**GaN substrates having a low dislocation density and a small off-angle variation prepared by hydride vapor phase epitaxy and maskless-3D**

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1. Introduction

GaN (0001) substrates are mainly used in III-nitride laser diodes1) and in high-power LEDs.2) It is known that the incorporation of In into the InGaN active layers is affected by the off-angle of their base substrate.3) The In component of InGaN active layers controls their emission wavelength and is therefore dependent on the off-angle. GaN substrates have been also used in the development of power devices.4,5) It is also known that the concentration of C in n-type GaN drift layers grown by metal-organic chemical vapor deposition is affected by the off-angle of the base substrate.6) Because C acts as an acceptor in GaN, the carrier concentration in the drift layer is dependent on the off-angle.

Most of the currently available GaN (0001) substrates show an off-angle variation, measured as the difference between the maximum and minimum off-angles. This off-angle variation induces nonhomogeneous characteristics in devices produced from the substrate and thereby reduces their yield. Furthermore, detrimental amounts of threading dislocations (TDs) remain in these substrates. These act as nonradiative recombination centers7) or leak paths;8) consequently, a high threading dislocation density (TDD) reduces the efficiency and reliability of devices. GaN substrates having small off-angle variations and low TDDs, along with large diameters and low costs, are therefore required to improve device productivity.

Such GaN substrates have been produced by growing GaN (0001) boules by hydride vapor phase epitaxy (HVPE)9,10) and then machining the boules flat. These boules are generally curved as a result of their internal stress gradient, and their c-plane is also curved. The off-angle is defined as the angle between the surface normal and the c-axis. Curvature of the c-plane consequently induces off-angle variations in the resulting substrate, and larger substrate diameters result in greater off-angle variations. To reduce the off-angle variation, therefore, the curvature of the GaN boule must be reduced. According to Foronda et al.,11) the curvature depends on the density of the inclined TDs. All the issues discussed above could therefore be solved merely by reducing the TDD.

Fusion or annihilation reactions of TDs are the primary mechanisms available for TDD reduction.12) For these reactions, it is imperative that TDs meet one another. In GaN crystals, some TDs have a tilted propagation direction;12) consequently, increasing the growth thickness can reduce the TDD naturally.13) Fujito et al. reported that the TDD can be reduced to $1.2 \times 10^6$ cm$^{-2}$ by growing GaN to a thickness of 5.22 mm.13) To further reduce the TDD, a greater increase in thickness is required. However, it is generally difficult to grow ultrathick GaN crystals because cracks are easily generated. Alternatively, epitaxial lateral overgrowth, which produces a three-dimensional (3D) growth shape and thereby markedly tilts the propagation direction of most TDs, is a more effective method for reducing the TDD without ultrathick growth.14–17) This, however, is a selective growth technique with a mask pattern on the seed substrate and therefore involves the use of complicated processes such as photolithography. The existing approaches for reducing the TDD of GaN therefore all have disadvantages in terms of low productivity and high costs. To keep costs low, it is necessary to produce a 3D growth shape without using any masks or any ex-situ treatments on a normal seed substrate.

Voronenkov et al. previously reported the application of low-temperature HVPE growth to give a 3D shape on a normal GaN/sapphire template without any masks.18,19) They also reported that the 3D shape consisted of pits and hillocks and that GaN with a flat surface could be grown on it at high temperatures. They used this 3D structure to relieve the growth stress and they consequently obtained GaN boules about 3 mm thick without any cracks, even on GaN/sapphire templates.18–20) Inspired by their report, we developed our “maskless-3D method” which effectively reduces the TDD of the GaN crystal. The process flow is as follows. First, we produce pits sparsely; these trigger the 3D structure on a seed GaN (0001) substrate without any mask. We then enlarge the aperture diameter of the pits, while maintaining the pit density, to eliminate all residual c-plane facets. Next, we backfill the all pits to obtain a flat growth surface. Finally, we produce GaN substrates from the area grown with a flat surface.
TDs collect in the center of pits because they propagate in the direction perpendicular to the growth interface. To reduce the TDD effectively, it is crucial to increase the distances between the areas in which TDs collect. Of course, it is also crucial that the interface consists of skew facets only. Hence, in maskless-3D, the TDD can be significantly reduced by appropriately separating the trigger pits and by completely eliminating the c-plane facets. Because the trigger pits originated from various imperfection in the seed substrate, such as TDs or inversion domains, we used self-standing GaN as seed substrates to provide an easy method for increasing the distance between pits. By this technique, we produced GaN substrates of very high quality at a low cost. Here, we report details of this method and the characteristics of the resulting GaN substrates.

2. Experimental methods

2.1. HVPE growth

In this study, we used normal GaN (0001) substrates as seed substrates. The seeds had a polished flat surface, a diameter of 2 inches, a TDD of $3 \times 10^{6}$ cm$^{-2}$, and an off-angle variation of 0.5°. We used a conventional atmospheric HVPE reactor for GaN growth experiments. GaCl and NH$_3$ gases were used as sources of gallium and nitrogen, respectively. GaCl was produced by the reaction of gaseous HCl with molten Ga in the upstream region of the reactor at 800°C. SiH$_2$Cl$_2$ gas was used for doping with Si. A mixed H$_2$–N$_2$ carrier gas transported the source materials onto the seed substrate. The growth temperature was set appropriately between 990°C and 1050°C.

2.2. Maskless-3D

As mentioned above, to reduce TDD effectively, it is vital to control the distance between trigger pits and to cover the growth interface with skew facets only. The growth shape can be generally controlled by a suitable choice of growth conditions, such as the growth atmosphere, growth temperature, growth pressure, and V/III ratio. In this study, we therefore investigated the effect of the HVPE growth conditions on the trigger pit density and the subsequent growth shape. As an example, we report our studies on the optimal growth temperature for maskless-3D.

Figure 1 shows a cross-sectional schematic of the maskless-3D. The growth shapes at times $t_1$, $t_2$, and $t_3$ are also shown. First, we investigated the trigger pit density ($D_1$) and the diameter ($p_1$) at several growth temperatures. The temperature was varied between 990°C and 1017°C, while the GaCl partial pressure, NH$_3$ partial pressure, and H$_2$ concentration in the carrier gas were maintained at 6.33 kPa, 21.38 kPa, and 65.6%, respectively. The average distance between the pits ($D_{ave}$) can be estimated from the expression:

$$D_{ave} = \frac{1}{\sqrt{D_1}}.$$  \hspace{1cm} (1)

Subsequently, we grew GaN under the same conditions to time $t_2$, and we investigated the pit diameter $p_2$. The pit enlargement ratio is therefore calculated as follows:

$$\frac{\Delta p}{\Delta t} = \frac{p_2 - p_1}{t_2 - t_1}.$$ \hspace{1cm} (2)

As mentioned above, to reduce TDD effectively, it is crucial to completely remove the c-plane facet from the growth surface. Hence, the pits need to be enlarged so that they completely cover the surface. It is therefore necessary to continue the 3D growth to at least a time $t_3$ that is estimated from the following expression:

$$t_3 = t_1 + (D_{ave} - p_1) \frac{\Delta t}{\Delta p}.$$ \hspace{1cm} (3)

In addition, it is suitable that the GaN substrate is produced from the so-called two-dimensional (2D) growth region which is grown with a flat growth shape because the substrate with uniform electrical and physical properties is preferred. After complete removal of the c-plane facet, we therefore changed the GaCl partial pressure, the NH$_3$ partial pressure, and the growth temperature to 9.5 kPa, 15.83 kPa, and 1050°C, respectively, to expunge the pits and to induce later growth to proceed with a 2D growth shape. This 2D growth proceeded with Si doping. We then sliced a GaN wafer from the 2D growth region. The periphery of this wafer was beveled and an orientation flat was also formed. After grinding both sides of the wafer, remaining damage on the N polar surface was removed by alkaline etching, and the Ga polar surface was polished.

2.3. Evaluation

The growth shape was examined by differential interference contrast (DIC) microscopy and scanning electron microscopy (SEM). The transition of the growth shape was confirmed from the cross-sectional fluorescence microscopy image. The autodoped oxygen concentration in the 3D growth region was evaluated by secondary-ion mass spectrometry. The sheet resistance and carrier mobility of the completed substrate were evaluated by the eddy-current method, and the carrier concentration was thereby calculated. The TDD was estimated from two-photon excitation photoluminescence (2PPL) mapping images. The state of the propagation and collection of TDs was also examined by 2PPL mapping. The area of each 2PPL image was 250 × 250 μm$^2$. The TDD was also estimated from the density of etch pits formed by treatment with molten KOH and NaOH. The off-angle variation, the curvature radius of the c-plane, and the lattice perfection were evaluated from X-ray rocking curve (XRC) measurements made by using a four-circle Malvern Panalytical X’Pert$^3$ MRD (using Cu Kα radiation) equipped with a hybrid monochromator, consisting of an X-ray mirror.
and a two-bounce Ge (220) monochromator. A 1/2° divergence slit was placed before the monochromator. The incident beam height \( H_i \) was 1.4 mm. When an X-ray beam is incident on a sample at a Bragg angle \( \theta_B \), the illuminated length \( L \) can be written as follows:

\[
L = \frac{H_i}{\sin \theta_B}
\]

(4)

Because these XRC evaluations are complicated, the details are described below in Sects. 2.4 and 2.5.

2.4. Off-angle variation and curvature radius

XRC measurements were carried out every 5 mm along a line passing through the center of the sample in the direction parallel or perpendicular to the off direction. GaN (0002) was used as the diffraction plane. From the definition of a radian, the relationship between the XRC peak top angle \( \omega \) and the measurement position \( x \) (the distance from the center) can be written as follows:

\[
\omega = \frac{1}{R} x + \omega_0,
\]

(5)

where \( R \) is the curvature radius of the c-plane, and \( \omega_0 \) is the XRC peak top angle at the center. \( R \) can therefore be calculated from the slope of the relationship. The off-angle variation was calculated from the difference between the maximum and minimum \( \omega \) values in the sample diameter, which was oriented parallel to the off direction.

2.5. Lattice perfection

Lattice perfection is generally estimated from the full width at half maximum (FWHM) of the XRC. The FWHM of GaN (0002) XRC and that of GaN (10–10) are termed, respectively, the tilt angle and the twist angle. In the case of a GaN (0001) sample, measurements of the GaN (10–10) XRC lack accuracy. The twist angle was therefore extrapolated from the FWHM of an inclined plane XRC, such as GaN (10–12). In this study, we therefore estimated twist angle by using the following expression:

\[
\alpha = \sqrt{(\beta_{0002} \cos \chi)^2 - \beta_{10–12}^2},
\]

(6)

where \( \alpha \) is the twist angle, \( \beta_{0002} \) is the tilt angle, \( \beta_{10–12} \) is the FWHM of GaN (10–12) XRC, and \( \chi \) is the angle between the GaN (10–12) and GaN (0002).

In addition, the tilt angle can include the influence of lattice curvature. Moram et al. have reported an empirical relationship between the tilt angle derived from curvature \( \beta_{\text{curv}} \) (in degrees) and the illuminated length \( L \) as follows:

\[
\beta_{\text{curv}} = 52 \frac{L}{R}
\]

(7)

In our XRC optics, the tilt angle of a perfect GaN crystal, which is the so-called intrinsic value, was estimated to be 25.7 arcsec, the \( \theta_B \) of GaN (0002) was 17.28°, and \( L \) was 4.71 mm approximately. The tilt angle of the GaN substrate, which had a curvature radius of less than 34.3 m, therefore included the curvature component.

3. Results and discussion

3.1. Appropriate growth conditions for maskless-3D

Figure 2 shows the growth-temperature dependence of the trigger pit density and the average distance between pits. The growth time was 10 min. The insets show examples of DIC images of the growth surface. We found that the trigger pit density decreased and the average distance between pits increased with increasing growth temperature. Subsequently, we grew GaN under the same conditions for longer than 10 min and we investigated the enlargement in the ratio of pits as calculated by using Eq. (2). By using Eq. (3), we estimated the required time to expunge the c-plane facet in the growth surface completely, and the results are shown in Fig. 3. We found that growth temperatures of above 1013 °C were not optimal because the value of \( t_3 \) required was too long.

Next, we confirmed experimentally that the estimated time \( t_3 \) was reasonable. A cross-sectional fluorescence microscopy image of a crystal grown under the same conditions as mentioned above at 1017 °C for three hours is shown in Fig. 4(d). In this image, the area showing a relatively dark fluorescence corresponds to a track of the area grown with a skew facet. As expected, the c-plane facet remained in the growth surface. In contrast, at a temperature below 1009 °C, it was confirmed that the c-plane facet was completely expunged in approximately the estimated time \( t_3 \), as shown in Figs. 4(a)–4(c).

In addition, we also investigated the dependence of the trigger pit density on the V/III ratio and on the \( H_2 \) concentration in the carrier gas. Here, specific numerical data are omitted and only the trends are described. On
increasing the V/III ratio, the pit density increased, whereas on increasing the H₂ concentration, the pit density decreased.

3.2. Production of high-quality GaN substrates by using maskless-3D

For the reason described above, maskless-3D was carried out at a growth temperature below 1000 °C and was continued until the c-plane facet was completely expunged. Subsequently, we changed the growth conditions to completely expunge the pits and to induce later growth to proceed with a 2D growth shape. The growth shape consequently changed from 3D to 2D, as shown in the cross-sectional fluorescence microscopy images in Fig. 5. It was found, however, that the number of pits that remained in the as-grown surfaces increased with decreasing 3D growth temperature, as shown in Fig. 6. We therefore found that the optimal temperature for maskless-3D is around 1008 °C. Figure 7(a) shows a cross-sectional 2PPL mapping image of a 3D growth region grown at 1008 °C. In this image, the area showing a relatively bright luminescence corresponds to the track of the area grown with a skew facet, similar to the cathodoluminescence mapping image. The other area corresponds to the track of the area grown with a c-plane facet, and the dark lines in the bright area correspond to dislocations. It was observed that many TDs were gathered and fused in the 3D structure. These four images are also spatially contiguous. It was found that TDD was reduced to about $4 \times 10^5 \text{cm}^{-2}$ and the TDs were uniformly distributed over a wide area of $500 \times 500 \mu\text{m}^2$. Finally, we sliced a GaN wafer from the 2D growth region and subjected it to the processes described in Sect. 2.2. Figure 8 shows the appearance of the treated substrate, which had a thickness of approximately 400 μm.

3.3. Properties of the completed GaN substrate

Table I shows the electrical properties of the completed GaN substrate. These values were well controlled and were approximately equivalent to those given in our previous report. Figure 9 shows the relationship between the XRC peak top angles of GaN (0002) and their corresponding measurement positions in the substrate. The curvature radius of the c-plane was approximately 50 m. It was therefore found that the off-angle variation was approximately 0.05° over a diameter of 2 inches. We were able to confirm experimentally that the lattice curvature and the off-angle variation could be improved by reducing the TDD, as explained by Foronda et al. Figure 10(a) shows the GaN (0002) XRC. Its tilt angle was 28.5 arcsec, a value very near the intrinsic value. For reference, data for our conventional GaN substrate produced by void-assisted separation (VAS) are also shown. The curvature radius of the VAS-GaN substrate was 4.66 m. Its tilt angle is affected by curvature, as mentioned in Sect. 2.5. The tilt angle derived from the curvature was estimated by Eq. (7) to be approximately 189 arcsec, which corresponded closely to its measured value. Figure 10(b) similarly shows their GaN (10−12) XRCs. The twist angles (α) were estimated by using Eq. (6), and the results are listed in Table II. It was found that the crystal perfection of the GaN substrate produced by maskless-3D was considerably better than that of the VAS-GaN.
substrate. Figure 11 shows a DIC image example of the surface etched by molten KOH and NaOH. This confirmed that the TDD of the substrate produced by maskless-3D was in the low $10^5$ cm$^{-2}$ range.

3.4. Discussion

As reported in our previous work, 29) plastic deformation occurs when a thick crystal of GaN is grown on a 2D interface on a seed having a 3D growth tracks in its surface. The deformation was attributed to the considerable lattice

Table I. Electrical properties of the completed substrate.

| Property            | Value     |
|---------------------|-----------|
| Resistivity (Ω cm)  | $1.60 \times 10^{-2}$ |
| Carrier mobility (cm$^2$ V$^{-1}$ s$^{-1}$) | 355 |
| Carrier concentration (cm$^{-3}$)     | $1.10 \times 10^{18}$ |

Fig. 7. (a) A cross-sectional 2PPL mapping image of the area surrounding the 3D growth region grown at 1008 °C. The image is composed of spatially contiguous three images. The area showing a relatively bright luminescence (i) corresponds to the track of the area grown with the skew facet. The other area (ii) corresponds to the track of the area grown with $c$-plane facet, and the dark lines in the bright area correspond to dislocations. (b) 2PPL mapping images of the as-grown surface of this crystal. The average TDD from these four images is $4 \times 10^5$ cm$^{-2}$.

Fig. 8. (Color online) The appearance of a completed GaN substrate fabricated by the maskless-3D method at 1008 °C.

Fig. 9. The relationship between the XRC peak top angles of GaN (0002) and their each measurement positions in a completed substrate fabricated by the maskless-3D method at 1008 °C. The measurement position was shifted along each orthogonal [10$\overline{1}0$] and [11$\overline{2}0$] directional line passing through the center of the substrate. Black dots and white dots correspond to [10$\overline{1}0$] and [11$\overline{2}0$], respectively, in the shift direction of the measurement position. The solid line is the result of fitting by Eq. (5).
mismatch between the 2D growth region and the seed. The mismatch was brought about by a difference in the oxygen concentration. The area grown with a 3D interface had an oxygen concentration of around $10^{19}$ cm$^{-3}$. In this work, however, no plastic deformation occurred, although the growth mode was similar. This was because the oxygen concentration in a similar area was relatively low. Hence, we investigated the oxygen concentration in the 3D growth region at 1008 °C. As the result, we found that this concentration was as low as $9 \times 10^{17}$ cm$^{-3}$, which would not affect the lattice constant. To understand why the oxygen incorporation efficiency was low in this area, we then investigated the orientation of the facet plane constructing the pits. Figure 12(a) shows a cross-sectional SEM image of the region near the 3D growth surface. The sample was forcibly cleaved along the (11−20) plane. We found that the angle between the horizontal and the ridge line was approximately 43°. In this case, the angle of the facet plane was 47.3° geometrically, as shown in Fig. 12(b). We also found that the facet plane was inclined in the [11−23] direction. The orientation was therefore (11−23). The growth rate of the facet plane, $v_f$, can be therefore expressed geometrically as follows:

$$v_f = v_c \cos 47.3° - \frac{1}{2} \frac{\Delta p}{\Delta t} \sin 47.3°,$$

where $v_c$ is the growth rate of the c-plane, which in this case was about 300 μm h$^{-1}$. By using the $\Delta p/\Delta t$ value in Fig. 3, $v_f$ was estimated to be approximately 130 μm h$^{-1}$. This value was much greater than the value reported in our previous work, where the 3D growth was carried out by the Na-flux method.29) This might explain why the oxygen concentration in the 3D growth region was low in the present study.

In addition, we were interested in the possibility of applying 3D growth multiple times and in the effects of such a process. Such growth can be carried out continuously because maskless-3D does not require any masks, and the process is therefore easier to carry out than a previous similar technique using masks.17) Figure 13(a) shows a cross-sectional fluorescence microscopy image of an experimentally produced crystal with a double 3D structures. As expected, we confirmed that such growth proceeded with no problems. Figure 13(b) shows a 2PPL mapping image of the as-grown surface of the crystal. To confirm this result, etch pits counting was also carried out as shown the DIC image in Fig. 13(c). We found that the TDD was further reduced to $10^4$ cm$^{-2}$ on applying the double 3D structure. The curvature radius of the c-plane, investigated after processing the 2D layer in a free-standing manner, was found to be approximately 215 m, and the off-angle variation was thereby estimated to be approximately 0.01° in a 2 inch diameter. This technique is therefore effective in producing GaN substrates of remarkably high quality.

Finally, we wish to discuss our intended future work on maskless-3D. As mentioned above, maskless-3D does not require any masks on the base, and its growth shape can be controlled as required by changing the HVPE growth conditions only. Even for the production of GaN substrates of diameters of over 4 inches, the maskless-3D technique could therefore be applied, and the TDD could be similarly reduced. The off-angle variation of 4 inch or 6 inch substrates produced by maskless-3D is expected to be 0.1° or 0.15°, respectively. GaN substrates having these values are suitable for use in either optical devices or power devices. Furthermore, the maskless-3D process could also be applied in conjunction with the VAS method, which should markedly reduce the cost of producing high-quality GaN substrates. We intend to report on demonstrations of the production of such substrates soon.

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**Table II.** Tilt angles and twist angles of GaN substrates produced by maskless-3D and by VAS.

| Producing method | $\beta_{0002}$ (arcsec) | $\alpha$ (arcsec) |
|------------------|------------------------|------------------|
| Maskless-3D      | 28.5                   | 33.4             |
| VAS              | 172.4                  | 731.5            |

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Fig. 10. (Color online) (a) GaN (0002) XRCs of completed GaN substrates. (b) GaN (10−12) XRCs of completed GaN substrates. The red lines correspond to data for the GaN substrate fabricated by the maskless-3D method. The black lines correspond to data for a substrate fabricated by the VAS method.9)
4. Conclusions

We have developed an epochal technique termed the “maskless-3D method” for the production of GaN (0001) substrates with a low TDD and a small off-angle variation. This method is momentous and economical, because it does not require the use of any masks in producing the 3D structures that cause marked tilting of the direction propagation of TDs. We investigated appropriate maskless-3D growth conditions and we succeeded in producing a usable GaN substrate with a diameter of 2 inches, a TDD of around $4 \times 10^5$ cm$^{-2}$, and an off-angle variation of 0.05°. In addition, we demonstrated the growth of GaN with a double maskless-3D structure to demonstrate the feasibility of further reducing the TDD to as little as $10^4$ cm$^{-2}$. We are convinced that GaN substrates produced by our maskless-3D method will...

Fig. 12. (Color online) (a) A cross-sectional SEM image of the region around the 3D growth surface produced by the maskless-3D method at 1008 °C. This sample was forcibly cleaved along the (11−20) plane. The angle between the horizontal and the ridge line is approximately 43°. (b) A geometrical illustration of this pit. Here, the shape of the pit opening is assumed to be a regular hexagon. When the ridge angle is 43°, the angle of the facet plane of the pit is estimated to be 47.12°. This is an approximation of the angle of the (11−23) facet, which is 47.3°.

Fig. 13. (Color online) (a) Cross-sectional fluorescence microscopy image of a GaN crystal with a double 3D structure. (b) 2PPL mapping image of its as-grown surface. Ten dark spots are observed. The TDD estimated from this image is therefore $1.54 \times 10^4$ cm$^{-2}$. (c) A DIC image of its surface showing the etch pits. The TDD estimated from this image is $1.17 \times 10^4$ cm$^{-2}$.
promote the development and production of excellent III-nitride-based devices.

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