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Research Article

Void Shapes Controlled by Using Interruption-Free Epitaxial Lateral Overgrowth of GaN Films on Patterned SiO_2 AlN/Sapphire Template

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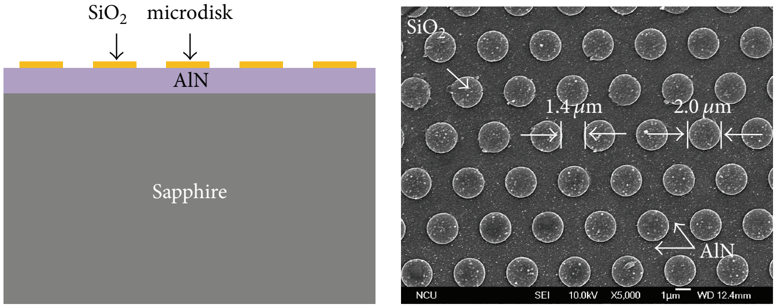
GaN epitaxial layers with embedded air voids grown on patterned SiO_2 AlN/sapphire templates were proposed. Using interruption-free epitaxial lateral overgrowth technology, we realized uninterrupted growth and controlled the shape of embedded air voids. These layers showed improved crystal quality using X-ray diffraction and measurement of etching pits density. Compared with conventional undoped-GaN film, the full width at half-maximum of the GaN (0 0 2) and (1 0 2) peaks decreased from 485 arcsec to 376 arcsec and from 600 arcsec to 322 arcsec, respectively. Transmission electron microscopy results showed that the coalesced GaN growth led to bending threading dislocation. We also proposed a growth model based on results of scanning electron microscopy.

1. Introduction

III-V compound semiconductors of AlN, GaN, and InN are suitable materials for light-emitting diodes (LEDs) because of their wurtzite crystal structures and direct band gap characteristics [1]. LEDs used for backlighting sources of liquid crystal displays demand solid-state lighting technology [2]. However, large lattice mismatches between substrates and epitaxial layers lead to the formation of threading dislocations (TDs), which decrease the lifetime of diodes and deteriorate the quality of crystals [3]. Epitaxial films with high crystal qualities are necessary for next-generation applications. Hence, reducing the TD density of epitaxial films is a primary challenge.

Recent studies have proposed several useful growth techniques to improve the crystal quality, such as epitaxial lateral overgrowth (ELOG) [4–6], pendeo-epitaxy (PE) [7], maskless PE [8], cantilever epitaxy [9], facet-controlled epitaxial lateral overgrowth (FACELO) [10, 11], SiN_x/GaN buffer layer [12], abbreviated growth mode [13–15], and freestanding GaN substrates [16–18]. Patterned sapphire substrate [19] and embedded air voids method [20, 21] have been developed to further enhance the light extraction efficiency (LEE) of light-emitting diodes (LEDs). However, embedded air voids method has been widely used. Several kinds of lateral overgrowth techniques have been reported to create air voids, such as using nanocolumns [22], nanorod [23], PE, or ELOG technique [24]. Ali et al. also showed that void shapes can be controlled using different hexagonally patterned maskless GaN templates. The TDs near the voids were bent differently with the various hexagonally patterned maskless GaN templates [25, 26]. Martinez-Criado et al. used the ELOG technique to embed air voids into GaN substrate and recommended the stress relaxation and crack suppression [16]. Dai et al. reported the higher light escaping probability with the chemical etched embedded rhombus-like air voids in light-emitting structures [27]. In addition, embedded air voids play a key role in freestanding GaN substrate fabrication. Lin et al. employed the GaN films grown on patterned sapphire substrate with large voids on the top region in the chemical lift-off process and found that the embedded voids can accelerate the wet etching process [17]. Bohyama et al. acquired spontaneously separated freestanding GaN substrate by concentrating the compressive stress at the seeds



(a) (b)

Figure 1: (a) Cross-sectional structure of patterned SiO_2 AlN/sapphire template. (b) Tilted SEM image of patterned SiO_2 AlN/sapphire template.

because of the intentional formation of voids [18]. Nevertheless, the above-mentioned techniques inevitably suffer from growth interruption and complicated procedures. In ELOG technique, a 2 μm-thick GaN epitaxial layer is deposited on the substrate, followed by photolithography with dry etching and photoresist techniques to obtain the templates [4–6]. To satisfy the next-generation application, a high efficiency with a low-cost fabrication method is required. Recently, Lai et al. reported an *ex situ* AlN buffer layer deposited by sputter, which yielded an interruption-free GaN epitaxy [28]. To further improve the light extraction efficiency, Sheu et al. implanted Ar into a sputtered AlN nucleation layer, and their results showed that the GaN-based epitaxial layer grown on implanted regions has lower growth rates than the implantation-free regions, which eventually form the embedded air voids [29–31].

In this study, we introduced the interruption-free epitaxial lateral overgrowth (IFELOG) technology, a relatively efficient technique developed to simplify template fabrication while keeping the advantages of ELOG. This technology can also control void shapes using a patterned SiO_2 AlN/sapphire template and obtain an uninterrupted growth in metal-organic chemical vapor deposition (MOCVD).

2. Experimental Procedure

GaN films used in this study were all prepared by Thomas Swan ($3 \times 2''$) MOCVD. A 25 nm thick AlN buffer layer was initially deposited on a c-plane sapphire substrate by sputter. The AlN plates on the separated sputtering guns were used as the sputtering targets for AlN buffer layer deposition. An 80 nm-thick SiO_2 film was deposited on the AlN surface by plasma-enhanced chemical vapor deposition. The sample was subsequently patterned by photolithography with photoresist and dry etching processes to form patterned SiO_2 microdisks. High-density plasma was used for SiO_2 etching. Figure 1(a) shows the specification of the patterned SiO_2 AlN/sapphire template. The pitch and the diameter of the patterned SiO_2 microdisk were 3.4 and 2 μm. Figure 1(b) shows the tilted view of the scanning electron microscope (SEM) image of the patterned SiO_2 AlN/sapphire template.

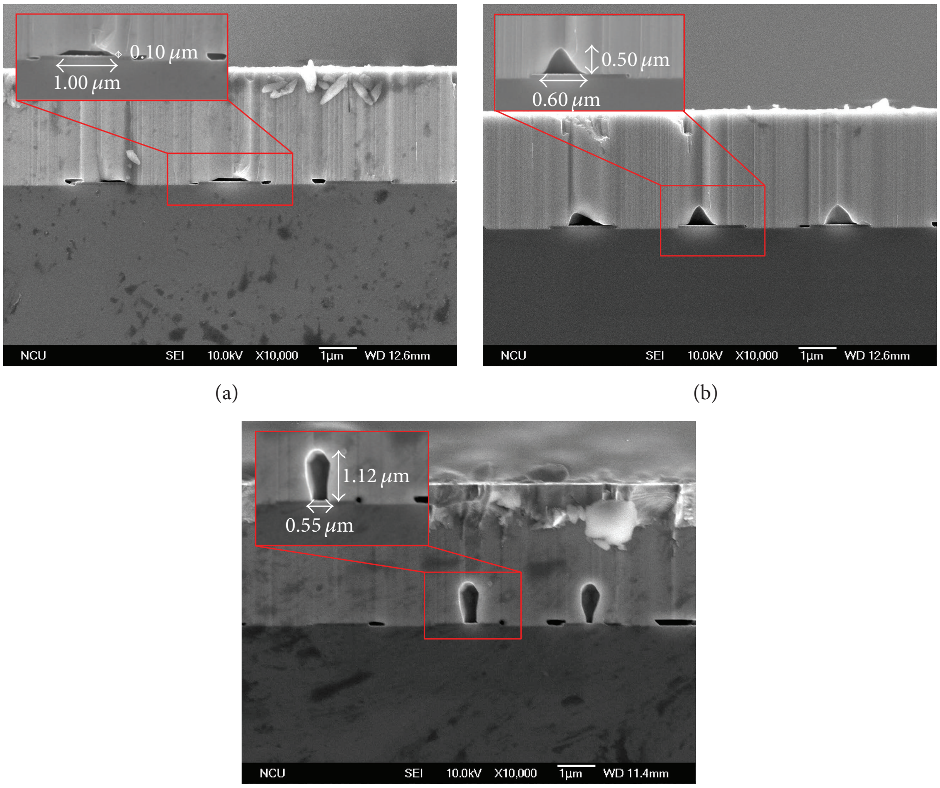
In this study we controlled void shapes by IFELOG comprising three growth steps, with each step having a specific function. The first step (Step 1) involved the initial formation of GaN seeds exposed on PVD AlN buffer layer. The second step (Step 2) involved the growth performed only against the c-plane growth, and the final step (Step 3) involved the coalescence. Trimethylgallium (TMGa) and ammonia (NH_3) were, respectively, used as gallium and nitrogen sources during growth. The GaN epitaxial layer grown on a patterned SiO_2 AlN/sapphire template with rectangular, triangular, and pillar voids was labeled as sample-R, sample-T, and sample-P, respectively.

At the onset of GaN growth, the growth temperature and chamber pressure in Step 1 were set at 1050°C and 400 torr. The growth times of sample-R, sample-T, and sample-P in the same step were 1500, 750, and 1500 s, respectively. The growth temperature and chamber pressure in Step 2 were 1050°C and 100 torr. Pulsed growth technique was applied to obtain c-plane growth in this step, which is generally used to grow GaN nanorod arrays [32]. Hence, this technique enhances the c-plane growth direction. The respective pulsed growth periods of sample-R, sample-T, and sample-P were 60, 60, and 360, respectively. The flow rates of TMGa and NH_3 were 17 sccm and 3.5 slm; the injection times of TMGa and NH_3 were 3 and 5 s. Following the pulsed growth, Step 3 was performed with growth temperature and chamber pressure set at 1080°C and 400 torr. Conventional undoped-GaN with neither IFELOG technology nor patterned SiO_2 AlN/sapphire template was prepared (i.e., sample-C) for comparative purposes.

The samples were examined by optical microscopy (OM), SEM, X-ray diffraction (XRD), atomic force microscopy (AFM), and transmission electron microscopy (TEM) to discuss the distribution of IFELOG in detail.

3. Results and Discussion

Figure 2 shows the cross-sectional SEM images of GaN epitaxial layers with differently shaped air voids utilizing IFELOG on the same template. Figure 2(a) shows the cross-sectional SEM images of sample-R with embedded rectangular air voids, each having a width and height of 1.00 and



(a) (b)

(c)

Figure 2: Cross-sectional SEM images of (a) sample-R, (b) sample-T, and (c) sample-P.

0.10 μm. Figure 2(b) shows the cross-sectional SEM images of sample-T with embedded triangular air voids, each having a width and height of 0.60 and 0.50 μm; the angle of inclination of the voids was 54°. Figure 2(c) shows the cross-sectional SEM images of sample-P with embedded pillar-shaped air voids, each having a width and height of 0.55 and 1.12 μm.

Figure 3(a) shows the growth model of the GaN epitaxial layer with various air voids. At the onset of IFELOG, we obtained different distances between GaN seeds by adjusting the growth time in Step 1 (Figures 3(a-2) and 3(b-2)). We also controlled the void heights by changing the pulsed growth periods in Step 2 (Figures 3(a-3) and 3(c-3)). Figure 4 shows the tilted and cross-sectional SEM images of the GaN epitaxial layers of sample-R after Step-2 growth in the IFELOG technology. The standing GaN seeds confirmed that this technique induced c-plane growth. We also found that the GaN seeds were not able to deposit the entire AlN area during Step-1 growth, which will be discussed in more detail in Figure 7. Considering that the samples had different growth time combinations in between steps, we obtained the various diameters and heights of GaN seeds. Ali etal. reported that the diameter of hexagonal holes between GaN affects void shape control [25]. In other words, the distances between GaN seeds define the void shape after coalescence. Figures 3(a-4), 3(b-4), and 3(c-4) show the coalesced growth in Step-3 growth. Narrowing the gap inhibits the source gas molecules from diffusing to the bottom, which eventually forms the embedded air voids that are responsible for the formation of differently shaped air voids (i.e., sample-R, sample-T, and sample-P) [33].

Table 1: Full width at half-maximum values of ω rocking curves measured by XRD.

Sample	FWHM (arcsecs)				
	(002)	(004)	(006)	(102)	(105)
Sample-C	485	479	480	600	603
Sample-R	376	362	355	322	384
Sample-T	433	454	420	408	459
Sample-P	416	420	409	360	426

The crystal quality of the GaN epitaxial layer was investigated by XRD and AFM. Heying et al. reported the pure edge TD to be insensitive to the symmetric (0 0 l) rocking curves with l nonzero and to distort only the (h k l) planes with either h or k nonzero. In other words, the decrease in the FWHM values is regarded as a reduction in TDs [34–36]. Table 1 shows the respective values of full width at half maximum (FWHM) of the GaN (0 0 2) peak, GaN (0 0 4) peak, GaN (0 0 6) peak, GaN (1 0 2) peak, and GaN (1 0 5) peak for sample-C, sample-R, sample-T, and sample-P, respectively. All of the XRD results showed an improvement in IFELOG, particularly for sample-R, which

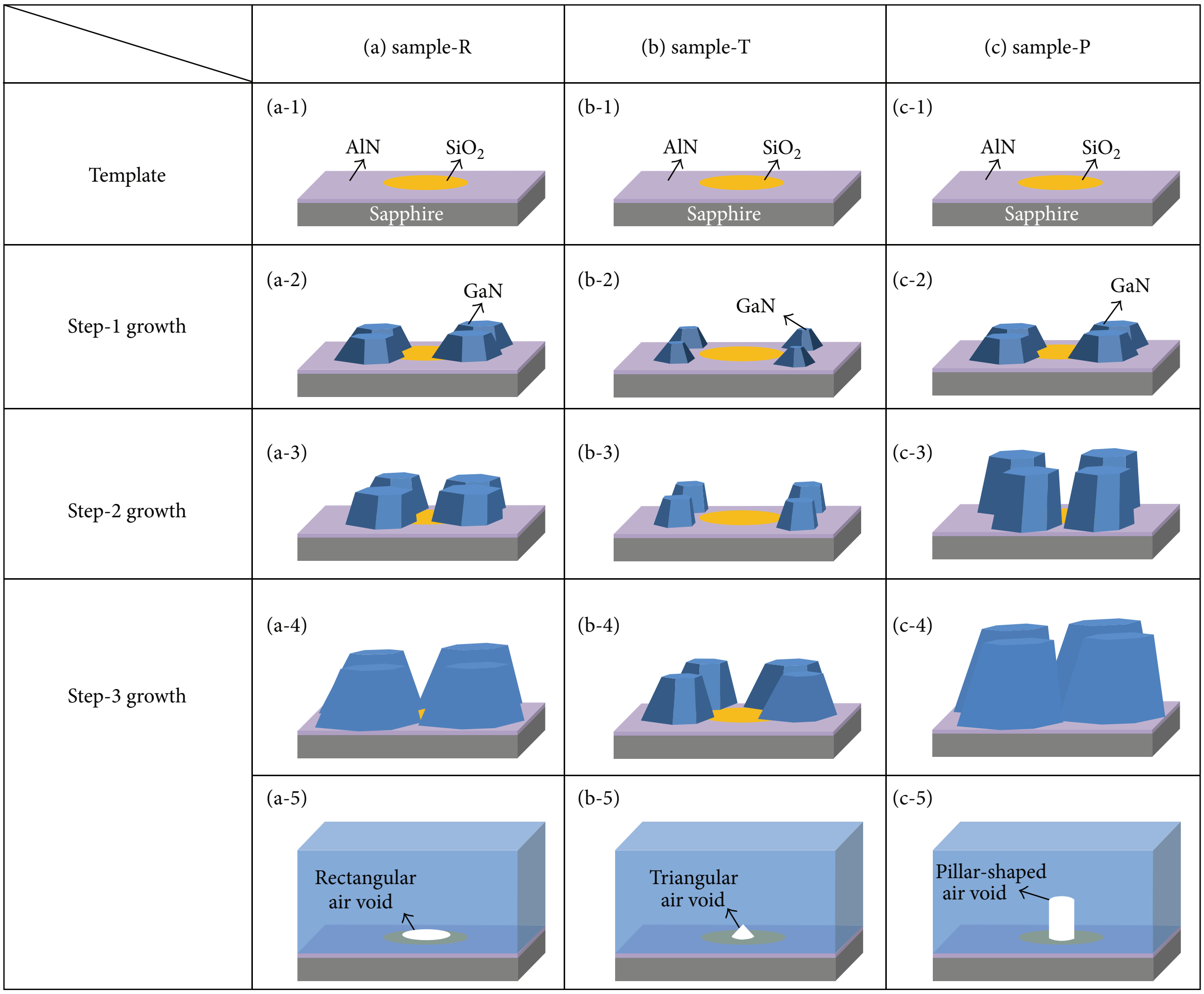
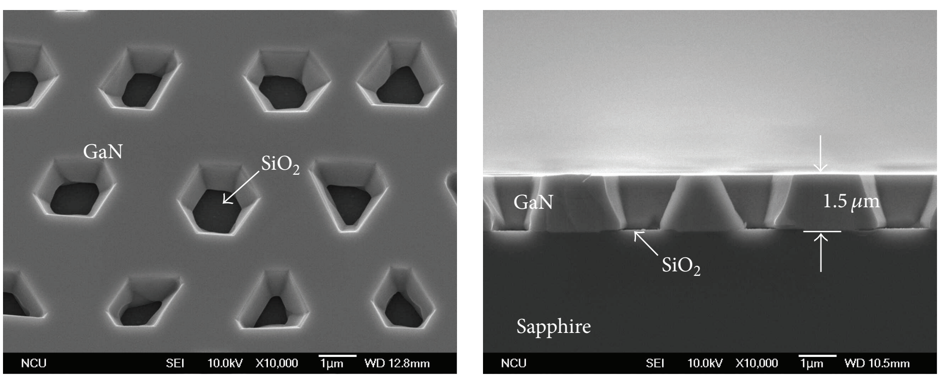


Figure 3: The proposed growth model of (a) sample-R, (b) sample-T, and (c) sample-P.



(a) (b)

Figure 4: (a) Tilted and (b) cross-sectional SEM images of the sample-R GaN epitaxial layers after Step-1 and Step-2 growth in the IFELOG technology.

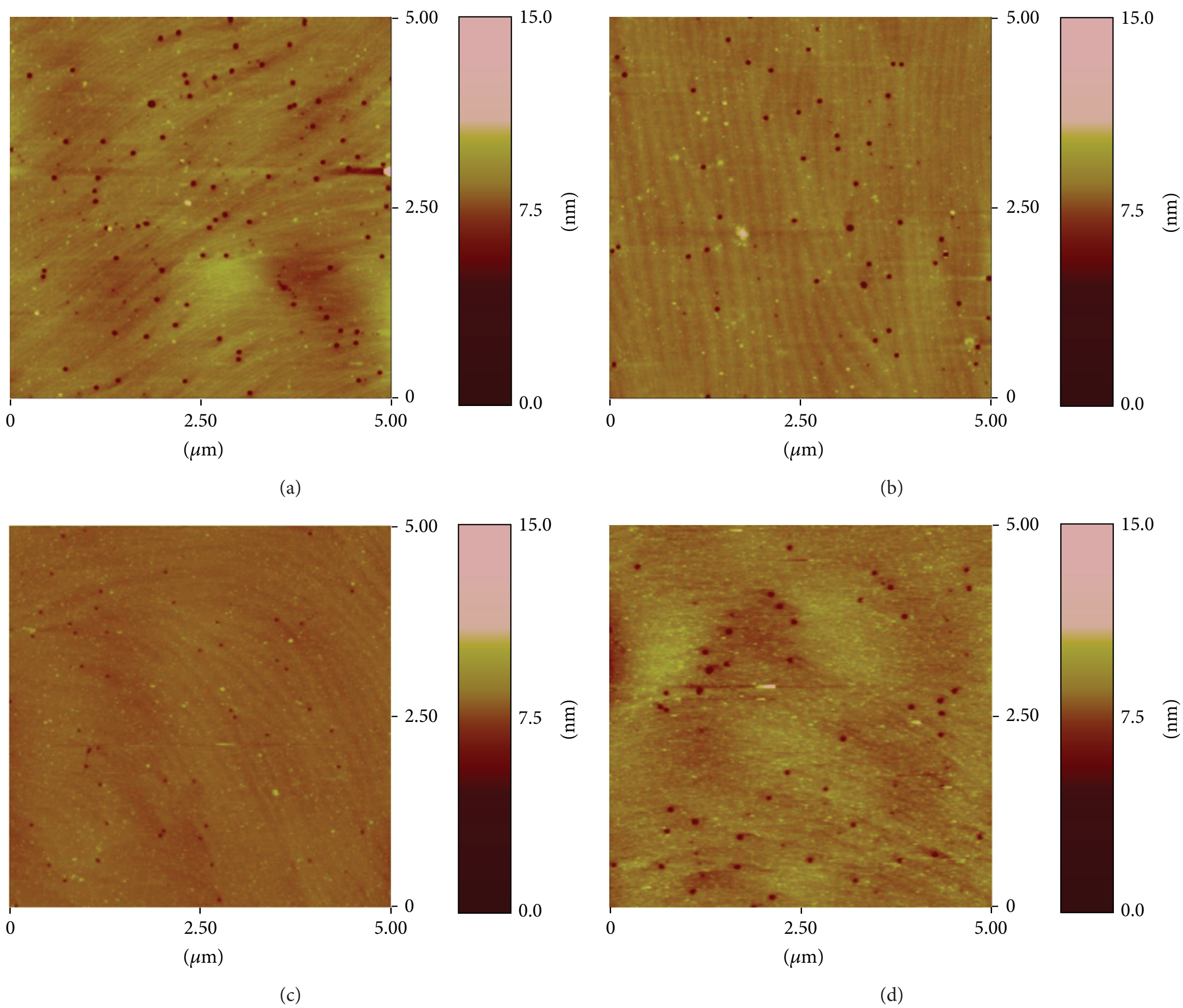


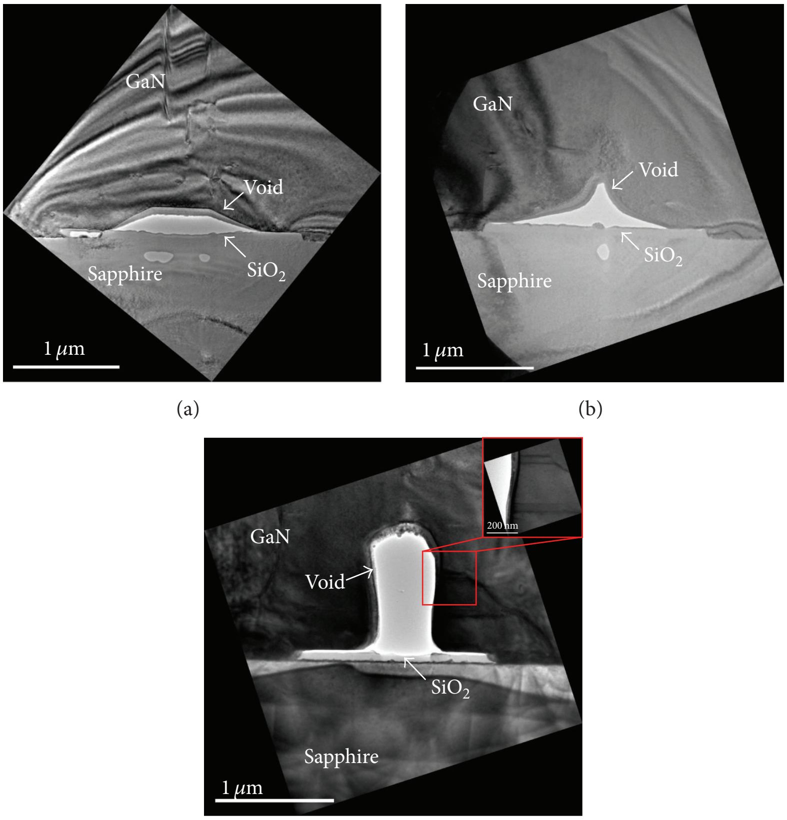
FIGURE 5: The EPD images over $5 \times 5\,\mu m^2$ scanning area of (a) sample-C, (b) sample-R, (c) sample-T, and (d) sample-P.

reduced the GaN (1 0 2) from 600 arcsec to 322 arcsec. XRD data showed that IFELOG significantly enhanced the crystal quality due to the lateral growth beside the embedded air voids. The bending dislocations were observed in the TEM images.

A wet etching experiment was conducted in H_3PO_4 solution at 250°C to determine the etching pit densities (EPDs) for GaN samples, and the samples were then examined using AFM. After wet etching, numerous hexagonal etching pits were observed on the surface. These etching pits were produced by the threading dislocations propagating to the surface of GaN, which originate from the interface between GaN and substrate. Figure 5 shows the EPD images over a $5 \times 5\,\mu m^2$ scanning area of sample-C, sample-R, sample-T, and sample-P. The EPDs of sample-C, sample-R, sample-T, and sample-P were 5.3×10^8, 2.4×10^8, 2.7×10^8, and $2.2 \times 10^8/cm^2$, respectively. These results indicated that the dislocation densities could be reduced in GaN epilayer using IFELOG technology, which also corresponded to the XRD result.

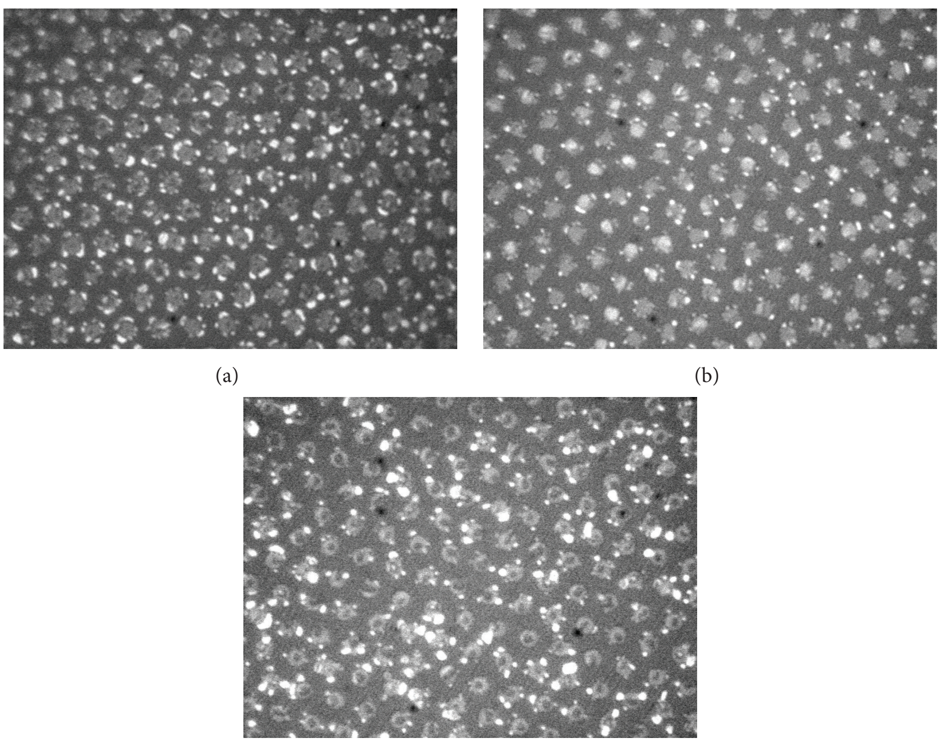
TEM was used to analyze the reduction in the dislocation density. Figure 6 shows the TEM images of the GaN epitaxial layer overgrown on the patterned SiO_2 AlN/sapphire template. The bended TDs were led by the coalesced growth beside the voids [26], which eventually developed into stacking faults (inset of Figure 6(c). Recent studies have reported that TDs can be blocked by SiO_2 and stacking faults mentioned above [37, 38]. Based on the TEM images, the decrease in FWHM of the GaN peak and EPDs was caused by the significant decrease in TDs of the GaN epitaxial layer through IFELOG.

Figures 7(a), 7(b), and 7(c) show the OM images of sample-R, sample-T, and sample-P, respectively. The OM images show that the patterned SiO_2 microdisk was surrounded by expanded voids. Among all the samples, sample-R and sample-T had the most and least numbers of expanded



(a) (b) (c)

Figure 6: The TEM images of (a) sample-R, (b) sample-T, and (c) sample-P.



(a) (b) (c)

Figure 7: The OM images of (a) sample-R, (b) sample-T, and (c) sample-P.

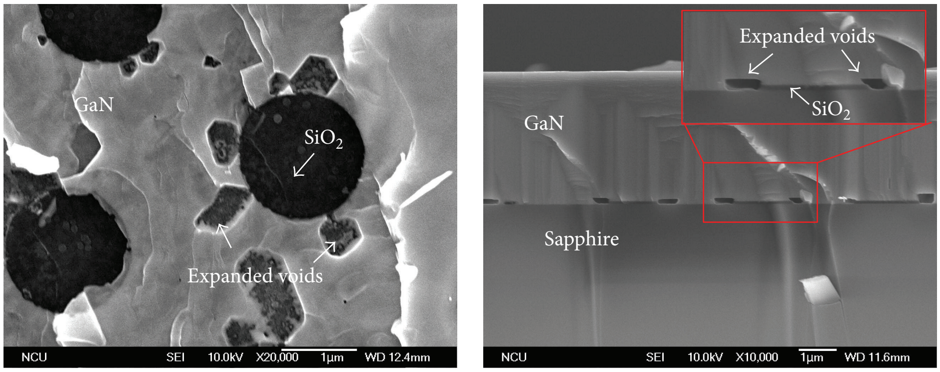


Figure 8: (a) The SEM image of sample-R lifted off by diamond cutter. (b) The cross-sectional SEM image of sample-R.

voids. Lift-off template surface and cross-section SEM measurements were performed to further clarify the cause of the expanded voids that surrounded the patterned SiO_2 microdisk. Sample-R was lifted off by a diamond cutter and it was found that the expanded voids surrounded the patterned SiO_2 microdisk (Figure 8(a)). Figure 8(b) shows the cross-sectional SEM images prepared to clearly observe the interface between SiO_2 and the GaN epitaxial layer. The formation of expanded voids was caused by the discontinuous island-type growth characteristic of the GaN seed layer in the beginning. In other words, GaN seeds were not able to deposit the entire AlN area during Step-1 growth, which resulted in residual vacancies beside the patterned SiO_2 microdisk. These vacancies were converted into expanded voids, which had the similar function with the voids generated from the nanorod template [23]. Such behavior can increase the lateral growth and lower the TDs. These expanded voids were suggested to have positive effects on the quality of the GaN epitaxial layer because of the enhancement in XRD data and EPD result of sample-R, which had the most number of expanded voids.

4. Conclusion

We successfully demonstrated an interruption-free epitaxial lateral overgrowth technology by combining sputter AlN buffer layer and pulsed growth method. By adjusting Step-1 growth time and the periods of Step-2 growth, we easily controlled the void shape by the same template. The growth model was proposed to explain the formation of differently shaped air voids based on the SEM results. AFM images show that the epitaxial layer grown by IFELOG technology has lower etching pit densities, thereby increasing the volume of defect-free regions and bending TDs. XRD data suggests that we can decrease the FWHM of the GaN (0 0 2) and (1 0 2) peaks from 485 arcsec to 376 arcsec and from 600 arcsec to 322 arcsec in sample-R. IFELOG technology not only simplified the fabrication of templates, but also greatly enhanced the quality of the GaN epitaxial layer and yielded an uninterrupted growth.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

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