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Influence of Growth Temperature on Surface Morphologies of GaN Crystals Grown on Dot-Patterned Substrate by Hydride Vapor Phase Epitaxy

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Abstract. This paper studies the influence of growth temperatures in the range 825 to 1050°C on the surface morphologies of GaN crystals grown on a SiO_2 dot-patterned substrate using Epitaxy Lateral Overgrowth (ELO) and Hydride Vapor Phase Epitaxy (HVPE) techniques. A lower growth temperature of 850°C prompts the formation of GaN hexagonal pyramidal crystals with a higher fraction of $\{1\bar{1}01\}/(0001)$ facet areas than those grown at high temperatures (>1000°C). In a subsequent coalescent (or lateral growth) process, a high temperature of 1050°C is applied to the original GaN hexagonal pyramidal crystals, and the morphologies of the GaN layers are inspected. It is established that the original $\{1\bar{1}01\}$ faceted morphology of the hexagonal pyramids changes to an irregularly-shaped surface comprising $\{1\bar{1}01\}$, $\{11\bar{2}2\}$ and high index facets, and that the nature of the surface morphology is influenced by the growth time and the application (or not) of Ga precursor support. Hence, the results show that the coalescence and planarization of the GaN layer can be controlled through an appropriate specification of the process parameters. At low temperatures in the region of 850°C, high index facets are observed on the tops of a small percentage of the hexagonal GaN columnar crystals. It is proposed that this phenomenon is caused by a reduction in the surface diffusion length of the precursors, e.g. NH_3 and GaCl, at lower temperature, which in turn, reduces the probability of desorption and increases the lifetime.

Keywords: GaN, HVPE, ELO, hexagonal column

1. Introduction

The 3.4 eV wide-band gap of the GaN semiconducting compound renders it suitable for the fabrication of a variety of devices, including highly efficient blue light emitting diodes [1], laser diodes [2], and high power devices [3]. However, the large number of lattice mismatches which occur between the GaN layer and its sapphire substrate degrade the performance of these devices. Accordingly, many researchers have attempted to synthesize low-defect GaN wafers. It has been shown that the use of Epitaxy Lateral Overgrowth (ELO) and Metalorganic Vapor Phase Epitaxy (MOVPE) techniques [4, 5] to grow GaN layers on hetero-substrates such as sapphire or silicon carbide in a two-step process (i.e. an initial low temperature buffer layer followed by a high temperature growth layer) reduces the density of the GaN threading dislocations and increases the epitaxial quality. Several approaches have been presented for the preparation of bulk GaN crystals. However, these approaches, which include solution growth under an ultra-high nitrogen pressure [6] and the sublimation method [7], tend to produce GaN single crystals which are of high quality, but of too small a size to be of any practical use.

Some researchers have successfully demonstrated the use of the Hydride Vapor Phase Epitaxy (HVPE) process to grow high-quality thick GaN layers on hetero-substrates such as sapphire [8, 9] and GaAs [10].

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The low cost and high-growth rate of this process render it a particularly attractive method.

In the MOVPE process, the surface morphology of the grown GaN layer depends significantly upon the choice of either N_2 or H_2 as the carrier gas. It has been reported that the use of an H_2 ambient enhances the desorption of the surface nitrogen atoms from the GaN layer, and hence yields a Ga-rich surface. Meanwhile, it has been shown that N-rich GaN layers can be grown at temperatures ranging from 850 to 1000°C when an N_2 ambient is specified [11]. Finally, it has been demonstrated that the epitaxial surface of a GaN layer grown in an H_2 carrier gas tends to be smooth, whereas the use of an N_2 ambient yields a rough GaN surface comprised of many hexagonal hillocks [12].

A review of the related literature [13–16] reveals that researchers have adopted pure N_2 and H_2 carrier gases to grow GaN layers on a SiO_2 strip- patterned mask/MOVPE-GaN/Sapphire using the Epitaxial Lateral Overgrowth (ELO) technique at high temperatures in excess of 1050°C. For example, V. Wagner et al. [13] presented a two-step growth process, in which the dislocation density was initially reduced in an N_2 and H_2 gas mixture to induce dislocation bending and forming $\{11\bar{2}2\}$ facets, and subsequently planarization (or coalescence) was performed in N_2 gas to the lateral growth of the GaN crystals on the strip-patterned mask. This two-step approach is effective in reducing the dislocation density and provides a good coalescence and planarization of the grown layer. K. Hiramatsu et al. [14] concluded that the surface morphologies of GaN facets grown on a strip-patterned mask are significantly influenced by the temperature and pressure parameters applied during the growth process. O. Parillaud et al. [15] indicated that the formation of GaN hexagonal pyramids and platelets in an N_2 ambient depends on the crystalline and amorphous seeder layers, respectively.

The purpose of the present study is to grow large GaN crystals with low threading dislocations. It has been reported that the formation of $\{11\bar{2}2\}$ facets prompts dislocation bending [13]. Since each GaN pyramid consists of six $\{1\bar{1}01\}$ facets, while each GaN trapezoid comprises two $\{11\bar{2}2\}$ facets, it is reasonable to assume that dislocation bending will be more evident in dot-patterned GaN pyramids than in strip-patterned GaN trapezoid crystals. This study investigates the influence of growth temperature on the morphologies of GaN crystals grown on a dot-patterned SiO_2 mask using an H_2 carrier gas in the range of 825 to 1050°C. Furthermore, the study attempts to identify suitable process parameters which yield an initial growth of uniform six-symmetrical hexagonal pyramids and then a good coalescence of these GaN crystals at a high temperature of 1050°C with no Ga precursor input. Finally, this study proposes a possible growth mechanism to explain the observed phenomenon of high index facets located on the tops of a small percentage of the GaN hexagonal columns at temperatures in the region of 850°C.

2. Experimental Procedure

Initially, a preliminary GaN template was formed by using a commercial MOVPE system to grow a 2 μm GaN layer on a c-plane sapphire substrate. The GaN template was then coated with a 300 nm thick SiO_2 layer deposited at 300°C using the Plasma Enhanced Chemical Vapor Deposition (PECVD) process. An annealing process was then performed at 800°C for 30 min to enhance the SiO_2 crystalline quality. Subsequently, standard photolithography and Reactive Ion Etching (RIE) processes were employed to pattern the mask with an array of dot openings aligned along the $[11\bar{2}0]$ direction. The individual openings were separated by a distance of 6 μm and had a diameter of 3 μm.

The GaN samples were grown on the dot-patterned substrate using an HVPE system developed by the current authors. A schematic illustration of the current HVPE experimental set-up is presented in Fig. 1. The system comprised a vertical quartz reactor at atmospheric pressure, and used Ga, HCl, and NH_3 as sources and H_2 as the carrier gas. During the HVPE process, the mixed NH_3/H_2 gas was flowed into the outer tube, and a mixed HCl/H_2 gas was flowed into the inner tube. The GaCl precursor gas was produced at 850°C in accordance with the following reaction [17]: $Ga_{liq} + HCl_g \rightarrow GaCl_g + 1/2H_{2g}$. The GaN layer was subsequently formed by: $GaCl_g + NH_{3g} \rightarrow GaN_s + HCl_g + H_{2g}$ and was deposited on the SiO_2 dot-patterned/ MOVPE-GaN/ sapphire substrate. To establish the influence of the growth temperature on the surface morphology of the GaN crystals, GaN layer samples were grown using different substrate temperatures in the range of 825 to 1050°C. The growth times ranged from 30∼120 min and the V/III ratio was specified to be 100. The NH_3, HCl, and H_2 flow rates were maintained at 1500, 15, and 6500 sccm, respectively. It was observed that the GaN patterns in the central ∼2 cm^2 region of the 2″ wafers were uniformly distributed, and hence GaN crystals from this particular region were chosen for subsequent morphology analysis purposes.

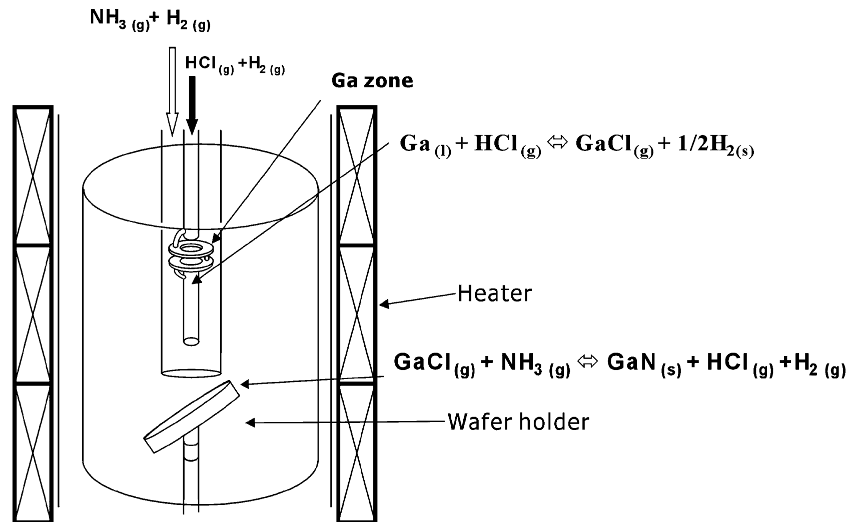

Fig. 1. Schematic diagram of vertical HVPE growth system.

In investigating the high temperature coalescence process, hexagonal GaN pyramids with six-symmetrical $\{1\bar{1}01\}$ facets were initially grown at a temperature of 850°C and then grown at 1050°C with an open Ga precursor support under the protection of NH_3 gas. Meanwhile, a second series of GaN crystals were prepared with no Ga precursor.

3. Results and Discussions

3.1. *Surface Morphology at Different Growth Temperatures*

Figure 2 presents SEM photographs of the GaN crystals grown on the current dot-patterned SiO_2 substrate at growth temperatures of 825, 850, 900, 950 and 1050°C. At the highest growth temperature of 1050°C (Fig. 2(a)), it is observed that the GaN crystals have only grown out of a small percentage $(15/(16 \times 13) = 0.072)$ of the mask openings. The magnified SEM image insert in Fig. 2(a) shows the presence of many very small isolated GaN islands within an individual mask opening. Since GaN exhibits a greater dissociation rate at higher temperatures, the NH_3 and GaCl molecules have a greater surface diffusion distance, and are hence less likely to coalesce into single large GaN crystals. The present results suggest that a lower temperature increases the possibility of the initial coalescence of GaN islands, and consequently the crystals will grow out of a greater number of dot openings, as shown in Fig. 2(c)–(e). Suppose every opening area obtains equal NH_3 and GaCl molecules per unit time, most of these molecules cross to adjacent openings on the SiO_2 mask layer while few of these remain within their original openings. Since very few of the openings have out-growing GaN crystals at a temperature of 1050°C, only a very small number of nuclei are available for subsequent lateral growth.

At the lower temperature of 950°C, Fig. 2(b) shows that virtually all of the openings $(26/(6 \times 5) = 0.87)$ are filled with GaN crystals. This observation suggests that the surface diffusion distance of the NH_3 and GaCl molecules is reduced at a lower growth temperature. Hence, some of these molecules cross to adjacent openings on the SiO_2 mask layer while others remain within their original openings. Consequently, the probability of GaN nucleation is significantly enhanced at a growth temperature of 950°C. This observation concurs with the conclusions of a previous study [18], in which it was reported that the density of the GaN islands is inversely related to the growth temperature and that large GaN islands are therefore associated with higher growth temperatures.

A comparison of Fig. 2(a) and (b) reveals that the GaN crystals grown at 1050°C are significantly larger than those grown at 950°C. The ratio of the volume of the larger crystals (1050°C) to that of the smaller crystals (950°C) is found to be in the order of $50^3/7^3$, i.e. 360:1. A possible explanation for the

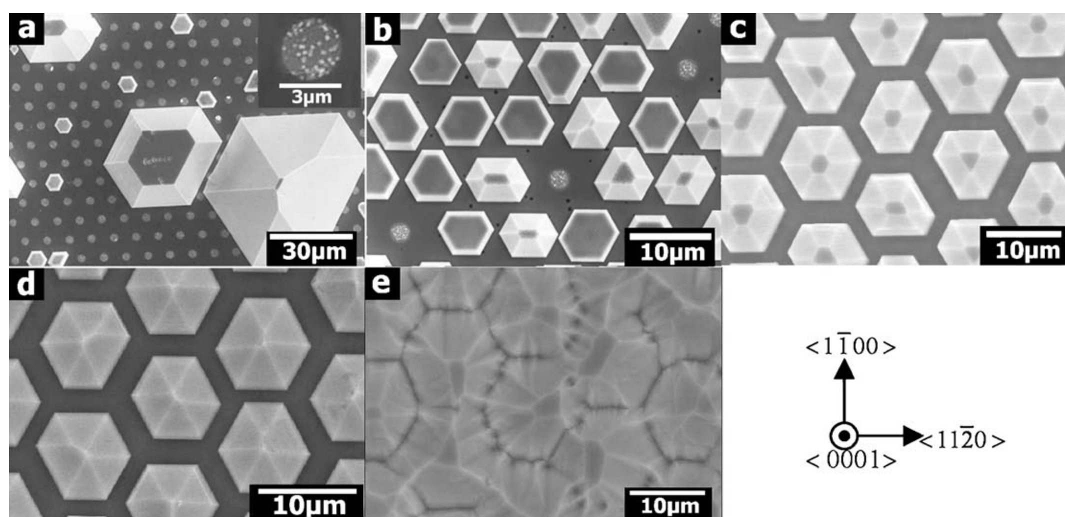

Fig. 2. SEM photographs of GaN crystals grown in H_2 carrier gas at different growth temperatures: (a) 1050°C, (b) 950°C, (c) 900°C, (d) 850°C, and (e) 825°C. Note that the insert in Fig. 1(a) provides a magnified view of the unfilled opening and shows the presence of many small randomly distributed GaN islands.

growth mechanism which accounts for this volumetric difference is that if the surface diffusion rate is taken to be the rate limiting step, almost all of the absorbed Ga atoms will diffuse laterally to existing GaN nuclei to react with the N atoms. Since it has been shown that very few GaN nuclei are formed at 1050°C, these nuclei subsequently grow into large GaN crystals. Conversely, at 950°C, where many GaN nuclei are present, the same amount of GaN material is deposited over a greater number of GaN nuclei. Consequently, the resulting GaN crystals will tend to be smaller and to be of a more uniform size.

Figure 2(a) and (b) show that at growth temperatures of 1050 and 950°C, some of the hexagonal GaN pyramids have top terraces of varying size in the form of a (0001) facet. It appears that this top terrace phenomenon is temperature dependent. In Fig. 2(c), which corresponds to a growth temperature of 900°C, the size of the (0001) facet is significantly reduced, while at a temperature of 850°C (Fig. 2(d)), the top terrace feature has virtually disappeared. It has been reported that the (0001) facet has the lowest dangling bond (DB) density of 11.4, followed by the {1$\bar{1}$00} facet (DB = 12), and finally the {1$\bar{1}$01} facet (DB = 16) [14]. A facet with a lower dangling bond density is known to possess a lower surface energy. Based on thermodynamics principles, a GaN crystal in the form of a hexagonal column with (0001) and {1$\bar{1}$00} facets should be expected. In the MOCVD process, the desorption rates of N and Ga decrease as the temperatures reduce [19]. Furthermore, the Ga desorption rate is lower than that of N at temperatures exceeding ∼800°C. In other words, kinetic data suggest that a Ga-rich (0001) facet will be favored at higher temperatures. This explains the current phenomenon, in which (0001) top facets are observed on the GaN crystals grown at temperatures in excess of 1000°C. It has been proposed that the threading dislocations tend to bend through 90°C from the [0001] direction to the [1$\bar{1}$00] direction as the GaN crystals grow out of the SiO_2 opening [13]. In other words, the [1$\bar{1}$01] facet will demonstrate a greater number of surface defects (e.g. dislocations, steps, and ledges) than the (0001) facet. Therefore, the growth rate along the (0001) facet will gradually increase and surpass the growth rate along the {1$\bar{1}$01} facet as the temperature decreases. Consequently, the (0001) facets gradually disappear as the growth temperatures reduce.

The SEM images in Fig. 2 confirm that the samples grown at a low temperature (850°C) exhibit a higher number (∼100%) of crystals growing out of the dot openings than the samples grown at 1050°C (∼7%). Figure 2 also shows that the GaN samples grown at 850°C exhibit a higher fraction of {1$\bar{1}$01}/(0001) facet areas than those grown at high temperatures (>1000°C). Therefore, it can be surmised that more dislocation bending features will be evident in samples grown at lower temperatures.

Surprisingly, Fig. 2(e) reveals that the samples grown at a temperature of 825°C have a rough surface with high index facets. Furthermore, it is noted that the individual GaN crystals are merged together. As discussed previously, in the MOCVD process, the Ga desorption rate is more rapid than that of N at temperatures below ~800°C [19]. Since Ga melts at 29°C and boils at 2200°C, which Ga atoms tend to form Ga liquid and flow down into the gaps between the individual GaN pyramids. Multi-faceted GaN, which is similar to that formed under 3D diffusion controlled growth, may be formed as the liquid Ga flows down into the gaps between the GaN pyramids.

3.2. *Lateral Growth and Coalescence of GaN Crystals*

As described previously in this paper, GaN pyramids with six-symmetrical $\{1\bar{1}01\}$ facets are formed at a growth temperature of 850°C (Fig. 2(d)). Therefore, this study first grows GaN pyramids under these temperature conditions, and then observes the changes in surface morphology and coalescent characteristics which occur as these GaN pyramids are subsequently grown at a higher temperature of 1050°C with no Ga precursor support under the protection of NH_3 and N_2 gas, and with Ga precursor support, respectively.

Figure 3 presents SEM photographs of samples originally grown at 850°C for 30 min, and then grown with Ga precursor support at 1050°C for 30 min (Fig. 3(a)) and at 1050°C for 60 min (Fig. 3(b)). For comparison purposes, Figure 3 also provides SEM images of samples originally grown at 850°C for 30 min, and then grown at a holding temperature of 1050°C for 30 min (Fig. 3(c)) and at 1050°C for 60 min (Fig. 3(d)) under the protection of NH_3 gas and with no Ga precursor support in both cases. Compared with the GaN pyramids grown at 850°C (Fig. 2(d)), following the second growth process at 1050°C for 30 min (Fig. 3(a)), it can be seen that the sample contains $\{11\bar{2}2\}$ facets in addition to the original $\{1\bar{1}01\}$ facets, while the sample grown at a holding temperature of 1050°C for 30 min (Fig. 3(c)) exhibits irregular $\{1\bar{1}01\}$, $\{11\bar{2}2\}$ and top-center pyramids of high index planes caused by surface atom diffusion effects.

Figure 3(b) indicates that the original GaN pyramids with six-symmetrical $\{1\bar{1}01\}$ facets merge with one another when grown further at 1050°C for 60 min with Ga precursor support. However, it can be seen that in a holding process with a temperature of 1050°C for 60 min and no precursor support (Fig. 3(d)), the grown crystals exhibit irregularly-layered (0001) surfaces as a result of stable (0001) surfaces formed by surface atom diffusion.

Since in the grown crystals shown in Fig. 3(d), there is no Ga precursor supply, it is proposed that the mechanism responsible for the change in the facet morphology is as follows: (1) the GaN is decomposed to Ga and N at the tip of the original pyramid ($GaN_s \rightarrow Ga_{liq} + 1/2N_{2g}$), (2) the Ga_{liq} flows downward toward the bottom of the pyramid through a process of surface diffusion, and (3) the GaN re-grows with a different facet plane ($Ga_{liq} + NH_{3g} \rightarrow GaN_s + 3/2H_{2g}$). Accordingly, it has been suggested that the GaN pyramids tend to merge gradually to form a smooth (0001) surface [20]. However, the high evaporation pressure of the GaN material at elevated temperatures requires particular consideration. The loss of nitrogen atoms is unavoidable as Ga_{liq} and N_{2g} are decomposed from the surface of the GaN layers. However, it is acknowledged that a suitable mixed gas ratio of NH_3/H_2 or NH_3/N_2 to restrain this GaN decomposition has yet to be established [21].

3.3. *Growth Mechanism of GaN Hexagonal Columns*

At a growth temperature of approximately 850°C, the majority of the GaN crystals are in the form of conventional hexagonal pyramids with six-symmetrical $\{1\bar{1}01\}$ facets. However, as shown in Fig. 4, some of the GaN crystals form a special type of hexagonal column in which high index facets, i.e.$\{1\bar{1}0X\}$, $(X > 1)$, are evident on the top of the column. The growth rate along the [0001] direction at 850°C is estimated to be 0.56 μm/min for the hexagonal columns and 0.27 μm/min for the conventional hexagonal pyramids. It is noted that the former type of hexagonal column occurs most readily at a growth temperature of approximately 850°C. Although this column structure can also be identified at a slightly lower temperature of 840°C (Fig. 5(b) and (e)), it takes longer to develop. Meanwhile, at the lowest temperature of 825°C (Fig. 5(a) and (d)), or at the highest temperature of 900°C (Fig. 5(c) and (f)), this particular morphology is not readily identified. Hence, it is proposed that the hexagonal column with high index facets located on its top is an intermediate meta-stable structure.

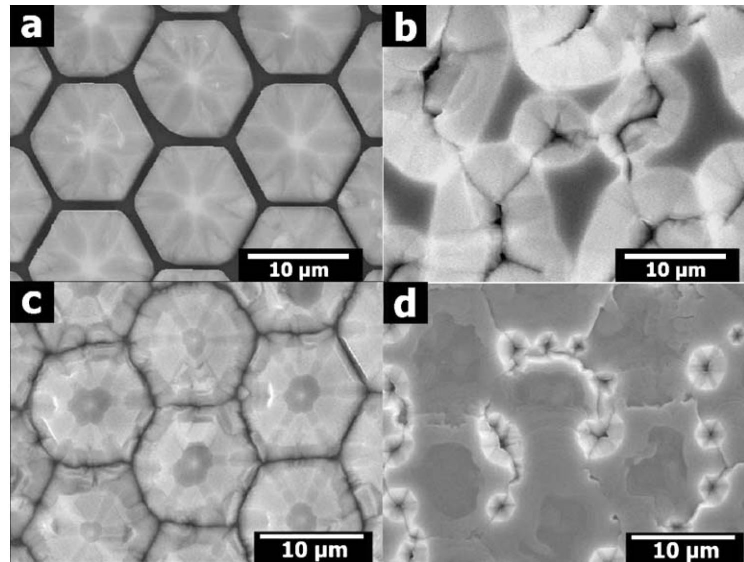

Fig. 3. SEM photographs of GaN crystals grown initially at 850°C for 30 min and then grown further under conditions of: (a) 1050°C for 30 min and (b)1050°C for 60 min, (c) a coalescent process at 1050°C for 30 min, and (d) a coalescent process at 1050°C for 60 min.

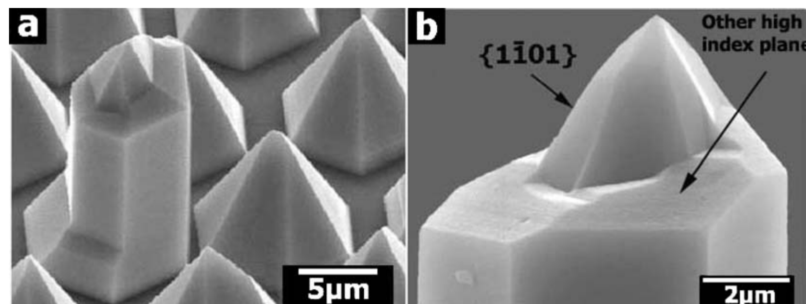

Fig. 4. SEM photographs of hexagonal columns with high index facets on column top grown at 850°C: (a) bird's eye view of column, and (b) magnified view of column top.

Figure 6(a) presents the SEM photograph of a typical hexagonal column. Figure 6(b) presents a proposed schematic illustration of the hexagonal column growth related to Fig. 6(a). A possible growth mechanism for the hexagonal column with high index facets on its top can be described as follows: initially, conventional GaN hexagonal pyramids are grown from the SiO_2 openings. As the height of the GaN pyramid increases, the majority of the threading dislocations bend 90° toward the $\{1\bar{1}01\}$ surface and GaN lateral overgrowth takes place (Fig. 6(b)). However, a small fraction of the threading dislocations in the inner central region of the GaN pyramid continue to grow along the [0001] direction. The growth rate along the [0001] direction is controlled by classical spiral growth with screw dislocations at the top of the GaN columns. If the Burgers vectors of the dislocations along the $[1\bar{1}00]$direction accumulate at the free surface, a step in the form of a $\{1\bar{1}00\}$ facet will be formed. This facet tends to increase in size since it has a relatively low dangling bond density compared to that of the $\{1\bar{1}01\}$ facet. Kinetically, the size of the $\{1\bar{1}00\}$ step must exceed a critical value, which acts as a nucleation barrier, before a stable $\{1\bar{1}00\}$ facet can be formed. Therefore, only a small percentage of the GaN crystals will form hexagonal columns. It has been reported [22] that if the mean diffusion distance of the adsorbed molecules is greater than the diameter of the column, but less than the column height, the column

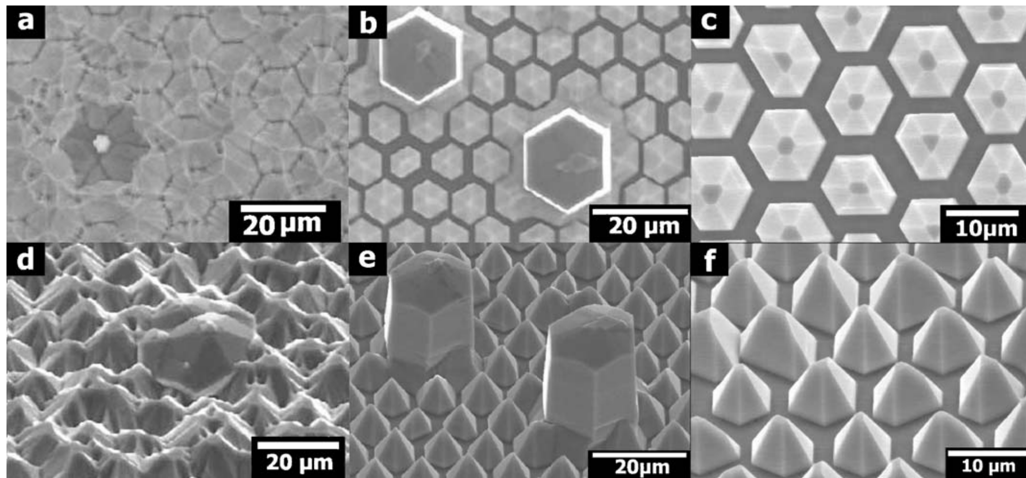

Fig. 5. SEM photographs of GaN crystals grown at different temperatures of: (a) 825°C, (b) 840°C, and (c) 900°C. Note that Fig. 5(d), (e), and (f) provide bird's eye views of Fig. 5(a), (b), and (c), respectively.

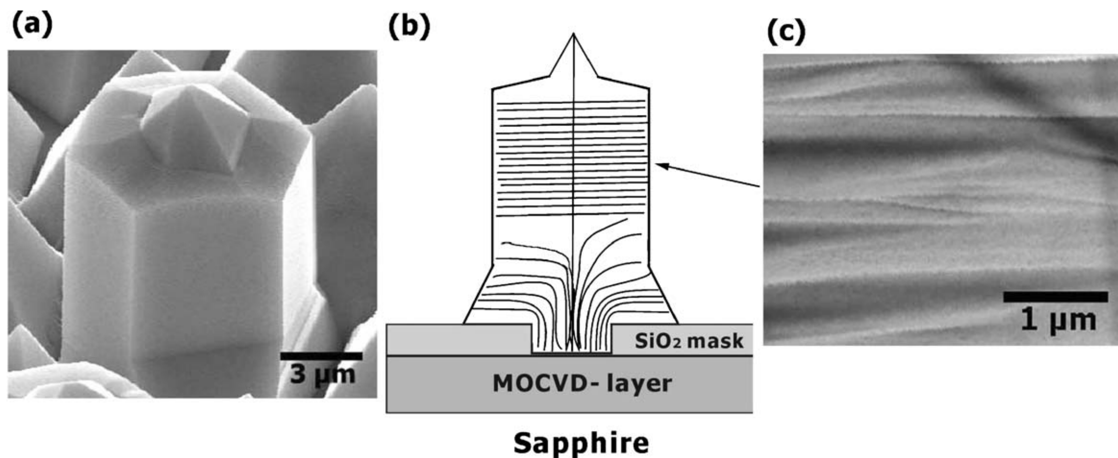

Fig. 6. (a) The SEM photograph of a GaN hexagonal column grown at 850°C (b) a proposed schematic diagram of hexagonal column grown from SiO_2 opening related to (a), and (c) TEM micrograph of lateral defect structure observed in GaN column with high index facets on top.

will grow faster in height than in diameter. Hence, the crystal morphology will shift gradually toward a column with a large height/diameter ratio.

It is proposed that the reduction in the surface diffusion length of precursors such as NH_3 and GaCl at lower temperatures reduce the probability of desorption and increases the lifetime. The condensation of Ga liquid droplets on the GaN surface changes the relative stability of the $\{1\bar{1}01\}$ facet. The TEM micrograph presented in Fig. 6(c) reveals the presence of un-identified lateral defects parallel to the (0001) plane. These lateral microstructures are suspected to be stacking faults and correspond to the horizontal lines shown in the schematic diagram of Fig. 6(b). Therefore, the formation of high index planes such as the $\{11\bar{2}2\}$ facet on top of the hexagonal column is related to the formation of lateral defects parallel to the (0001) plane.

4. Conclusions

(1) The GaN nucleation rate at the SiO_2 openings increases as the temperature decreases. The Ga-rich (0001) facet is favored at high temperatures

exceeding 1000°C (Fig. 2(a)), while an N-rich $\{1\bar{1}01\}$ facet is evident at a growth temperature of approximately 900°C (Fig. 2(c)). Meanwhile, at a low temperature of approximately 850°C, high index facets are evident on the top of a small percentage of the hexagonal GaN columnar crystals (Fig. 4(a)).

(2) In the holding temperature process, the GaN pyramids tend to merge with one another through Ga surface diffusion effects under the protection of NH_3 gas. The surface morphology gradually changes from irregularly shaped $\{1\bar{1}01\}$and $\{11\bar{2}2\}$ facets and top-center pyramids of high index planes (Fig. 3(c)) to a stable and smoother (0001) surface (Fig. 3(d)) as the growth time is extended.

(3) It is proposed that the special form of hexagonal column with high index facets observed at a growth temperature of 850°C is an intermediate meta-stable structure. The condensation of Ga liquid droplets on the GaN surface changes the relative stability of the $\{1\bar{1}01\}$ facet, which may assist in the nucleation of the $\{1\bar{1}00\}$ step. Once the$\{1\bar{1}00\}$ step size exceeds the critical nucleation size, growth of the hexagonal column will proceed at a higher rate than that of the pyramid.

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