued to decrease when the GaN was grown thicker. This additional reduction was a result of dislocation bending and annihilation. Dislocation bending was induced by roughening the GaN surface, which was initiated during the early stages of Ga-lean buffer growth. Dislocation bending brought dislocations closer to each other, allowing them to interact. During these interactions, the edge TDs with Burgers vectors of different signs could be recombined or annihilated [5,6]. Therefore, the crystal quality was improved with the increase of film thickness because more TD interactions occurred. In a previous study [13], we showed that, when GaN was grown on a rough AlN buffer prepared at low temperature, the edge TD density could be reduced from 1.5×10^{10} to 3.3×10^9 cm^{-2} by a similar mechanism. However, the AlN buffer grown at lower temperature also provided many nucleation sites, which acted as origins for screw TDs [21]. Therefore, the number of screw TDs increased by more than two orders of magnitude, from 4.1×10^6 to 6.17×10^8 cm^{-2}. Although the total dislocation density in GaN can be reduced with a low temperature AlN buffer, the increase of screw TD is undesirable because it has been shown to promote reverse-bias leakage current in GaN devices [22]. On the other hand, the XRD results estimated that the edge TD density of GaN samples in this study was reduced by approximately one order of magnitude from 1.0×10^{10} to 1.4×10^9 cm^{-2}, despite a slight increase in screw TD density from 1.2×10^7 to 2.4×10^7 cm^{-2}. This demonstrates the ability of Ga-lean GaN buffer to reduce edge TDs while suppressing the formation of new screw TDs if the MEE process takes place under optimal conditions which will be discussed below. Fig. 2 (a) shows that the Ga-lean surface was 'roughened' by trenches with inclined surfaces rather than by small three-dimensional mound-like features that could induce new screw dislocation formation. The slight increase in screw TD may be due to the non-optimized MEE process. If some of the Ga atoms deposited on the flat mesa areas of Ga-lean buffer did not diffuse into the trenches or re-evaporate from the surface during the first MEE half-cycle, they would form small GaN clusters upon the arrival of nitrogen radicals. These small GaN clusters would then generate new screw dislocations in the sample. Due to the specific Burgers vector, screw dislocations are difficult to reduce by dislocation bending. Therefore, the screw dislocation density should be kept as low as possible from the beginning of the growth process, and new dislocation formation should be suppressed during growth. A short period of annealing after every MEE half-cycle [17] may be needed to improve the incorporation of Ga atoms into the step and kink sites on the trench areas.

Cross-sectional TEM analysis was used to observe the bending behavior of TDs in GaN films grown on Ga-lean buffer. The dark-field TEM image of Sample A taken under two beam condition with g = [10-10] (Fig. 5 (a)) shows an abrupt change of dislocation density from the buffer region (below the dash line) to the GaN film. The boundary of Ga-lean buffer was estimated based on the growth rate of buffer. Under this observation condition, the edge TDs, which are the predominant dislocation type in GaN, and mixed TDs are visible. Fig. 5 (a) shows that most of the dislocations appeared to be confined within the buffer layer. In contrast, the dislocations in Sample B (Fig. 5 (b)) were propagated from the buffer to the GaN surface when a Ga-lean buffer was not used. The traces of TD bending that were discussed in the previous sections were hardly resolved in this region in Sample A because of the high dislocation density in the buffer. However, for material grown above the buffer layer, the TD bending was clearly observed in a higher magnification TEM image taken around the Ga-lean buffer region (Fig. 6 (a)). A number of bent TDs (labeled as BTD) were seen interacting with other TDs, and they were then recombined or annihilated via a mechanism similar to the TD reduction mechanisms discussed by Shen et al. [23]. The inclination angles of these bent TDs were between 10° and 50°. A cross-sectional surface

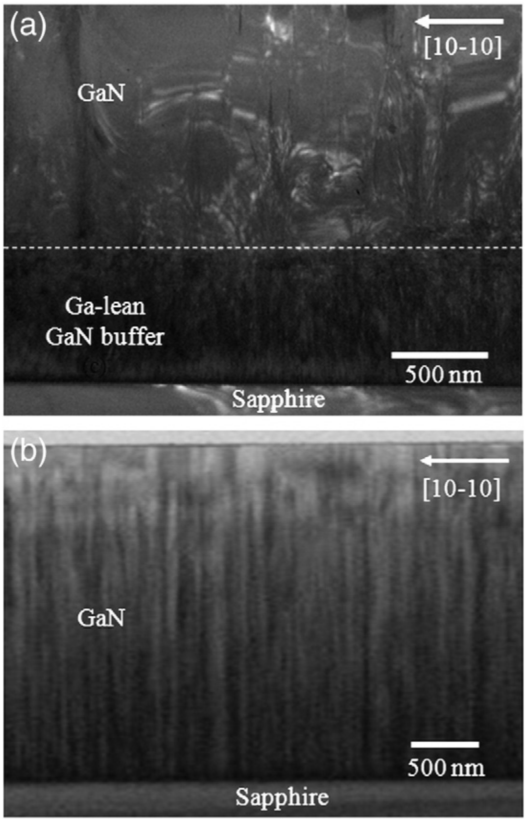

Fig. 5. Cross-sectional TEM images of GaN grown (a) with and (b) without Ga-lean GaN buffer layer. The dashed line in (a) shows the boundary between the Ga-lean buffer and GaN film.

profile of the Ga-lean buffer obtained from an AFM image (Fig. 2 (a)) is shown in Fig. 6 (b). It was found that the trench wall slopes on Ga-lean buffers matched well with the inclination angles of the bent TDs in the TEM image, suggesting that the inclined material surface induced TD bending. Fig. 6 (a) shows that the upper region of GaN film is relatively free of dislocations, with the exception of some TDs that grew vertically from the buffer to the sample surface (labeled as VTD). These dislocations might localize to a mesa center on the buffer and they did not interact with any other dislocation during the growth process. In the control sample, all TDs grew in this manner (Fig. 5 (b)) because an effective bending mechanism was not present.

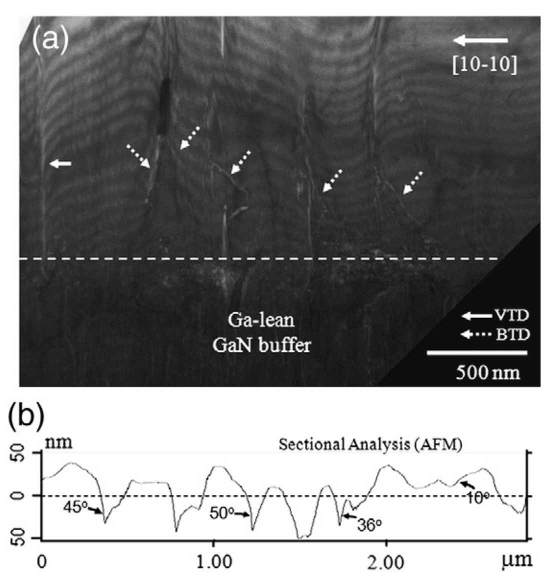

Fig. 6. (a) Cross-sectional TEM image of GaN film near the Ga-lean buffer region. The dashed line shows the top of the Ga-lean buffer layer. Arrows indicate the locations of vertical TDs (VTD) and bent TDs (BTD). (b) Cross-sectional profile obtained from the AFM image in Fig. 2(a). Some of the slopes on the profile are indicated.

From the TEM observations, the TD density in the upper portion of the GaN film in Sample A was 2×10^{8} cm^{-2}, which is almost two orders of magnitude lower than in the control sample. This value is also much lower than that reported by Manfra et al. (5×10^{9} cm^{-2}) [7] using a Ga-lean buffer with less significant trenches. This indicates that the dislocation reduction was greatly enhanced with a Ga-lean buffer with deeper trenches. More-prominent dislocation bending and dislocation reduction were both observed in Sample A, compared with in a sample grown on vicinal substrates (TD density ~4×10^{8} cm^{-2} [24]). In this study, we found a discrepancy in the estimated TD densities using TEM and XRD methods. TEM analysis showed a more pronounced effect of dislocation reduction due to the abrupt changes in TD density from the Ga-lean buffer to the GaN film. The XRD results over-estimated the dislocation density because its rocking curves values were averaged over the sample thickness. By considering the incident angle of the X-ray applied in this study and the absorption coefficient of GaN material, the penetration depth of the X-ray was estimated to be more than 7 μm [25].

4. Conclusion

The usefulness of MEE to recover a rough GaN surface has been demonstrated. With MEE, a Ga-lean GaN buffer with deeper trenches can be used to enhance the dislocation reduction in a GaN grown by MBE while maintaining the smoothness of the material surface. By using this Ga-lean buffer, the TD density in GaN was reduced by almost two orders of magnitude to 2×10^{8} cm^{-2}. Moreover, the generation of new screw TDs can also be suppressed by this method if the MEE process is optimized. Growth interruptions were applied to investigate the evolutions of the GaN surface morphology and crystal quality under the MEE process. Instead, the whole process can be accomplished inside the MBE chamber by monitoring the change of RHEED pattern. We have shown that, with the combination of a Ga-lean buffer and the MEE method, a high quality GaN material with smooth surface morphology can be grown on a foreign substrate for device applications using MBE with a single growth-run manner.

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