Homoepitaxial growth by halide vapor phase epitaxy of semi-polar GaN on ammonothermal seeds

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During crystallization of GaN in the c-direction by halide vapor phase epitaxy the formation of semi-polar facets as well as growth in semi-polar directions are observed. Crystalization process on these semi-polar facets is described in this paper in detail. Ammonothermal GaN crystals were used as seeds. Morphology, structural quality, growth rate, impurities concentration and free carrier concentration are analyzed and compared to data obtained for crystals grown in the c-direction. © 2019 The Japan Society of Applied Physics

1. Introduction

Halide Vapor Phase Epitaxy (HVPE) is the most common approach for manufacturing gallium nitride substrates.1–6 Main advantages of this technique are relatively high growth rate and possibility to obtain high-purity material.5,7–11 HVPE-GaN crystals are usually grown in the c-direction. Unfortunately, during crystallization runs in this direction growth in lateral directions appears.11,12 This phenomenon leads to reduction in diameter of the c-plane and formation of three semi-polar (10–11), (10–12), and (11–22) facets on the growing crystal’s edges.13 In order to understand the influence of lateral growth on GaN crystallization in the c-direction, crystallization in these naturally occurring semi-polar directions should be investigated.

In this paper, crystallization processes on the above-listed semi-polar surfaces is described in detail. Ammonothermal GaN crystals were used as seeds.14–20 All of them as well as as-grown GaN layers were examined by Differential Interference Contrast (DIC) microscopy, X-ray diffraction (XRD), Raman spectroscopy, and secondary ion mass spectrometry (SIMS). Influence of growth direction on growth rate and properties (morphology, structural quality, impurities concentration and free carrier concentration) of obtained crystals is demonstrated and discussed. All these results are analyzed in terms of GaN growth in the c-direction.

2. Experimental methods

The semi-polar and c-plane ammonothermal GaN crystals (Am-GaN) were placed on a susceptor of a home-made HVPE-reactor, in the manner presented in the Fig. 1(a). The distribution of reactants on the surface of the susceptor was constant. It can be assumed that the growth conditions for all substrates were the same. Then, 2 h long HVPE crystallization experiment was performed. A scheme of the HVPE reactor is presented in Fig. 1(b). GaCl was supplied vertically over the surface of a susceptor with the use of a shower head type quartz nozzles. NH3 was supplied by a quartz nozzle located on the same level as the susceptor and a few millimeters distant from it. The following growth conditions were applied: growth temperature of 1045 °C, temperature of GaCl synthesis of 850 °C, HCl flow of the level of 48 ml min−1 diluted in H2, NH3 flow of 960 ml min−1 diluted in H2, and 4000 ml min−1 of H2 as the carrier gas.

After the crystallization processes all samples were studied by DIC microscopy, XRD, Raman spectroscopy, and SIMS. The growth rate was determined by a comparison of the crystal’s weight before and after an HVPE experiment.

All samples: seeds and new grown layers were characterized by XRD rocking curve scans with a Panalytical Empyrean diffractometer (CuKα, 40 kV, 30 mA) equipped with a Pixcel3D detector. In order to determine the bowing radii of the crystallographic planes changes of incident angle for the reflection (10.1), (10.2), (11.2), and (00.6) were analyzed on the crystal surface on a distance of 5 mm with a 1 mm step. Raman spectra were taken at room temperature in a backscattering configuration using a Jobin-Yvon T64000 spectrometer equipped with a CCD camera. A 514.53 nm line of an argon laser as an exciton source were used. SIMS was applied on the Am-GaN seeds and HVPE layers to investigate incorporation of impurities. All the SIMS measurements were performed on mechanically polished samples placed together (seeds and then a new grown material) in the measuring chamber.

3. Results and discussion

3.1. Results

Ammonothermal GaN crystals with the following surfaces: {10–11}, {10–12}, {11–22}, and {0001}, were used as seeds for HVPE growth run. Their surfaces were prepared to an epiready state by mechanical and mechnano-chemical polishing and cleaning. The semi-polar wafers were obtained, by proper slicing, lapping and polishing procedures, from 1 cm thick Am-GaN boule grown in the –c direction. This bulk material was a result of a few growth runs in the –c direction and, therefore, on the examined semi-polar surfaces some interfaces were well visible under UV illumination (see Fig. 2).
In turn, (0001)-plane substrate was very uniform in sense of color under UV illumination. All crystals used as seeds for the HVPE growth were of high structural quality (see description below). The impurities concentrations determined by SIMS in the applied seeds are presented in Table I. The samples were examined in the center of the surfaces. Additionally, in order to check seed’s uniformity, one of them was checked in three points [see Fig. 2(c)].

Figure 3 presents a bird’s eye view and DIC images of different surface morphologies for the HVPE-GaN samples grown in [0001], [10–11], [10–12], and [11–22] directions. One can see areas without strong instabilities. Crystallization in the [0001] direction was governed by step flow. Close to the edge, formation of hillocks and a propagation of macrosteps from tops of hillocks [see Fig. 3(a)] was observed. In case of the [10–11] direction, a macroscopically stable growth mode with macrosteps flow was found [see Fig. 3(b)]. Figure 3(c) presents morphology of the crystal grown on (10–12) plane. Macroscopically unstable growth dominated by step bunching was detected. A stable growth mode dominated by hillocks was observed during the crystallization run in [11–22] direction [Fig. 3(d)].

Data of XRD measurements of the ammonothermal seeds and HVPE layers are presented in Table II. In case of the seeds values of FWHM were in the range 15–40 arcsec. These values for HVPE layers increased to 35–80 arcsec. XRD measurements showed deterioration of the structural quality of the HVPE crystals. Values of FWHM of the layers crystallized on the planes (10–11) and (10–12) were twice wider than those for the used substrates (see Table II). In the case of growth in the [11–22] direction, the peak was four times wider. This expansion was not related to the bending of

**Table I.** Results of SIMS measurements performed in areas marked in Fig. 1 of ammonothermal seeds.

|     | (0001) | (10–11) | (10–12) | 1   | (11–22) | 2   | 3   |
|-----|--------|---------|---------|-----|---------|-----|-----|
| H [cm\(^{-3}\)] | 1 · 10\(^{19}\) | 7 · 10\(^{18}\) | 4 · 10\(^{18}\) | 1 · 10\(^{19}\) | 6 · 10\(^{18}\) | 5 · 10\(^{19}\) |
| C [cm\(^{-3}\)] | 8 · 10\(^{16}\) | 9.5 · 10\(^{17}\) | 3 · 10\(^{17}\) | 1.5 · 10\(^{18}\) | 9.5 · 10\(^{16}\) | 4.5 · 10\(^{17}\) |
| O [cm\(^{-3}\)] | 1.5 · 10\(^{19}\) | 5 · 10\(^{18}\) | 3.5 · 10\(^{18}\) | 6.5 · 10\(^{18}\) | 4.5 · 10\(^{18}\) | 5 · 10\(^{19}\) |
| Si [cm\(^{-3}\)] | 1 · 10\(^{17}\) | 2 · 10\(^{17}\) | 2.5 · 10\(^{17}\) | 2 · 10\(^{17}\) | 1.5 · 10\(^{18}\) | 2 · 10\(^{18}\) |
| Na [cm\(^{-3}\)] | 1 · 10\(^{16}\) | 1 · 10\(^{16}\) | 1.5 · 10\(^{16}\) | 1 · 10\(^{16}\) | 2.5 · 10\(^{16}\) | 7 · 10\(^{17}\) |
| Mg [cm\(^{-3}\)] | 5 · 10\(^{15}\) | 8 · 10\(^{16}\) | 6 · 10\(^{16}\) | 9.5 · 10\(^{16}\) | 2 · 10\(^{17}\) | 8 · 10\(^{17}\) |
| Mn [cm\(^{-3}\)] | 6 · 10\(^{15}\) | 1.5 · 10\(^{17}\) | 1 · 10\(^{17}\) | 2 · 10\(^{17}\) | 2 · 10\(^{17}\) | 1 · 10\(^{18}\) |
| Fe [cm\(^{-3}\)] | 3 · 10\(^{15}\) | 5 · 10\(^{15}\) | 4 · 10\(^{15}\) | 6 · 10\(^{15}\) | 1.5 · 10\(^{16}\) | 2 · 10\(^{16}\) |
| Zn [cm\(^{-3}\)] | 8 · 10\(^{16}\) | 4.5 · 10\(^{17}\) | 4.5 · 10\(^{17}\) | 5 · 10\(^{17}\) | 1.5 · 10\(^{18}\) | 2.5 · 10\(^{18}\) |

**Table II.** XRD results for seeds and HVPE crystals grown on polar and semi-polar planes.

|     | (10–11) | (10–12) | (11–22) | (0001) |
|-----|---------|---------|---------|--------|
| Seeds |
| FWHM [arcsec] | 18 | 24 | 15 | 38.5 |
| R [m] | 8.5 | 17 | 14 | 11.5 |
| HVPE layers |
| FWHM [arcsec] | 34.5 | 46 | 62.5 | 78 |
| R [m] | 7 | 13.5 | 18.5 | 10 |
the samples, which remained at the same level. Probably, the observed deterioration was caused by other defects. The detailed X-ray analysis revealed low-angle boundaries in the new grown layers. The samples had a mosaic structure that dramatically affected the structural quality. It can be assumed that the obtained layers had an increased amount of dislocations by etching in the molten eutectic KOH/NaOH turned out to be impossible.

Table III presents average growth rates for GaN layers grown in different crystallographic directions. It should be noted that the fastest growth was observed in the [10–12] direction. In turn, the rate in the [10–11] direction was the lowest.

Raman spectrometry measurements for all grown samples are presented in Fig. 4. The spectrum for GaN growth in the c-direction showed two peaks, $E_{2}^{\text{high}}$ and $A_{1}(\text{LO})$, positioned at 568 cm$^{-1}$ and 734.5 cm$^{-1}$, respectively [see Fig. 4(a)]. For the crystal grown in the [11–22] direction [Fig. 4(b)] the position of $E_{2}^{\text{high}}$ peak did not change in comparison to growth in c-direction. It should be, however, noted that $A_{1}(\text{LO})$ peak disappeared. Two additional peaks appeared and were correlated with longitudinal plasmon-phonon modes, LPP$^+$ and LPP$^-$. The LPP$^+$ mode at 2549.5 cm$^{-1}$ was very broad. According to21,22) this value corresponds to LPP$^+$ mode at around 555 cm$^{-1}$. This means that the LPP$^+$ peak was expected to occur between peak $A_{1}(\text{TO})$, which was located at 531.5 cm$^{-1}$, and $E_{1}(\text{TO})$—located at 558.5 cm$^{-1}$. However, the LPP$^+$ is spectrally broad and expected to be extremely weak compared to the surrounding TO-modes. For GaN grown in the [10–11] direction [Fig. 4(c)] the $E_{2}^{\text{high}}$ peak was also at the same position. It is difficult to detect the LPP$^+$ peak but after a detailed study it can be found at 1353 cm$^{-1}$. Unfortunately, LPP$^-$ peak was not detected. From theoretical predictions, its position can be estimated at around 450 cm$^{-1}$. For the sample grown in the [10–12] direction [Fig. 4(d)] the $E_{1}(\text{TO})$ peak appeared at 556.5 cm$^{-1}$ and $A_{1}(\text{TO})$ mode disappeared. The LPP$^-$ mode is well visible at 498 cm$^{-1}$. This value corresponds to LPP$^-$ mode at around 1420 cm$^{-1}$.

In heavily doped semiconductors, longitudinal modes are coupled with plasmons resulting in new LPP modes which positions are very sensitive to the free carrier concentration.23,24) Therefore, from the positions of the LPP modes concentration of free carriers can be estimated with the following relation:

$$ n = \left( \frac{\omega_{L} - \omega_{\mu}}{\omega_{T}} \right)^{2} \frac{\varepsilon_{\infty} \varepsilon_{0} m^{*}}{e^{2}}, $$

where $\omega_{L}$ is the position of the low frequency LPP (LPP$^-$) mode and the high frequency LPP (LPP$^+$) mode, $\omega_{\mu}$ is the transversal phonon frequency, $\varepsilon_{\infty}$ is the high frequency dielectric constant of GaN, $\varepsilon_{0}$ is the vacuum permittivity, $m^{*}$ is the effective mass of electron, and $e$ is electron charge.

Values of free electron concentrations calculated from Eq. (1) are shown in Table IV. This table also presents results of all SIMS measurements. Concentrations of hydrogen, carbon, iron, manganese and magnesium were at the same level for all samples. The highest concentration of oxygen donors was observed in crystals grown in the semi-polar directions. It varied from $1 \times 10^{19}$ cm$^{-3}$ to $6 \times 10^{15}$ cm$^{-3}$. The silicon concentration was in the range $6–9 \times 10^{16}$ cm$^{-3}$. In the crystal grown in the c direction the Si concentration was $8 \times 10^{16}$ cm$^{-3}$ and oxygen concentration was lower than SIMS detection limit.

3.2. Discussion

In this paper an influence of growth directions on the morphology, structural quality, growth rate, carrier concentration, and impurities contaminations of three semi-polar crystals: (10–11), (10–12), and (11–22) was investigated in detail and compared to growth on the (0001) plane. As already mentioned, the semi-polar seeds were sliced from a boule obtained after a few subsequent ammonothermal growth runs. The investigated ammonothermal substrates were not homogeneous in terms of their optical properties.
There were areas with different shades of blue and gray, and the (10–11) substrate even had a yellow area. SIMS analysis showed that differences in color is associated with differences in the concentration of impurities in particular regions (see Table I). Therefore, the electrical properties of individual areas were also different. Nevertheless, despite such diversity within each substrate, they all could be prepared by mechanical and mechano-chemical polishing to the epi-ready state. In addition, X-ray measurements showed high structural quality of the samples. Some values of FWHM of rocking curves were close to the limit of equipment used for measurements. Worse values of FWHM for the HVPE-GaN layers can be associated with formation of defects in the new grown layers due to inhomogeneous concentration of dopants in the seeds (see Fig. 2 and Table I). The lack of homogeneity may lead to local changes in the lattice parameters of the seeds and generation of stress in the new grown layers. Additional studies are required to explain presented phenomenon.

It should be noted that the growth conditions were selected for a stable growth on substrates oriented towards the c-direction. The morphology of the obtained HVPE crystals showed that these chosen conditions seem to be suitable also for crystallization of GaN in the semi-polar directions. Generally, the growth modes of the obtained layers showed no instabilities. Crystallization on the (11–22) plane was dominated by formation of hillocks and propagation of macrosteps tops of the hillocks. Numerous hills in the form of cones and bubbles were clearly visible on the surface. Lack of explicitly oriented facets may indicate a poor selectivity of growth. The layers grown in the [10–11] and [10–12] directions were dominated by step flow and step bunching. Directions of the steps were the same in both cases and they were perpendicular to the c direction. In addition, in the case of crystal grown in the [10–12] direction, the formation of new side facets was observed [see Fig. 3(c)] and the closing of the main crystallization front plane was performed.

Raman spectra of the crystallized semi-polar layers were significantly different from the spectrum of crystal grown in the c direction. The main difference was due to the free carrier concentration. While the crystal grown on the c-plane had an electron concentration below $1 \times 10^{17}$ cm$^{-3}$, the semi-polar layers had two to three orders of magnitude higher

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**Table IV.** Results of free carrier concentration calculated from Raman spectra and SIMS measurements for obtained crystals.

|      | (10–11) | (10–12) | (11–22) | (0001) |
|------|---------|---------|---------|--------|
| n [cm$^{-3}$] | $1.5 \cdot 10^{19}$ | $2 \cdot 10^{19}$ | $8.5 \cdot 10^{19}$ | $<10^{17}$ |
| H [cm$^{-3}$] | $9 \cdot 10^{16}$ | $8 \cdot 10^{16}$ | $2 \cdot 10^{17}$ | $1 \cdot 10^{17}$ |
| C [cm$^{-3}$] | $1.5 \cdot 10^{16}$ | $2 \cdot 10^{16}$ | $4 \cdot 10^{18}$ | $1 \cdot 10^{19}$ |
| Fe [cm$^{-3}$] | $3 \cdot 10^{15}$ | $2 \cdot 10^{15}$ | $2 \cdot 10^{18}$ | $5 \cdot 10^{18}$ |
| Mn [cm$^{-3}$] | $2 \cdot 10^{17}$ | $2.5 \cdot 10^{15}$ | $2 \cdot 10^{18}$ | $1 \cdot 10^{18}$ |
| Mg [cm$^{-3}$] | $1 \cdot 10^{16}$ | $7 \cdot 10^{14}$ | $5 \cdot 10^{14}$ | $5 \cdot 10^{14}$ |
| O [cm$^{-3}$] | $1 \cdot 10^{19}$ | $1.5 \cdot 10^{19}$ | $6 \cdot 10^{19}$ | $4 \cdot 10^{16}$ |
| Si [cm$^{-3}$] | $9 \cdot 10^{16}$ | $6 \cdot 10^{16}$ | $9 \cdot 10^{16}$ | $8 \cdot 10^{16}$ |
values—up to $8.5 \times 10^{19}$ cm$^{-3}$. The SIMS analysis disclosed that the main donor was oxygen. All unintentional impurities were, in general, at the same level for both semi-polar crystals and the reference sample crystallized in the $c$-direction. The noticeable difference, was observed in case of oxygen. The concentration of this element in the obtained samples coincided perfectly with the electron concentration calculated from the positions of the LPP modes.

One of important factors limiting the HVPE-GaN growth in the $c$-direction is associated with the anisotropy of the growth and crystallization occurring in lateral directions. During the growth of GaN in the $c$-direction, even if the growth starts from a round shape of a seed, the natural hexagonal shape of GaN appears. Reference 13 published a detailed discussion on the formation of a dodecagon shape even if the growth starts on a round substrate. It is well known that for GaN the $a$-direction is the fastest growth direction and the $m$-direction the slowest one. Therefore, during a crystallization process at first all $(11\bar{2}0)$ facets disappear and all $(10\bar{1}0)$ facets appear and start to dominate. Then, the inclined facets $(10\bar{1}1)$ and $(10\bar{1}2)$ on $m$-plane sides and $(11\bar{2}2)$ facets on $a$-plane sides are formed. It is clearly shown in Fig. 5(a) where a part of HVPE-GaN growing on the round seed is presented with lines marking the seed and appearing facets. The formation of this equilibrium shape through the collapse of the growth facet reduces the size of the $c$-plane. Figures 5(b) and 5(c) present cross-sectional schematic images of the bulk GaN crystal for $a$ and $m$ planes. A DIC image of $m$-plane bulk GaN cross-section is presented in Fig. 5(d). Three areas are marked: seed, part of GaN grown in $c$ direction, and laterally grown part of the crystal.

The second important factor limiting the HVPE-GaN growth in the $c$-direction is associated with high stresses appearing in the obtained material close to the edge. As simulations and Raman spectroscopy showed,\textsuperscript{25,26} the area near the edge of the crystal is the main source of stress. Forces acting on the obtained crystal, associated with the existence of lateral growth, can be so large that they can lead to deterioration of structural quality and even the generation of defects or cracks in the crystal’s volume. The sources of stress are the emerging differences in the lattice constants between $c$-GaN and wing,\textsuperscript{27} which are the result of very large differences in the concentration of free carriers. According to the SIMS analysis there is a significant difference in oxygen concentration in the material grown in the $c$- and semi-polar directions.

It seems that there are a few ways to solve the problem of stress generation close to the edges of the growing crystal. One of them is the lattice constants engineering. The main dopant influencing the free carrier concentration and thus, lattice constants, is oxygen. In order to decrease this effect oxygen donors should be compensated. Such an approach requires finding the right acceptor, which will be built-in selectively in the laterally grown part of the crystal. Proper growth conditions should be determined in order to obtain such effect and reduce the stress coming from the edges of the crystal. For example, selective (only on semi-polar and non-polar planes) doping with Mg would compensate oxygen donors and decrease the value of “$a$” lattice parameter to the value of undoped GaN. This was observed for GaN crystals grown by high nitrogen pressure solution method.\textsuperscript{28–30} On the other hand, one can imagine a reverse process; the oxygen incorporation on the $c$-plane is the same like on the edges of the growing crystals. Then, the stress close to the edges will not be generated. The next solution is to skillfully control the temperature field in the crystallization zone. Only one facet of the crystal is grown ($c$-plane). This should completely inhibit lateral growth. Both presented ways are connected to non-trivial technological challenges. Implementation of these ideas would require to rebuilt the growth zone of the HVPE reactor and/or find proper dopants.
4. Summary
During crystallization of GaN in the c direction, semi-polar (10–11), (10–12), and (11–22) facets appear. In this work, crystallization on these naturally occurring semi-polar surfaces was described in detail. It was demonstrated that the semi-polar GaN substrates, obtained by slicing of multiregrown ammonothermal bulk GaN, could be used as seeds for the HVPE crystallization. The growth rate, morphology, and structural properties of obtained crystals depended on the crystallographic growth direction. It was presented that the electrical as well as optical properties of the crystals grown in naturally occurring semi-polar directions were significantly different from the properties of crystals grown the c-direction. Crystals grown in the semi-polar directions were highly n-type and the main source of free carrier concentration was oxygen. Moreover, it was presented that anisotropy of the growth is important factor limiting the HVPE-GaN growth in the c-direction. Ideas of how to overcome this challenge were briefly presented.

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