

Reduction of dislocations in GaN on silicon substrate using in-situ etching

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A gallium nitride (GaN) epitaxial layer with a low density of threading dislocations was successfully grown on a silicon substrate by using in-situ gas etching. Silicon nitride (SiN_x) film was used as a mask, and ammonia was intermittently supplied in hydrogen ambient during the etching. After etching, high-density deep pits appeared on the surface of a GaN template layer and corresponded to the threading dislocations in the layer. In this novel method, before growing an additional GaN layer on the

template GaN layer, a second SiN_x layer is deposited after the etching process, and this layer prevents GaN nuclei from growing on the upper side-walls of the pits. By using this method, the density of threading dislocations of the GaN surface was reduced to $6.7 \times 10^7/\text{cm}^2$. This method is cost effective, completing all the necessary processes in one growth run without taking samples out from an MOCVD reactor.

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1 Introduction Gallium nitride (GaN) has been widely used as a material for light emitting devices (LED), especially backlights for displays and general lighting. Furthermore, it is also expected to be used as a material for high power devices and high frequency devices. Silicon substrates are attractive for GaN epitaxial growth because they allow for a large diameter, are low in cost, and have a high thermal conductivity compared with conventional sapphire substrates. In addition, integrating silicon devices and GaN devices is possible and was demonstrated [1]. While silicon substrates have such advantages for GaN growth, the lattice constant and thermal expansion coefficient between GaN and silicon are quite different. These differences lead to wafer warpage, cracking and high-density threading dislocations in the GaN epitaxial layer. In particular, it was reported that high-density threading dislocations cause the emission efficiency in LEDs to be reduced [2] and induce a decrease in carrier mobility by scattering and increase in the on-resistance and current collapse in power devices [3, 4].

To reduce the density of GaN threading dislocations on silicon substrates, a silicon nitride (SiN_x) mask layer has been reported by many research groups as a way of partially stopping the propagation of threading dislocations into the overlayer [5, 6, 7]. However, we found that, even with a SiN_x mask layer, threading dislocations in the underlying GaN template layer easily propagate into additional GaN islands because these islands tend to grow at the edges of dislocations. Another approach to reducing the density of threading dislocations is selective etching of threading dislocations followed by selective SiO₂ passivation at etched pits and epitaxial overgrowth. Methods of etching at threading dislocations are considered to be more effective at reducing the density of dislocations and have been attempted by some groups [8, 9]. However, these methods were applied to low-defect-density GaN epitaxial films on sapphire substrates, and etching involves ex-situ processes such as wet etching or SiO₂ deposition. Therefore, these methods have disadvantages from the viewpoint of manufacturing cost. In this study, a method of forming pits at the edges of threading dislocations is established that uses a

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SiN_x layer and thermal etching performed by intermittently supplying ammonia (NH_3). Furthermore, an additional SiN_x layer on the pits effectively suppresses the further threading of dislocations into the overlying GaN layer. Thus, a cost effective method is achieved by integrating all processes in one metalorganic chemical vapour deposition (MOCVD) process to reduce the density of threading dislocations.

2 Experimental Section All structures in this study were grown in an MOCVD reactor, in which trimethylaluminum, trimethylgallium and monomethylsilane (MMSi) were used as group-III precursors for the growth of aluminium nitride (AlN), GaN and the deposition of the SiN_x layers, respectively. NH_3 was used as a group-V precursor for all of the layers, and the carrier gas was hydrogen (H_2).

Before epitaxial growth, a 2-inch silicon (111) substrate was cleaned in hydrofluoric acid (HF) to remove native silicon oxide on the substrate. A 0.1- μm AlN buffer layer was grown on the surface of the substrate, followed by the growth of a 1- μm GaN template layer. The growth temperature, pressure and V/III ratio during GaN growth were 1050°C, 400 hPa and 2232, respectively. The first SiN_x layer was deposited on the template GaN layer. The duration of SiN_x deposition was 12 min, and the MMSi and NH_3 flow were 0.001 sccm and 4 SLM, respectively. After that, the GaN template layer was etched in H_2 and NH_3 ambient. Two etching methods were applied. One was the continuous supply of H_2 and NH_3 gas, and the other was the intermittent supply of NH_3 in H_2 carrier gas. For the process of intermittently supplying NH_3 gas, the NH_3 gas supply and evacuation were repeated 20 times within several seconds. Schematic drawings of the etching process are shown in Fig. 1. The total volume of the gas was held constant during the intermittent supply etching. When NH_3 gas was supplied during the process, the NH_3 and H_2 flows were 4.3 and 8.6 SLM, respectively. The total duration of the etching process was approximately 12 min. After the process, a second SiN_x layer was deposited on the surface of the GaN template layer with pits. The deposition conditions of this layer were the same as the first SiN_x layer prior to the etching process. After disposition of the second SiN_x layer, additional GaN layer was grown on the second SiN_x layer. The growth temperature of the additional GaN layer was about 1100°C. The growing process for this was divided into two steps to enhance 3D island growth at the initial stage. In the first step, the pressure and V/III ratio were 400 hPa and 558, and in the second step, these parameters were changed to 100 hPa and 2232, respectively. The procedure of the process is shown in Fig. 2.

To investigate the positions where additional GaN islands start to grow with the conventional method using SiN_x , the above additional GaN layer was grown without the etching process. An SiN_x layer was deposited on a 1- μm GaN template layer, and the deposition conditions of

the layer were the same as that of SiN_x deposition prior to the etching process. After deposition, additional GaN was grown without the etching process in the form of islands as shown in Fig. 2 (d).

The surface morphology of the samples was characterized by using a scanning electron microscope (SEM), and dislocations were observed by using a bright-field cross-sectional transmission electron microscope (TEM) under a zone axis condition. The density of the threading dislocations was measured by atomic force microscopy (AFM) by counting the pit density of the GaN surface. The crystal quality of the samples was also characterized by X-ray diffraction (XRD).

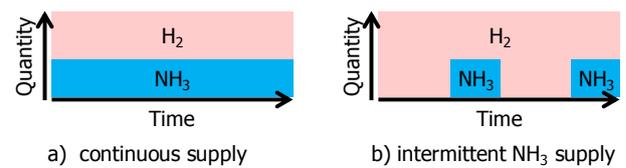


Figure 1 Schematic drawings of gas supply in etching process

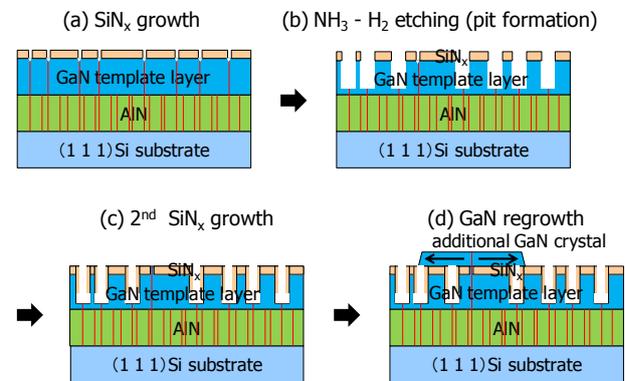


Figure 2 Schematic drawings of process. Red lines indicate dislocations in AlN and GaN.

3 Results and Discussion

3.1 Initial growth on conventional SiN_x layer

It is well known that GaN grown by MOCVD on a SiN_x layer grows in an island shape owing to the anti-surfactant effect of the SiN_x layer [5, 10-12]. However, the starting point of GaN island growth has still been incompletely understood. To investigate the initial growth on a conventional SiN_x layer, the same position of the surface morphology of the SiN_x layer and that after GaN growth on the layer was observed. Figure 3 (a) shows an SEM image of the SiN_x surface on the underlying GaN template layer, and Fig. 3 (b) shows plenty of GaN islands grown on the layer shown in Fig. 3 (a). According to refs. 10 and 11, when GaN grows on a SiN_x layer, it grows in 3D growth mode. As shown in Fig. 3 (b), the GaN grew in this mode. Therefore, it is thought that the SiN_x layer was definitely deposited on the GaN template layer. The black area in Fig. 3 (b) was coated with high density carbon because of the

high-magnitude observation in Fig. 3 (a). The density of the GaN islands in this area was lower than that in the other areas. Therefore, using this area as a marker, the red area where GaN islands grew normally was observed at the same position. Figure 4 shows magnified surface morphologies of the area inside the broke red line in Figs. 3. As shown in Fig. 4 (a), high-density dimples appeared after SiN_x deposition. The density of the dimples was $2.4 \times 10^9/\text{cm}^2$, which was almost the same as the density of threading dislocations in the underlying GaN layer. Figure 4 (b) shows the surface morphology of the initial growth on the GaN template layer covered with the SiN_x layer. Figure 5 shows a bright-field cross-sectional TEM image of Fig. 4(a). As shown in Fig. 5, it is clear that the dimples in Fig. 4 (a) corresponded to the threading dislocations in the GaN template layer. Dimples on a GaN surface annealed with SiH_4 and NH_3 were reported by R. A. Oliver et al. [13]. In ref. 13, it was also mentioned that the dimples relate to threading dislocations. The dimple size in the previous paper was much smaller than that of this study. The gas source, the concentration of the Si precursor and the deposition temperature were different from the previous paper. The difference in these conditions may have affected the difference in the shape of the dimples. The red points in Fig. 4 (b) represent the positions corresponding to the dimples in Fig. 4 (a), and the white grains are the additional GaN islands. As can be seen in Fig. 4 (b), the islands grew on the dimples, which were formed during the deposition of SiN_x . It is anticipated that the surface of the GaN template layer on which threading dislocations finally emerge will not be covered with the SiN_x layer and that additional GaN islands will start to grow from the position of the dislocations in the GaN template layer. Therefore, the additional GaN growth on the conventional SiN_x layer caused the threading dislocations to easily propagate from the GaN template layer into the additional GaN islands, as shown in Fig. 6. The reason the density of the dislocations was reduced with a conventional SiN_x layer was suggested by H. Lahrèche et al. [11]. When using a SiN_x layer, additional GaN grows in 3D mode, and GaN islands have $\{1-101\}$ facets. When some dislocations meet the facets, the dislocations are displaced in the $(1-101)$ plane. After that, the displacing dislocation meets another dislocation to form a dislocation loop.

3.2 GaN growth using in-situ etching

Figure 7 shows the surface morphology and a cross-sectional image of the GaN template layer, which was covered with SiN_x followed by etching in 100% H_2 ambient. The layer was deeply etched, and the shape of the surface became a bump, as shown in Fig. 7 (b). The positional relationship between the threading dislocations and the recesses of the GaN surface could not be clarified under this etching condition. However, the surface roughness was suppressed, and high-density pits were produced by adding a small amount of NH_3 in H_2 , as shown in Fig. 8. The small pits corresponded

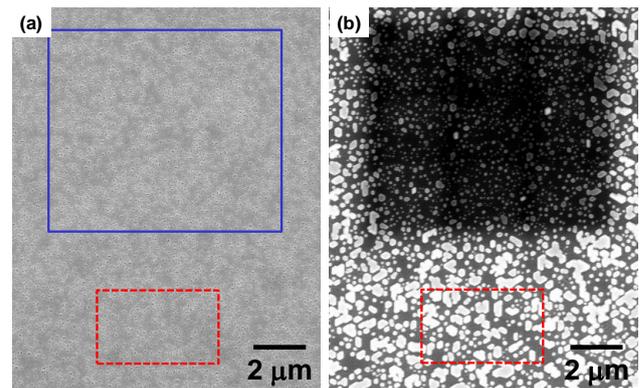


Figure 3 Surface SEM images. (a) GaN layer covered with SiN_x layer and (b) additional GaN islands on GaN layer covered with SiN_x layer. Images were observed at same position. Blue square in Fig. 3 (a) indicates area observed by high magnification after Fig. 3 (a) was observed. Area was coated with high density carbon, and GaN islands grew abnormally on this area. Using this area as marker, red area where GaN islands grew normally was observed at same position.

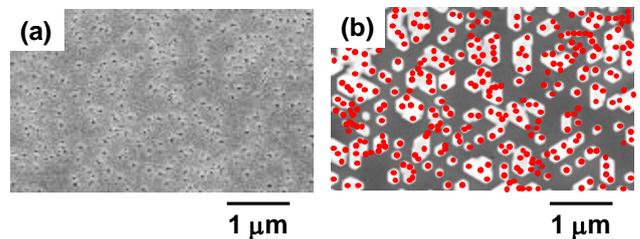


Figure 4 Surface SEM images enclosed by red square in Fig. 3. (a) GaN layer covered with SiN_x layer and (b) additional GaN islands grown on GaN layer covered with SiN_x layer. (a) and (b) correspond to Figs. 3 (a) and (b), respectively. White grains in Fig. 4 (b) are additional GaN islands. Red points represent dimples formed after SiN_x layer deposition.

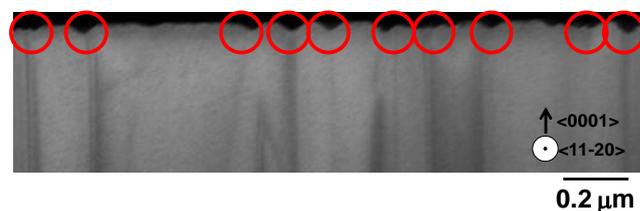


Figure 5 Bright-field cross-sectional TEM image of GaN surface after SiN_x layer deposition. Red circles emphasize dimples of surface of GaN layer. Dimples corresponded to threading dislocations in GaN template layer.

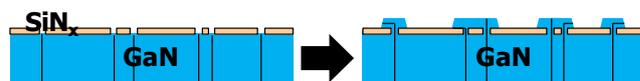


Figure 6 Schematic drawing of formation of SiN_x layer and following GaN re-growth on SiN_x layer. Black lines in GaN layer indicate dislocations.

to the dislocations in the GaN template layer, as shown by the red arrows of Fig. 8 (b). As D. D. Koleske et al. and E. V. Yakovlev et al. revealed, GaN is decomposed in H_2 ambient, but the etching rate of GaN is reduced by raising the partial pressure of NH_3 [14, 15]. The H_2 ambient caused the GaN template layer to decompose and made the surface rough, as shown in Fig. 7 (a). However, the added NH_3 suppressed decomposition, which consequently prevented deep etching at the dislocation edges. The reason the etch pits appeared at the threading dislocations is that the bonds around the dislocations were weaker than those of perfect crystal, so etching preferentially progressed around the dislocations.

Figure 9 shows bright-field cross-sectional TEM image after additional GaN growth on the GaN template layer, which was covered with the first SiN_x layer and etched in a continuous supply of 2% NH_3 in H_2 . As is shown, the etch pits were so shallow that the additional GaN layer preferentially started to grow at the bottom of the small pits. Therefore, the threading dislocations in the GaN template layer easily propagated into the additional GaN layer.

As mentioned above, etching with continuously supplied H_2 and NH_3 was not so effective at suppressing the propagation of dislocations into the overlying GaN layer from the underlying GaN template layer. On the contrary, as shown in Fig. 10, the deep pits were produced on the surface of the GaN template layer by etching with an intermittent supply of NH_3 in H_2 . The density of the pits was $1.0 \times 10^9/cm^2$, and the dislocation density of the GaN template layer was $2.0 \times 10^9/cm^2$, as measured by TEM. Thus, the density of the pits was slightly lower than the dislocation density of the GaN template layer. Considering that one pit contained several dislocations, however, the pit density was thought to be almost the same as the density of dislocations in the GaN template layer. The mechanism of deep-pit formation during intermittent supply of NH_3 can be presumed as follows. In the H_2 ambient step, the etching intensely occurred at the position of the threading dislocations because GaN decomposition was larger than that in NH_3 containing gas. In the NH_3 supplying step, the decomposition was suppressed by the existence of NH_3 . In addition, NH_3 facilitated mass transport, in which atoms migrate [16] in the GaN template layer. This mass transport flattens the surface of the GaN template layer and side walls of pits. Thus, repeating the steps for supplying H_2 and NH_3 maintains the flat surface of the GaN template layer while forming deep pits around the threading dislocations.

However, in spite of the intermittent supply of NH_3 , if there was no SiN_x layer, deep pits did not form on the surface of the GaN template layer, as shown in Fig. 11. This result implies that the SiN_x layer allowed for the selectivity of etching at the dislocation edges to be improved since the layer was preferentially deposited on the area without dislocations, as shown in Fig. 4.

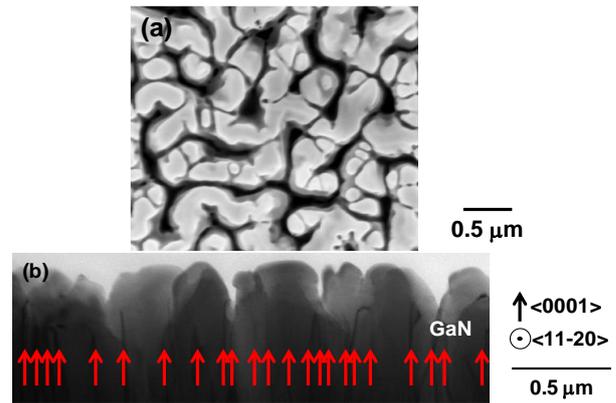


Figure 7 (a) Surface SEM image of etched template GaN surface, and (b) bright-field cross-sectional TEM image. GaN template layer was covered with first SiN_x layer and etched in 100% H_2 . Red arrows indicate positions of threading dislocations.

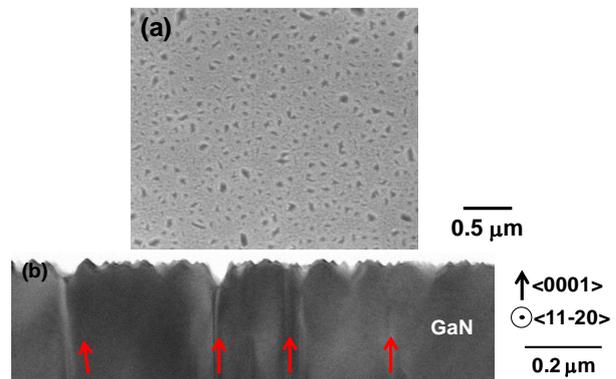


Figure 8 (a) Surface SEM image of etched surface of GaN template layer and (b) bright-field cross-sectional TEM image. GaN template layer was covered with first SiN_x layer and etched in continuous supply of 2% NH_3 in H_2 .

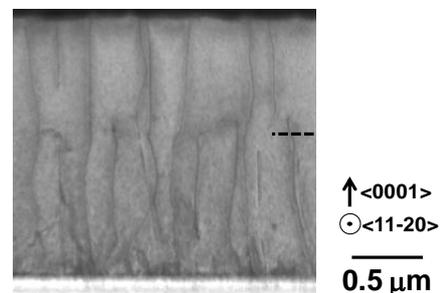


Figure 9 Bright-field cross-sectional TEM image after additional GaN growth on template GaN layer, which was covered with first SiN_x layer and etched in continuous supply of 2% NH_3 in H_2 . Dotted line indicates interface where SiN_x layer was deposited and surface was etched in NH_3 and H_2 ambient. Many dislocations penetrated into additional GaN layer.

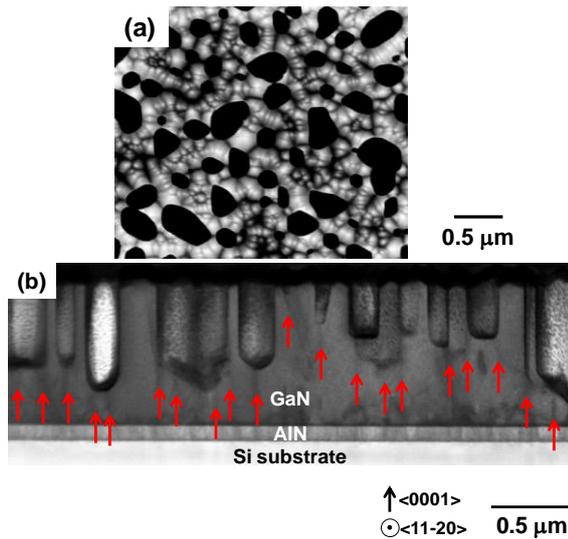


Figure 10 (a) Surface SEM image of etched surface of GaN template layer and (b) bright-field cross-sectional TEM image. The GaN template layer was covered with the first SiN_x layer and etched in an intermittent supply of 33% NH_3 in H_2 .

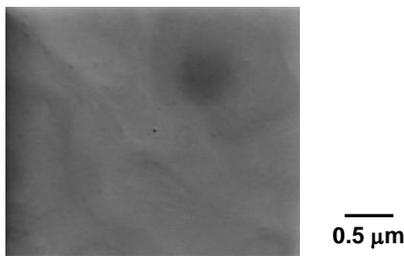


Figure 11 Surface SEM image of surface of etched GaN template layer, which was not covered with first SiN_x layer and etched in intermittent supply of 33% NH_3 in H_2 .

Figures 12 show the effect of the disposition of the second SiN_x layer on the additional GaN growth after pit formation in the GaN template layer. As shown in Fig. 12 (a), the pits were invisible when the second SiN_x layer was not used to cover the template GaN layer. In comparison, the pits could be observed on the surface of the GaN template layer, and small islands could be observed in the case of growth on the second SiN_x layer, as can be seen in Fig. 12 (b).

Figure 13 shows a cross-sectional TEM image of the initial growth of additional GaN without the second SiN_x layer. In the case of GaN growth without the second SiN_x layer, the additional GaN started to grow on the upper side-walls of the pits because there was no SiN_x layer on the side walls. This growth leads to the coalescence of GaN crystals above the pits. Therefore, GaN crystals that coalesced above the pits inevitably generated the dislocations. In fact, many dislocations can be seen above the void in Fig. 13.

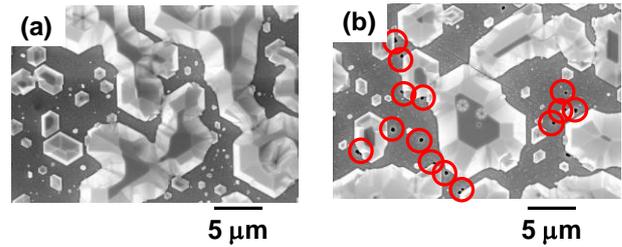


Figure 12 Surface SEM images of samples after additional GaN islands. Islands were regrown for 30 minutes on surface of GaN template layer with pits. Layer (a) without and (b) with second SiN_x layer. Red circles emphasize etch pits in template GaN layer.

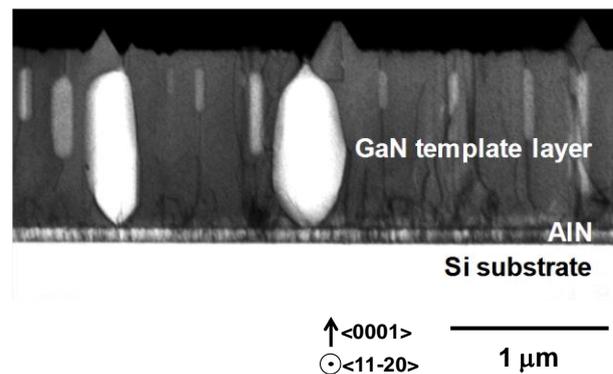


Figure 13 Bright-field cross-sectional TEM image of sample after additional GaN islands were re-grown for 2 minutes on pits without second SiN_x layer.

Figure 14 (a) shows a surface SEM image of additional GaN growth on the etched template GaN layer on which the second SiN_x layer was deposited. Figure 14 (b) shows GaN islands on the conventional SiN_x layer without etching. The growth on the pits covered with the second SiN_x layer provided a lower density of GaN islands than that on the conventional SiN_x layer without etching. As previously mentioned, in the case of growth on the SiN_x layer without etching, the GaN islands were grown on the threading dislocations because of the existence of dimples on the dislocations, as shown in Fig. 4 (b). In comparison, when the pits were formed at the threading dislocations, the GaN crystals around the pits had no dislocations. As a result, there were no dimples around the pits after the SiN_x layer was deposited on them, so the SiN_x layer could completely cover the surface of the edge of the pits. Therefore, the growth of the additional GaN islands around the pits was suppressed by the surfactant effect of the SiN_x layer. When the additional GaN islands grew on the etched GaN template layer covered with the second SiN_x layer, the additional GaN islands seemed to grow on the threading dislocations, which were not etched or were etched to form shallow pits with the etching process, as shown in Fig. 10 (b), in the GaN template layer. As a result, the growth on

the pits covered with the second SiN_x layer reduced the density of the additional GaN islands and suppressed the coalescence of the islands. Since density of dislocations can be reduced with less GaN coalescence, the growth on the pits covered with SiN_x provided less dislocation in the additional GaN layer.

Figure 15 (a) shows a bright-field cross-sectional TEM image taken after flattening the additional GaN surface, and Fig. 15 (b) shows schematic drawings of the mechanism of dislocation bending. A surface AFM image of the GaN layer of Fig. 15 (a) is shown in Fig. 16. As shown in Fig. 15 (a), the total thickness of the GaN template layer and the additional GaN layer was $3.8 \mu\text{m}$. The density of the threading dislocation in the GaN template layer was about $2 \times 10^9/\text{cm}^2$. However, many of the dislocations in the GaN template GaN terminated at the interface between the GaN template layer and the additional GaN layer. The density of the threading dislocations at the surface of the additional GaN layer was measured by counting the small pits of Fig. 16. The density was estimated to be $6.7 \times 10^7/\text{cm}^2$. The FWHMs of GaN (0002) and (10-12) measured by X-ray rocking curve were 390 and 332 arcsec, respectively. In Fig. 15 (a), it was observed that many pits were filled by the additional GaN growth. The reason for this filling was determined to be GaN crystal growth from the bottom of the pits, which were not covered by the second SiN_x layer because of the lack of reactive species in the bottom of the pits during SiN_x deposition. The pits seemed to be filled with the GaN crystals from the bottom of the pits before the GaN islands grown on the surface coalesced with the GaN crystals from the pits. The reason the dislocations terminated at the interface between the template GaN layer and the additional GaN layer despite the pits being buried is related to the depth of the pits. As shown in Fig. 15 (b), when the pits were deep, it is speculated that the GaN islands at the surface of the template GaN layer were large enough to over-grow on the GaN crystals grown in the pits. Then, the GaN islands on the surface of the additional GaN seemed to coalesce with the GaN crystals grown in the pits at the surface of the template GaN layer. When the large GaN islands on the surface and the small GaN crystals from the pit coalesced, many dislocations were bent in a vertical direction against the growth direction effectively because of the facet growth of the large GaN islands. After that, the dislocations bent horizontally and met other dislocations to form a dislocation loop and terminated. Therefore, the density of the threading dislocations at the surface can be reduced owing to the decrease in the coalescence between the additional GaN islands because of the low density of the islands on the surface of the GaN template layer.

The crack density of the flattened GaN surface was approximately $100/\text{cm}$, and this was almost the same density as the sample with the conventional SiN_x method. The crack density seemed to be more susceptible to the difference in thermal expansion between the GaN and silicon

substrate rather than the dislocation density. It is presumed that the crack density can be reduced by using an AlGaIn buffer layer [17] or AlN interlayer technique [18] with the in-situ gas etching method developed in this study.

Finally, the effects of each process and the crystal qualities of the GaN layer are summarized in Tables 1 and 2, respectively.

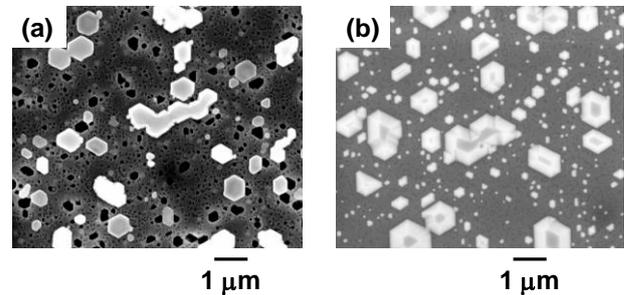


Figure 14 Surface SEM images of etched samples. Additional GaN islands were re-grown on GaN template GaN layer (a) with pits covered with second SiN_x layer and (b) covered by conventional SiN_x layer without pits.

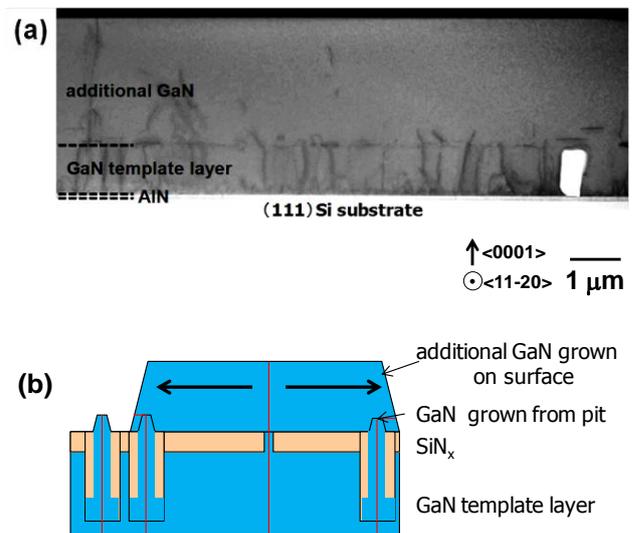


Figure 15 (a) Bright-field cross-sectional TEM image of GaN layer when in-situ etching process was used. (b) Schematic drawing of mechanism of dislocation bending. Dislocations grown from pits were bent in vertical direction against growth direction effectively because of facet growth of large GaN islands grown on template GaN.



Figure 16 Surface AFM image of GaN layer when in-situ etching process was used. Area size was $3 \times 3 \mu\text{m}^2$. Black arrows indicate threading dislocation pits. Density of threading dislocations of GaN surface was estimated to be $6.7 \times 10^7/\text{cm}^2$.

Table 1 Summary of effects of each process in this study.

Process	Effect
First SiN_x layer on GaN template layer	Improves selectivity of etching at dislocation edge.
Intermittent supply of NH_3 in H_2	Maintains flat surface of template GaN and forms deep pits around threading dislocations.
Second SiN_x layer on etched surface	Reduces density of additional GaN islands and suppresses growth on upper side-wall of pits.

Table 2 Summary of evaluated crystal quality of GaN layer.

Evaluation method	Result
Dislocation density by AFM	$6.7 \times 10^7/\text{cm}^2$
FWHMs of GaN (0002) by XRC	390 arcsec
FWHMs of GaN (10-12) by XRC	332 arcsec

4 Conclusion We achieved a low density of threading dislocations for GaN layers grown on Si substrates by using in-situ etching. Deep pits corresponding to the threading dislocations in the GaN layer were formed by intermittently supplying NH_3 during H_2 etching with a SiN_x layer. A second SiN_x layer on the pits prevented the following GaN growth at the upper side-walls of the pits and propagation of the dislocations into the upper GaN layer. The GaN growth after pit formation with a second SiN_x layer suppressed the growth of GaN islands on the GaN template layer and the generation of threading dislocations in the upper GaN compared with a method of using a single conventional SiN_x mask. The density of threading dislocations on the GaN layer on the silicon substrate was $6.7 \times 10^7/\text{cm}^2$ in this study. Our method was highly effective at reducing the density of threading dislocations and cost ef-

fective, completing all the necessary processes in an MOCVD reactor.

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