

**Research Article** 

# Single-crystal GaN growth and polarity control using an E-beam evaporated aluminum layer

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**Abstract:** We report on a method for controlling the polarity of gallium nitride (GaN) using an E-beam evaporated aluminum (Al) layer on a sapphire substrate. A high-temperature nitridation process was designed to enable the amorphous Al layer to serve as a nucleation layer for single-crystal Ga-polar GaN growth. The Al layer also acts as a mask that prevents N-polar GaN growth. As a result, Ga-polar and N-polar GaN can be grown on the Al layer and sapphire surface, respectively. This method is not only advantageous for the selective polarity control but also to simplify the fabrication process of lateral polarity structures.

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#### 1. Introduction

Group-III nitrides are polar materials that exhibit both metal- and nitrogen-polarity (N-polarity) along the c-axis owing to the absence of inversion symmetry in their structure. Although there are no inherent differences in their optical and electronic properties, metal-polar III-nitrides have attracted considerably more attention than N-polar III-nitrides because their higher quality makes them suitable for application in devices. Although it is more difficult to make high quality N-polar III-nitrides, several advantages can be obtained from their inverted structures [1–3].

Lateral polarity structures, such as periodically poled structures, have forms in which metalpolar and N-polar domains are arranged side-by-side. This structure exhibits unique properties that cannot be observed in unipolar structures because of their laterally alternating polarity. For example, the sign of the second-order nonlinear susceptibility changes with polarity. This property enables the application of lateral polarity structures as quasi-phase-matched crystals for second-harmonic generation [4–6]. In addition, electrons in the metal-polar domain can be transported laterally to the N-polar domain by the in-plane potential difference between the two polar domains. This causes a large overlap of electron and hole wave functions in lateral polarity structures, enhancing the luminescence [7–9]. In addition to these applications, various studies have been reported on electronic and optoelectronic devices that use the properties of lateral polarity structures [10–13].

There are several fabrication methods for lateral polarity structures [3]. The most common method is to use a patterned aluminum nitride (AlN) nucleation layer on a sapphire substrate [14–16]. In this method, the growth of N-polar domains is induced by the surface nitridation of the sapphire substrate [17,18]. Simultaneously, a patterned AlN nucleation layer prevents the nitridation of the sapphire surface and leads to the growth of metal-polar domains on it. Another method of fabricating lateral polarity structures is to invert the polarity on unipolar III-nitride templates or substrates. In this case, the polarity of the selected area is inverted using an AlN layer, an aluminum oxide layer, or high magnesium doping [19–21]. These fabrication methods can control the polarity of III-nitrides for selective areas. However, they require additional

processes such as etching and regrowth. Consequently, fabricating a lateral polarity structure is more complex, costly, and time-consuming than growing unipolar III-nitrides [22,23]. Therefore, it is necessary to simplify the polarity control process.

In this work, we present a method for controlling the polarity of GaN using an E-beam evaporated Al layer on a sapphire substrate. It has been reported that excess Al atoms can induce the growth of metal-polar III-nitrides [24,25]. This method takes advantage of this property. However, because the E-beam evaporated Al layer is amorphous, it cannot be used directly as a nucleation layer for the growth of single-crystal III-nitride, unlike the single-crystal Al layer or conventional AlN nucleation layer [26]. Therefore, we designed a high-temperature nitridation process to enable the E-beam evaporated Al layer to serve as a nucleation layer for the growth of single-crystal Ga-polar GaN. As a result, a simple, single growth process achieved the selective growth of Ga- and N-polar GaN on a patterned Al layer and a sapphire surface, respectively. This method is not only advantageous for the selective control of GaN polarity, but also to simplify the fabrication process of lateral polarity structures, compared to the conventional methods.

## 2. Experimental method

Polarity-controlled GaN was grown on c-plane sapphire substrates by metal–organic chemical vapor deposition (MOCVD). The sapphire substrate has a  $0.2^{\circ}$  off-cut angle toward the m-plane direction. An Al layer was deposited on the sapphire substrate by E-beam evaporation. The Al layer was exposed to air and moved to the MOCVD chamber for GaN growth. Figure 1 presents a schematic diagram of this method. This method has three steps: (1) Al layer deposition by E-beam evaporation, (2) low-temperature GaN nucleation layer growth after high-temperature nitridation, and (3) high-temperature GaN growth. Using this method, Ga-polar and N-polar GaN can be grown on the Al layer and the sapphire surface, respectively. The growth process in MOCVD is similar to general two-step N-polar GaN growth on a sapphire substrate. First, the E-beam evaporated Al layer was nitrided for 210 sec using ammonia (NH<sub>3</sub>) at 1050°C under an N<sub>2</sub> atmosphere. Subsequently, a GaN nucleation layer was deposited at 550°C with a V/III ratio of



**Fig. 1.** Schematic diagram of a method for controlling the polarity of GaN using an E-beam evaporated aluminum (Al) layer on a sapphire substrate. The growth processes of Ga- and N-polar GaN are the same. The only difference is the deposition of an Al layer before GaN growth.

3100. Trimethylgallium (TMGa) and  $NH_3$  were used as the Ga and N sources, respectively, with hydrogen (H<sub>2</sub>) as the carrier gas. Finally, high-temperature GaN was grown for 30 min at  $1050^{\circ}$ C with a V/III ratio of 1400. To demonstrate that this method can control the polarity of the selected area, two types of lateral polarity structures were fabricated according to the method described above using patterned Al layers on sapphire substrates. One was fabricated using a circular patterned Al layer with a diameter of 20 µm and a pitch of 60 µm, and the other was fabricated using a striped patterned Al layer with a width of 30 µm and a pitch of 60 µm. The polarity of all GaN samples was investigated by selective etching in 2 M potassium hydroxide (KOH) solution at 75°C for 5 min. It is well known that the metal-polar surface is chemically stable to the KOH solution, while the N-polar surface is etched regardless of the growth method or condition [27,28]. The surface morphology of GaN was observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The optical properties were characterized by room-temperature photoluminescence (PL) spectroscopy using a 266-nm diode-pumped solid-state laser as the excitation source. The stress state of the GaN films was investigated by Raman spectroscopy using a 514-nm Ar-ion laser as the excitation source. The crystalline quality of the GaN films was investigated by high-resolution X-ray diffraction (XRD).

## 3. Results and discussion

Figure 2 shows SEM images of GaN grown on Al layers with different thicknesses. The GaN film grown on a sapphire substrate without an Al layer had a rough surface covered with hexagonal hillocks (Fig. 2(a)), which is a general feature of N-polar GaN [29]. This GaN film was completely etched away by the KOH solution as shown in the inset of Fig. 2(a). This shows that the GaN film grown without the Al layer had N-polarity. The thickness of this GaN varied between 1.0 and 1.4 µm depending on the position due to its hexagonal hillocks.



**Fig. 2.** SEM images of GaN grown on Al layers with different thicknesses: (a) without the Al layer, (b) 5 nm, (c) 10 nm, and (d) 15 nm. The inset of (a) shows the surface after potassium hydroxide (KOH) etching.

Figure 2(b) shows a GaN film grown on a 5-nm thick Al layer. It had a smooth surface and was stable in the KOH solution. This indicates that this GaN film had Ga-polarity. The thickness

of this GaN was almost constant at 1.2 µm. Meanwhile, when the thickness of the Al layer was in the range 2–5 nm, flat GaN films could be grown on it (not shown). However, as the thickness of the Al layer went above 5 nm, pits formed on the surface, and the shape of GaN gradually changed from a film to islands. Figure 2(c) shows the GaN grown on a 10-nm thick Al layer. It had Ga-polarity but was in the form of islands that had not fully coalesced. As the thickness of the Al layer further increased to 15 nm, randomly oriented GaN islands grew on it (Fig. 2(d)). These results imply that the crystal structure of GaN gradually changed from single crystal to polycrystal as the Al layer became thicker. Since the growing GaN is affected by the crystal structure of the Al layer, it can be seen that the thicker Al layer was recrystallized into a polycrystalline layer in the high-temperature nitridation process. The formation of a polycrystalline layer can be attributed to the random nucleation and growth (RNG) during the recrystallization process. Unlike solid-phase epitaxy in which crystallization occurs at the interface during annealing, RNG occurs at randomly distributed locations and forms polycrystalline grains, which cause the growth of polycrystalline GaN [30,31]. Meanwhile, it should be noted that the formation of polycrystalline grains by RNG may be affected by both nitrogen atoms supplied by NH<sub>3</sub> and amorphous aluminum oxide formed on the surface of the Al layer due to its oxidation in air.



**Fig. 3.** AFM images and root-mean-square (RMS) roughness values: (a) Sapphire substrate, (b) Nitrided sapphire substrate, (c) 5-nm thick Al layer deposited on a sapphire substrate using E-beam evaporation, and (d) Nitrided 5-nm thick Al layer. Nitridation was conducted using NH<sub>3</sub> at 1050°C under an N<sub>2</sub> atmosphere.

To confirm the presence of the Al layer after high-temperature nitridation, AFM measurements were conducted. Figures 3(a) and 3(c) show the surface of the sapphire substrate and 5-nm thick Al layer deposited on the sapphire substrate, respectively. The surface of the sapphire had a smooth step-terrace structure, whereas the E-beam evaporated Al layer appeared in the form of three-dimensional (3D) islands covering the sapphire substrate. After nitridation at 1050°C under an N<sub>2</sub> atmosphere, the step-terrace structure of the sapphire became blurred (Fig. 3(b)). In the case of the Al layer, it did not disappear by thermal etching during the high-temperature

nitridation and still existed in the form of 3D islands (Fig. 3(d)). The presence of the Al layer after high-temperature nitridation implies that the Ga-polar GaN film shown in Fig. 2(b) grew on the high-temperature nitrided Al layer instead of on the nitrided surface of the sapphire substrate.

To grow high-quality GaN film on the Al layer, we investigated the effect of high-temperature nitridation and the role of the Al layer as a nucleation layer. Figure 4(a) shows GaN grown on a 5-nm thick Al layer without high-temperature nitridation. Although the growth steps were the same except for the annealing step (high-temperature nitridation), the GaN grew in the form of islands instead of a film. This is because the Al layer did not recrystallize due to the absence of the annealing step. In the theory of solid-phase epitaxy, the annealing temperature affects the recrystallization of materials [30]. If the annealing temperature is not sufficiently high, solid-phase epitaxy will take a long time or cannot proceed. Therefore, annealing must be performed at appropriate temperatures to completely recrystallize the Al layer. To find the temperature at which recrystallization begins, GaN was grown on Al layers annealed at various temperatures with  $NH_3$  (not shown here). From the growth results, it is estimated that the recrystallization of the E-beam evaporated Al layer begins within the temperature range 700-800°C at which morphological changes occur. However, even if recrystallization occurred, the Al layer was not sufficiently recrystallized below 900°C because recrystallization was slow at low temperatures, and thus pits formed on the GaN surface. The formation of these pits can be prevented by increasing the annealing temperature, as shown in Fig. 2(b). In addition to annealing, nitridation influenced the growth of GaN. When the Al layer was annealed at 1050°C without NH<sub>3</sub>, GaN grew in the form of a film. However, it had numerous pits on the surface (Fig. 4(b)). This implies that the formation of pits is related to insufficient nitridation of the Al layer. The formation of these pits can be prevented by supplying  $NH_3$  in the annealing step, as shown in GaN in Fig. 2(b). Meanwhile, when the high-temperature GaN was directly grown without a GaN nucleation layer on the high-temperature nitrided Al layer, the resultant GaN grew in the form of a film but had a relatively rough surface due to the formation of round hillocks (Fig. 4(c)). This may be because the Al layer is too thin. For this reason, prior to the growth of high-temperature GaN, a GaN nucleation layer was additionally grown on the Al layer. As a result, a high-quality Ga-polar GaN film, similar to a conventional Ga-polar GaN film, could be grown on the E-beam evaporated Al layer, as shown in Fig. 2(b).



**Fig. 4.** SEM images of GaN grown on the 5-nm thick Al layers treated under different conditions: (a) no high-temperature nitridation, (b) annealing at  $1050^{\circ}$ C without NH<sub>3</sub>, and (c) annealing at  $1050^{\circ}$ C with NH<sub>3</sub>. In cases (a) and (b), a GaN nucleation layer grew on the Al layer after treatment but not in the case of (c).

Thus far, it has been shown that GaN film can be grown on E-beam evaporated Al layers through high-temperature nitridation. It can be inferred through the concept of solid-phase epitaxy that the E-beam evaporated Al layer can be recrystallized by annealing. However, it is somewhat difficult to accurately describe the process by which the Al layer recrystallizes. When the Al layer is exposed to air, amorphous aluminum oxide forms on the surface of the Al layer [32,33]. This amorphous aluminum oxide can be recrystallized into  $\alpha$ -phase Al<sub>2</sub>O<sub>3</sub> by solid-phase epitaxy [34,35]. In addition, since NH<sub>3</sub> was supplied during the annealing process,

the nitrogen atoms may affect the recrystallization. Therefore, further studies are needed to clarify the recrystallization process of the Al layer.

To evaluate the structural and optical properties of Ga- and N-polar GaN grown by this method, comparative analyses were performed with conventional Ga-polar GaN grown on a sapphire substrate. Figure 5(a) shows the Raman spectra of the GaN samples. The stress of the GaN films can be investigated by comparing the  $E_2$ (high) mode with a value of stress-free GaN [36]. For this comparison, the  $E_2$ (high) mode of bulk GaN was measured and used as the value of stress-free GaN. The measured  $E_2$ (high) mode was 567.2 cm<sup>-1</sup>, which is well-matched with the reported value of stress-free GaN [37,38]. The corresponding value for the Ga-polar GaN/5-nm Al layer was 569.6  $\rm cm^{-1}$ , which is higher than that for the conventional Ga-polar GaN. This indicates that the Ga-polar GaN/5-nm Al layer exhibits higher compressive stress than conventional Ga-polar GaN. In the case of N-polar GaN, the  $E_2$ (high) mode frequency was located at 567.2  $\text{cm}^{-1}$ , indicating that the stress in N-polar GaN is almost relaxed. Figure 5(b) shows the room-temperature PL spectra. The PL peak energies of the Ga-polar GaN/5-nm Al layer and N-polar GaN were 3.41 eV and 3.39 eV, respectively. These values are slightly lower than the 3.42 eV of the conventional Ga-polar GaN. It should be noted that PL emission in the Ga-polar GaN/5-nm Al layer should be spectrally blue-shifted when considering only the strain effect but became actually red-shifted. Generally, compressive (tensile) strain induces the spectral blue-shift (red-shift) of band-edge emission in the PL spectrum due to band structure modulation. However, a large defect density can lead to spectral red-shift and exhibits a long tail on the lower energy side in the PL spectrum because of bandgap fluctuations in connection with defect states [39]. Therefore, the shift of the PL peak energy should take both into account for a combined effect of the residual strain and native defects. In Fig. 5(b), a relatively long tail on the lower energy side in Ga-polar GaN/5-nm Al layer compared to the conventional Ga-polar GaN demonstrates the presence of defects. The presence of defects could possibly compensate for the strain-induced spectral blue-shift and further induces spectral red-shift in PL spectra.



**Fig. 5.** Comparative analysis of a Ga-polar GaN grown on the 5-nm thick Al layer, N-polar GaN, and conventional Ga-polar GaN: (a) Raman spectra and (b) Photoluminescence spectra.

Figure 6(a) shows the XRD phi scan of Ga-polar GaN grown on a 5-nm thick Al layer. The phi scan curve of the GaN ( $10\overline{1}2$ ) plane represents six peaks separated by 60° intervals, and it indicates the six-fold symmetry of the GaN. In addition, the peaks of the sapphire ( $10\overline{1}4$ ) plane and the GaN ( $10\overline{1}2$ ) plane were separated by 30°. This shows that the GaN film grown on a 5-nm thick Al layer was rotated by 30° with respect to the sapphire substrate, similar to conventional GaN grown on a sapphire substrate. Figures 6(b) and 6(c) show the normalized X-ray rocking curves (XRC) of the (0002) and ( $10\overline{1}2$ ) planes, respectively. In the XRCs of the (0002) reflection, the FWHM of the Ga-polar GaN/5-nm Al layer was 684 arcsec, which is larger

than the 453 arcsec of conventional Ga-polar GaN. In contrast, in the  $(10\overline{1}2)$  reflection, the FWHM of the Ga-polar GaN/5-nm Al layer was 964 arcsec, which is slightly smaller than the 1036 arcsec of conventional Ga-polar GaN. Because the broadening of the symmetric (0002) XRC is only sensitive to the screw and mixed threading dislocations, it can be seen that the Ga-polar GaN/5-nm Al layer has a higher screw and mixed threading dislocations compared to conventional Ga-polar GaN [40]. In the case of N-polar GaN, the FWHM value was 424 arcsec for the (0002) reflection and 709 arcsec for the ( $10\overline{1}2$ ) reflection, which were the lowest values for both reflections compared to the Ga-polar GaN samples.



**Fig. 6.** X-ray diffraction spectra of Ga-polar GaN grown on a 5-nm thick Al layer, N-polar GaN, and conventional Ga-polar GaN: (a) Phi scan of the Ga-polar GaN/5-nm Al layer for the GaN ( $10\overline{1}2$ ) and sapphire ( $10\overline{1}4$ ) planes and rocking curves of GaN films for the (b) (0002) and (c) ( $10\overline{1}2$ ) reflections. The thicknesses of both Ga-polar GaN/5-nm Al layer and conventional Ga-polar GaN were 1.2 µm and the thickness of N-polar GaN varied at 1.0–1.4 µm due to its hexagonal hillocks.

In addition to controlling the polarity of unipolar GaN films, two types of lateral polarity structures were fabricated using patterned Al layers to demonstrate that this method can selectively control the polarity of the selected area. Figures 7(a) and 7(c) show SEM images of lateral polarity structures grown on circular and striped patterned Al layers, respectively. The GaN grown on patterned Al layers had a smooth surface in both samples. However, the GaN grown on the sapphire surface had a rough surface covered by hexagonal hillocks. The surface morphologies of each area were similar to those of Ga- and N-polar GaN described in Figs. 2(b) and 2(a), respectively. The polarity of GaN grown in each area was investigated through polarity-dependent etching by KOH solution. Figures 7(b) and 7(d) show the surface of the lateral polarity structures after KOH etching. GaN grown on the patterned Al layers remained in both structures. This shows that Ga-polar GaN grew on the patterned Al layer. In contrast, the GaN grown on the sapphire surface was completely etched away by KOH solution. Thus, N-polar GaN was grown in areas where the Al layer was not deposited. These results indicate that the polarity of GaN can be controlled according to the pattern of the Al layer. Since this method can easily control the polarity of the selected area without complex processes, it is expected to be utilized in various applications requiring polarity control.



**Fig. 7.** SEM images of GaN lateral polarity structures grown on (a) the circular patterned Al layer with a diameter of 20  $\mu$ m and a pitch of 60  $\mu$ m and (c) the striped patterned Al layer with a width of 30  $\mu$ m and a pitch of 60  $\mu$ m. The KOH etched lateral polarity structures grown on (b) circular and (d) striped patterned Al layers.

## 4. Conclusions

In summary, we introduced a method for controlling the polarity of GaN using an E-beam evaporated Al layer on a sapphire substrate. For this purpose, a high-temperature nitridation process was designed to enable the amorphous Al layer to act as a nucleation layer for Ga-polar GaN growth. Using this method, we successfully grew single-crystal Ga-polar GaN on the E-beam evaporated Al layer. When the thickness of the Al layer was less than 5 nm, GaN grew in the form of a film, but as the thickness of the Al layer increased, the resulting GaN was in the form of islands. There was no significant difference in the optical and structural properties of Ga-polar GaN grown on a 5-nm thick Al layer and conventional Ga-polar GaN. Furthermore, on the surface of sapphire where the Al layer was not deposited, N-polar GaN could be grown via the same growth process. This implies that the polarity can be controlled by depositing an Al layer. In addition to controlling the polarity of unipolar GaN films, two types of lateral polarity structures were fabricated using patterned Al layers on sapphire substrates. The patterned Al layers prevented surface nitridation of sapphire and acted as a nucleation layer for Ga-polar GaN growth. As a result, it was demonstrated that Ga-polar and N-polar GaN could be selectively grown on the patterned Al layer and the sapphire surface, respectively. These results indicate that the polarity of GaN can be selectively controlled according to the pattern of the Al layer. Since the method using an E-beam evaporated Al layer can be used to control the polarity of GaN without complex processes, it is expected to be utilized in various applications requiring polarity control.

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