Metalorganic Vapor Phase Epitaxy of III-Nitride Light-Emitting Diodes on Nanopatterned AGOG Sapphire Substrate by Abbreviated Growth Mode

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***Abstract*—Metalorganic vapor phase epitaxial (MOVPE) growth of GaN on nanopatterned AGOG sapphire substrates was performed, and characteristics of the light-emitting diode (LED) devices grown on patterned sapphire and planar substrates were compared. The nanopatterned sapphire substrates were fabricated by a novel process (AGOG) whereby aluminum nanomesas were epitaxially converted into crystalline Al_2O_3 via a two-stage annealing process. The GaN template grown on the nanopatterned sapphire substrate was done via an abbreviated growth mode, where a 15-nm thick, low-temperature GaN buffer layer was used, without the use of an etch-back and recovery process during the epitaxy. InGaN quantum wells (QWs) LEDs were grown on the GaN template on the nanopatterned sapphire, employing the abbreviated growth mode. The optimized InGaN QW LEDs grown on the patterned AGOG sapphire substrate exhibited a 24% improvement in output power as compared to LEDs on GaN templates grown using the conventional method. The increase in output power of the LEDs is attributed to improved internal quantum efficiency of the LEDs.**

***Index Terms*—Dislocation density, InGaN quantum wells, light-emitting diodes, MOVPE growth, nanoheteroepitaxy, sapphire.**

I. Introduction

SAPPHIRE is most commonly used as the substrate for nitride light-emitting diodes (LEDs) in solid state lighting due to its physical robustness and high temperature stability. GaN native substrates are still at present very expensive and not viable for large-scale device production, in particular for addressing low-cost, solid-state lighting applications. Direct growth of high-temperature GaN on sapphire often results in poor film quality and severe epitaxy film cracking. This is due to the large lattice mismatch of 16% between wurtzite GaN and c-plane sapphire. To overcome this, conventional GaN growth on sapphire by metalorganic vapor phase epitaxy (MOVPE) employs low-temperature GaN buffer [1] or AlN buffer [2] layers, prior to the growth of high-temperature GaN layer. The essence of the low-temperature, buffer-layer technique is to reduce the interfacial free energy between the epitaxial layer and the highly mismatched substrate by using a more compliant softer material [3]. To reduce the dislocation density of MOVPE GaN through the *in situ* method, hydrogen (H_2) etch back of the low-temperature GaN buffer layer and intentional delay of the nucleation island coalescence (recovery) is often adopted [4]. The etch-back and recovery technique typically adds 30 to 45 min to the MOVPE GaN growth time, thus adding significant cost to the epitaxy of nitride-based LEDs.

The threading dislocation density of the GaN template grown by the conventional approach is still high, in the mid 10^8–10^{10} cm^{-2} range [5]. High density of threading dislocations has been linked to failure of lasers [6] and breakdown of p–n junctions [7]. Hence, for high performance and reliable nitride-based LEDs and laser diodes, threading dislocation density has to be reduced. Several approaches have been implemented to reduce the threading dislocation density in GaN epitaxial layers, such as lateral epitaxial overgrowth (LEO) [8], pendeo epitaxy [9], and cantilever epitaxy [10]. These approaches have demonstrated reduced dislocation densities in the GaN layers in the range of 10^6–10^7 cm^{-2}, but the low-defect regions are only limited to 5–10 μm wide stripe ridges. Recently, Hersee *et al.* proposed a technique based on nanoheteroepitaxy (NHE) of GaN on nanopatterned silicon substrates [11], which leads to reduction in the dislocation density of the GaN film through the introduction of compliant substrate surface structures. Other research groups have also grown III-Nitride LEDs via conventional GaN growth on patterned sapphire substrate for the goal to increase the light extraction efficiency of LEDs [12]–[15]. However, the approach to enhance the light extraction efficiency requires relatively deep etch patterns in the range of 1–5 μm, as well as typical pattern size and center-to-center spacing of 3 and 4 μm, respectively [12]–[15].

In this paper, we present NHE of GaN template grown on nanopatterned AGOG c-plane sapphire substrate employing a novel "abbreviated growth mode" by MOVPE. We also compare the device characteristics of nitride LED devices grown on our current template with those grown on conventional GaN templates.

The advantages of using nanopatterned sapphire substrates are twofold: 1) increased internal quantum efficiency of nitride LEDs with improved reliability from the reduction in dislocation density and 2) ability to utilize the novel abbreviated growth mode for the epitaxy of GaN that avoids the need for

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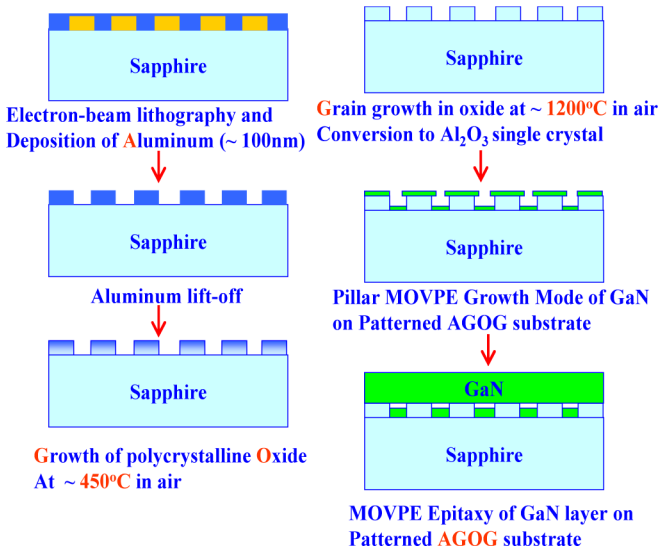

Fig. 1. Fabrication process of nanopatterned AGOG sapphire and MOVPE of GaN on nanopatterned AGOG sapphire substrate.

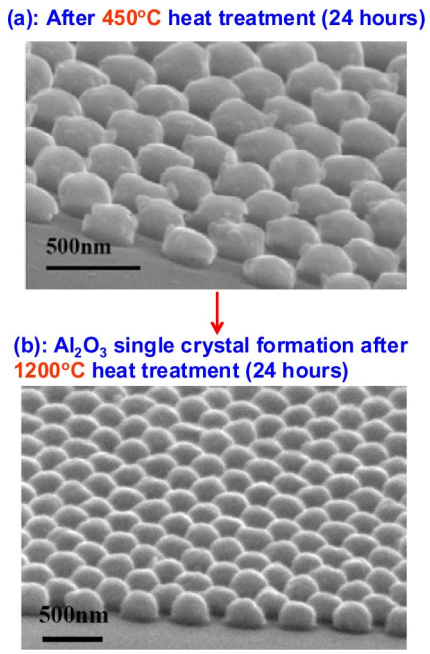

Fig. 2. (a) SEM of aluminum nanostructure array after oxidation at 450 °C and (b) epitaxial conversion to single crystal Al_2O_3 after 1200 °C anneal.

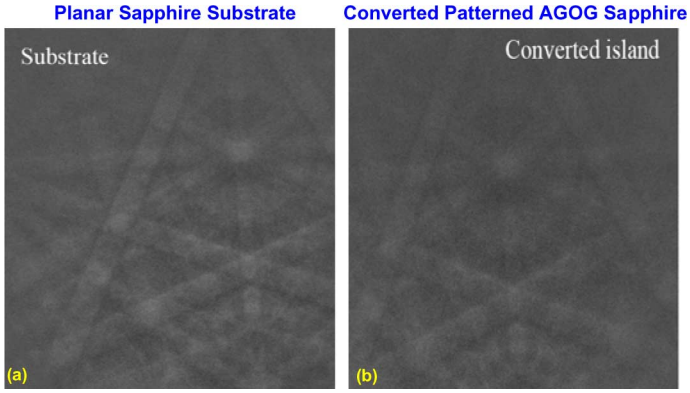

Fig. 3. Electron backscatter diffraction image of planar sapphire substrate and nanopatterned region after the AGOG process.

an etch-back and recovery technique. This, in turn, leads to a significant reduction in cost and time of the epitaxy. From our studies, we found that use of the abbreviated growth mode was important for optimized growth of GaN templates on nanopatterned sapphire, and improvements in LED output power were observed for the InGaN QW LEDs grown on nanopatterned sapphire substrates.

II. Nanopatterning of Sapphire Substrate Through Novel AGOG Process

In our approach, instead of patterning the sapphire substrate via an etching technique [12]–[15], we employed a novel AGOG process that enables creation of single crystal Al_2O_3 islands on the sapphire substrates. The "AGOG" is an acronym for the process of conversion Al into single crystal sapphire, by employing aluminum deposition, growth of oxide, and grain growth [16], [17]. It is the unique process of creating these nanostructures via oxidation of deposited aluminum (Al) islands to form Al_2O_3 and subsequently annealing to convert the islands to single crystal [16], [17]. In our current approach, by converting nanopatterned Al-metals deposited on sapphire substrate via AGOG process, nanopatterned AGOG sapphire substrate can be realized. The details of the nanopatterning of the sapphire via the AGOG process and MOVPE epitaxy of the GaN on the patterned AGOG substrate are presented in Fig. 1, with the following steps: 1) electron-beam lithography of metallic nanostructures on sapphire; 2) deposition of 100 nm **A**l-layer for lift off; 3) annealing treatment to **g**row a polycrystalline **o**xide; and 4) high-temperature **g**rain growth.

In our experiments, the Al-metal lift off mask patterned by electron-beam lithography is composed of an array of hexagons approximately 200 nm wide with center-to-center spacing of 400 nm. In our proof-of-concept experiment, the size of the patterned AGOG region was limited to 1 mm $\times$1 mm. A layer of 100 nm Al was then deposited, followed by the lift off process. The patterned Al metal on *c*-plane sapphire underwent two heat treatments: 1) 450 °C oxidation anneal in air for 24 h [scanning electron microscopy (SEM) image shown in Fig. 2(a)], followed by 2) 1200 °C in air for 24 h to induce grain growth of the underlying sapphire single crystal to consume the oxide layer [shown in Fig. 2(b)].

Electron backscatter diffraction (EBSD) was conducted on the planar sapphire [Fig. 3(a)] and nanopatterned AGOG sapphire [Fig. 3(b)] islands. Indexing of the patterns confirmed that the patterned AGOG nanostructures consisted of sapphire with the same orientation as the (0001) substrate [see Fig. 3(a) and (b)]. Further details on this finding have been reported elsewhere in [16]. The EBSD pattern of the AGOG nanopatterned region is not visible for cases in which the islands are not converted into single crystal. Hence, the EBSD pattern of the converted island in Fig. 3 was from the AGOG nanostructures and not the underlying sapphire substrate. The GaN layer was then grown on nanopatterned AGOG sapphire, using nanopillar growth mode. Ideally, the nanopillar growth mode enables the strain to distribute in three dimensions, which leads to a reduction in strain

energy and stress-induced defect formation. The dislocations in the GaN grown on the patterned sapphire islands are terminated when the GaN film coalesces.

III. MOVPE of GaN Templates on Nanopatterned Sapphire Employing Abbreviated Growth Mode

In the conventional GaN template growth, a 30-nm thick, low-temperature (535 °C) GaN buffer layer is grown on c-plane sapphire, followed by a H_2 etch-back process, and a delayed recovery stage during high-temperature (1080 °C) GaN growth. During the H_2 etch-back process, the low-temperature buffer layer is broken down into micron-sized crystallites from which the high-temperature GaN can nucleate and grow. This step is critical for the successful growth of the high-temperature GaN. The AGOG nanopatterns on the sapphire substrate have much smaller dimensions at 200 nm. Though the nanoisland size is not small enough to completely eliminate strain energy, it enables the strain to distribute in three dimensions, which leads to reduction in strain energy and stress-induced defect formation. Nonetheless, there is significant surface roughness introduced by the nanopatterned surface as compared to a standard planar sapphire substrate.

To investigate whether high-temperature GaN is able to nucleate on nanopatterned sapphire without the presence of a buffer layer, an experiment was conducted in which high-temperature GaN growth was carried out directly on a sapphire substrate with small AGOG nanopatterned regions surrounded by conventional planar surfaces. Fig. 4(a) and (b) show SEM images of 0.25-μm thick, high-temperature GaN grown directly on this substrate. From Fig. 4(a), it was observed that the high-temperature GaN grows readily on the nanopatterned region of the sapphire. In contrast, the high-temperature GaN *did not nucleate* on the adjacent planar region. The scalloped appearance of the bottom edge of the nanopatterned sapphire in Fig. 4(a) is an artifact due to imperfect metal lift-off during processing. A higher magnification SEM micrograph of the high-temperature GaN grown on a nanopatterned region of the sapphire substrate is shown in Fig. 5. Together, these images confirm that the use of nanopatterned AGOG sapphire leads to improvement in the nucleation process of the GaN without the need for a low-temperature GaN buffer layer and etch-back and recovery process. However, closer examination of the high-temperature GaN grown in this way reveals that the surface has undesirable roughness on the c-plane surface.

As a compromise between surface quality and speed of growth, surface morphology studies were conducted using GaN grown on nanopatterned sapphire by an "abbreviated growth mode" technique. In this technique, a 15-nm thick, low-temperature GaN buffer is grown, followed by the growth of high-temperature GaN *without* the intermediate etch-back and recovery process. Fig. 6 shows SEM micrographs of the nanopatterned and planar sapphire after the growth of 15-nm GaN buffer layer and 0.1 μm of high-temperature GaN. The surface morphology of the two regions was very different. With a thin buffer layer—and with etch-back and recovery processes bypassed—GaN was preferentially grown on the nanopatterned

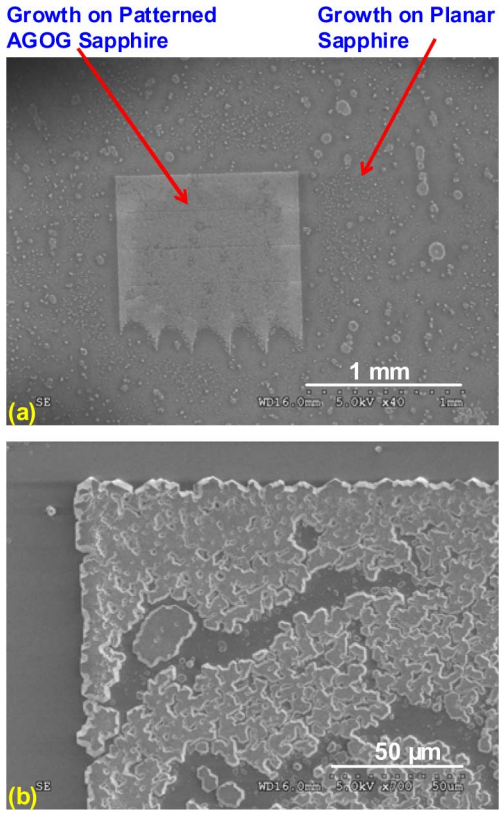

Fig. 4. SEM of (a) high-temperature GaN grown directly on the nanopatterned region as well as the surrounding planar region, (b) top left corner of nanopatterned region.

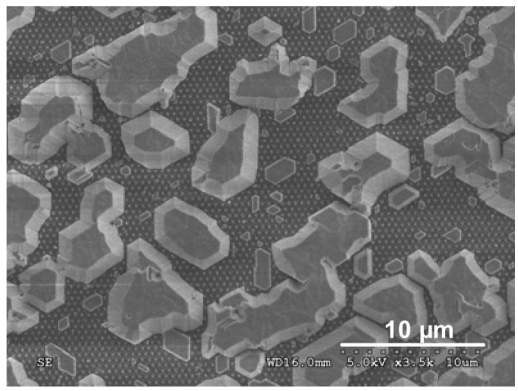

Fig. 5. SEM of 0.25-μm-thick GaN grown directly on nanopatterned sapphire without a buffer layer.

sapphire region in a similar fashion to the case with no buffer layer. Despite the relatively thin GaN layer of 0.1 μm, coalescence of GaN on the patterned region was significantly advanced over that of the 0.25-μm-thick GaN grown directly on nanopatterned sapphire, resulting in relatively planar material early in the growth process.

To further investigate the surface morphology evolution during GaN growth, studies were also conducted on 0.25-μm thick, high-temperature GaN grown on a similar nanopatterned sapphire substrate with a 15-nm GaN buffer layer. Fig. 7 shows a SEM micrograph of the GaN grown on a nanopatterned region of

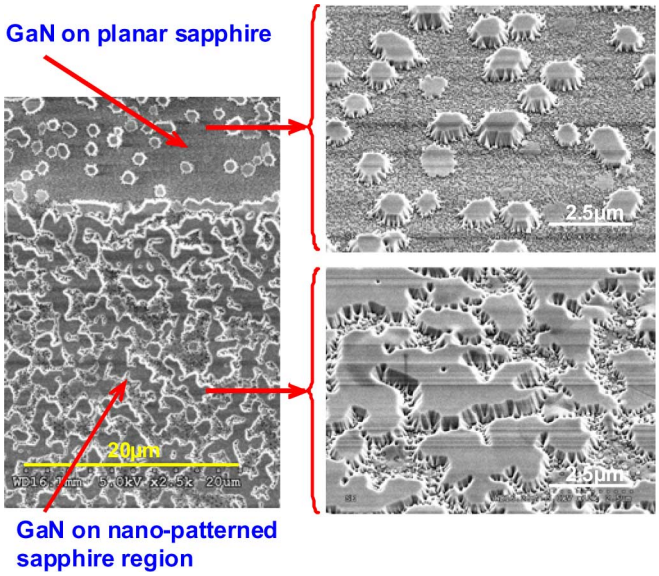

Fig. 6. SEM of 0.1-μm-thick GaN grown on nanopatterned sapphire and planar sapphire with a 15-nm buffer layer.

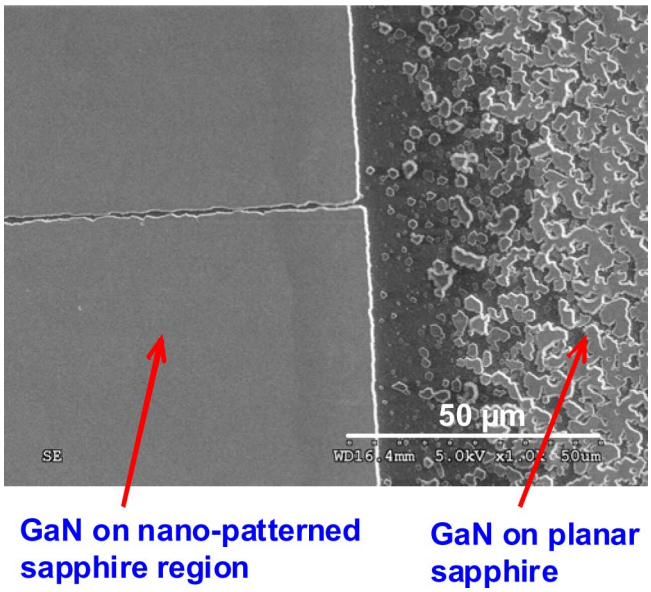

Fig. 7. SEM of 0.25-μm-thick GaN grown on nanopatterned sapphire and planar sapphire with a 15-nm buffer layer.

the substrate, as well as that grown on the adjacent planar region. From Fig. 7, we observe that at 0.25 μm the high-temperature GaN has completely coalesced forming a smooth film on the nanopatterned sapphire, but not on the planar region. The use of the abbreviated growth mode on nanopatterned sapphire is a significant advantage over the conventional approach since it reduces the epitaxy time, leading to cost reduction. Note that our abbreviated growth mode is different from the previously reported [18] GaN growth mode on etched patterned sapphire, which still require the use of conventional growth method utilizing the etch-back and recovery processes [18].

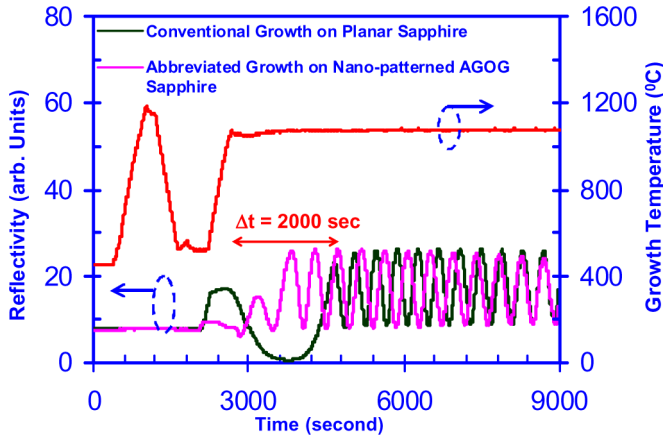

Fig. 8. Comparison of reflectivity during the growth of n-doped GaN template using the (a) conventional and (b) thinner buffer layer method without etch-back and recovery process. The temperature profile is shown for growth employing abbreviated mode.

IV. Growth of InGaN QW LEDs

As a first step toward fabrication of InGaN quantum well (QW) LEDs, the successful high-temperature GaN growth conducted on a thin buffer layer without the usual etch-back and recovery processes was extended to the growth of n-GaN templates on patterned and planar sapphire substrates. Conventional growth on planar sapphire was performed for comparison. During the growth of the high-temperature GaN, the molar flow rate of trimethylgallium (TMGa) was 5.407 μmol/min, and NH_3 was used as group V source with a flow rate of 2800 sccm, corresponding to a V/III ratio of about 3700. Fig. 8 shows a plot of reflectivity and growth temperature during epitaxial growth of the n-doped GaN templates. For the conventional growth, the etch-back and recovery stages are shown in the reflectivity curve. These growth steps were bypassed during the abbreviated growth for a savings of approximately 30 min. The GaN templates grown using the conventional and abbreviated methods were both 2.8 μm thick.

The InGaN QW LED devices were grown on the three comparison GaN templates as follows: abbreviated growth mode on nanopatterned AGOG sapphire (sample 1), conventional growth mode on planar sapphire (sample 2), and abbreviated growth mode on planar sapphire (sample 3). The InGaN QW LED structures were grown on the comparison GaN templates at the same time, and the active region consisted of four-period 2.5-nm $In_{0.15}Ga_{0.85}N$ QW and 12-nm GaN barriers ($T_g = 740\,^{\circ}$C). The growth of the active regions and barriers employed N_2 as the carrier gas with a flow rate of 2500 sccm. The molar flow rates of triethylgallium (TEGa) and trimethylindium (TMIn) were 1.104 and 0.613 μmol/min, respectively. The In-content of the InGaN QW studied here was found to be 15%, as calibrated via X-ray diffraction.

V. LED Device Characteristics

Fig. 9 shows the LED light output power versus injection current of three comparison LEDs. The LED devices were measured under continuous wave (CW) conditions at room

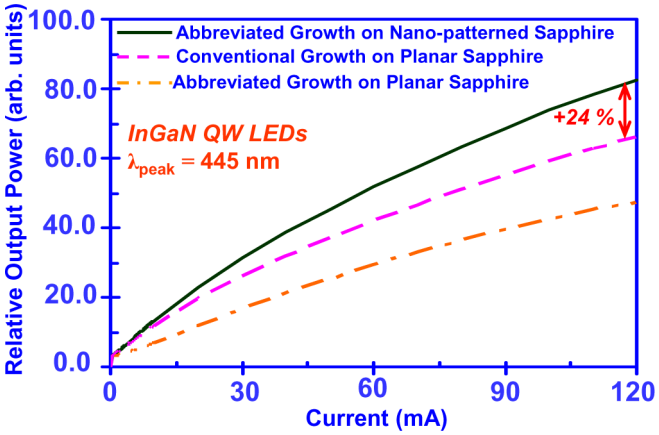

Fig. 9. Comparison of room temperature CW output power—injection current of $In_{0.15}Ga_{0.85}N$ QW LEDs grown on three comparison templates (samples 1, 2, and 3).

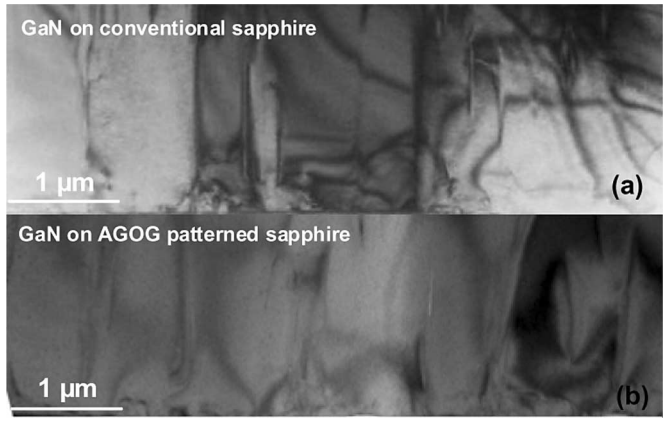

Fig. 10. Cross-sectional transmission electron microscopy of (a) GaN grown on planar sapphire using conventional growth and (b) GaN grown on nanopatterned sapphire with abbreviated growth mode.

temperature. The on-wafer output power of the LEDs with an area of 1.25×10^{-3} cm^2 was measured in a dark room. Not surprisingly, the InGaN QWs LED grown using abbreviated growth mode on planar sapphire (sample 3) had an output power 28% lower than the LED grown using the conventional method on planar sapphire (sample 2). However, for the InGaN QWs LED grown using abbreviated mode on patterned AGOG sapphire (sample 1), the output power and internal quantum efficiency were enhanced by 24% in comparison to the conventional LED (sample 2).

Fig. 10(a) and (b) show cross-sectional transmission electron microscopy (TEM) images for LED device structures on GaN template grown on planar sapphire by using conventional method (sample 2) and LED device structures on GaN template grown on nanopatterned AGOG sapphire by using abbreviated growth mode (sample 1), respectively. These preliminary measurements indicate that the threading dislocation density of the GaN grown on patterned AGOG sapphire is approximately an order magnitude lower than that of GaN grown on planar sapphire. The reduction in dislocation density can be observed in Fig. 10(b); however, it is difficult to provide accurate quantification of the threading dislocation density from cross-sectional TEM. Future works on plan-view [0001] TEM measurements [19] on both sample 1 and sample 2 are required to provide accurate quantification of the dislocation density.

This proof-of-concept experiment demonstrates that the abbreviated growth mode on patterned sapphire formed via the AGOG process shows promise for achieving improved radiative efficiency of InGaN QW LEDs. At this stage, it must be acknowledged that electron beam patterning of the substrate is impractical as a production tool. The advantage offered by the use of patterned AGOG sapphire substrates can be exploited only when the patterning can be accomplished by large-scale and low-cost lithography processes (e.g., holography lithography). Once this is achieved, the thermal processing of the substrates can be performed as a batch process at relatively low additional cost.

VI. Conclusion

In summary, proof-of-concept MOVPE growth experiments were conducted with GaN template on nanopatterned AGOG sapphire substrates. The use of patterned AGOG sapphire enabled introduction of an "abbreviated growth mode" that significantly reduces the epitaxy time for a typical GaN template. This growth mode is dependent on the patterned sapphire surface for success. Although a thin-GaN buffer layer is still needed to facilitate coalescence of the high-temperature GaN, the conventional etch-back and recovery steps are no longer required.

Comparison studies were carried out on various InGaN QW LED devices grown on three comparison GaN templates: abbreviated growth mode on nanopatterned AGOG sapphire, conventional mode on planar sapphire, and abbreviated growth mode on planar sapphire. The output power of the LEDs grown with the abbreviated mode on the nanopatterned substrate demonstrated improved optical output power of 24% over the LEDs grown using the conventional method. This improvement can be attributed to the increase in radiative efficiency of the material. Further structural characterizations are still required to provide a better understanding of the mechanism underlying this improvement. In addition to the improved radiative efficiency in InGaN/GaN LED devices, the abbreviated growth mode of GaN template on patterned AGOG sapphire—accompanied by large-scale and low-cost substrate patterning—could potentially lead to a significant reduction in the growth time and cost for LED epitaxy.

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Dr. Tansu was a panel member for the U.S. National Science Foundation, the U.S. Department of Defense, and other agencies in U.S. and abroad. He has also given numerous lectures, seminars, and invited talks (total $>$35) in universities, research institutions, and conferences in USA, Canada, Europe, and Asia. He is the Primary Guest Editor of the IEEE JOURNAL OF SELECTED TOPICS IN QUANTUM ELECTRONICS Special Issue on Solid State Lighting in 2008–2009, and he also serves as an Associate Editor for IEEE PHOTONICS JOURNAL (2009-present) and as Assistant/Associate Editor for Nanoscale Research Letters (2007-present). Dr. Tansu was also an invited General Participant at the 2008 National Academy of Engineering (NAE)'s U.S. Frontiers of Engineering (FOE) Symposium, and he also serves as the Organizing Committee for the 2009 NAE's U.S. Frontiers of Engineering Symposium. He was a recipient of the Bohn Scholarship, the WARF Graduate University Fellowship, the Vilas Graduate University Fellowship, and the Graduate Dissertator Travel Funding Award, the 2003 Harold A. Peterson ECE Best Research Award (1st Prize) at the University of Wisconsin-Madison, and the 2008 Libsch Early Career Research Award at Lehigh University.