Selective area formation of GaN nanowires on GaN substrates by the use of amorphous Al$_x$O$_y$ nucleation layer

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Abstract
Examples are presented that application of amorphous Al$_x$O$_y$ nucleation layer is an efficient way of controlling spatial distribution of GaN nanowires grown by plasma-assisted molecular beam epitaxy. On GaN/sapphire substrates Al$_x$O$_y$ stripes induce formation of GaN nanowires while a compact GaN layer is formed outside the stripes. We show that the ratio of nanowire length $h$ to the thickness of the compact layer $d$ can be tailored by adjusting impinging gallium and nitrogen fluxes. Calculations of the $h/d$ aspect ratio were performed taking into account dependence of nanowire incubation time on the growth parameters. In agreement with calculations we found that the value of $h/d$ ratio can be increased by increasing the N/Ga flux ratio in the way that the N-limited growth regime determines nanowire axial growth rate while growth of compact layer remains Ga-limited. This ensures the highest value of the $h/d$ aspect ratio. Local modification of GaN growth kinetics caused by surface diffusion of Ga adatoms through the boundary separating the Al$_x$O$_y$ stripe and the GaN/sapphire substrate is discussed. We show that during the nanowire incubation period gallium is transported out of the Al$_x$O$_y$ stripe, which delays nanowire nucleation onset and leads to reduced length of GaN nanowires in the vicinity of the stripe edge. Simultaneously the growth on the GaN/sapphire substrate is locally enhanced, so the planar GaN layers adopts a typical edge shape of mesa structures grown by selective area growth. Ga diffusion length on a-Al$_x$O$_y$ surface of ~500 nm is inferred from our results.

Keywords: gallium nitride nanowires, selective area growth, PAMBE

(Some figures may appear in colour only in the online journal)

1. Introduction

Since the discovery of their growth by Yoshizawa et al [1] and by Sanchez-Garcia et al [2] gallium nitride nanowires (NWs) have become known to exhibit very interesting physical properties, which are expected to trigger many applications in a next generation of electronic and optoelectronics devices [3, 4]. The growth under conditions of high V–III ratio offers unique possibility of creation of two dimensional GaN nanostructures having crystallographic quality not achievable in comparable planar heterostructures [5, 6]. Catalyst-mediated vapor–liquid–solid (VLS) growth mechanism is often employed for formation of GaN NWs (see [7] and references therein) with Ni [8] or Au-Ni [9] catalyst seeds. It has been reported, however, that the cost of incorporation of Ni dopant is a higher defect density and weaker light emission [10]. Therefore, an intriguing aspect is that GaN NWs can be nucleated through self-assembly and grown catalyst-free, thus avoiding their contamination by the catalyst [1, 2, 11, 12]. A specifically attractive benefit offered by the self-assembled formation of GaN NWs in plasma-assisted molecular beam epitaxy (PAMBE) is the possibility of creating vertically well-
aligned and single-crystalline NWs even on amorphous layers such as SiO$_2$ [13–17], SiN$_x$ [18–25] and Al$_2$O$_3$ [26–32] as well as on different types of metallic films and foils [33–37].

However, for development of reliable and reproducible nanodevices the precise control of the positions of the NWs is important while self-assembled growth is a random process with a very limited control of the NW density and spatial distribution on the substrate. Selective area growth (SAG) is an efficient way to overcome that problem. In the most standard version of the technique GaN is grown on GaN/sapphire or GaN/Si templates masked with an array of nanoholes. Thin films of Ti [38–49], SiO$_2$ [41, 50–52] or SiN [53–56] are the most commonly used substrate masking materials. Under suitable growth conditions GaN crystallization starts inside the openings in the mask and then proceeds in a form of vertical NWs while GaN nucleation on the mask is prohibited. The nucleation steps and morphology evolution of the NWs are determined by the homoepitaxial growth and by the fact that NW diameter is fixed by the mask nanohole size. Importantly, dislocations present in the GaN buffer layer easily propagate to the NWs, although they are efficient filtered in the bottom region. As shown by Kishino et al [46] for GaN nanowires with diameter less than 200 nm, dislocation-free crystals can be obtained in the upper part of the NWs despite a high density of dislocations ($\sim 10^{11}$ cm$^{-2}$) in the GaN/Si substrate.

On the contrary diameter and shape evolution of self-assembled GaN NWs are fully determined by the growth conditions and the energy interplays between strain, surface, and edge contributions to the total crystal energy [57]. GaN NWs self-assembled by PAMBE on amorphous substrates exhibit superior crystal quality: they are dislocation-free and fully relaxed [4, 58, 59], which is due to a very weak epitaxial constraints from the substrate. The problem of intrinsically high randomness of positions of self-assembled NWs can be circumvented by using of patterned substrate on which a regular array of nucleation centers is created, as for example nitridized aluminum dots [60, 61]. In this way the high crystal quality obtained in self-organization can be effectively combined with the advantage of controlled NW positioning on the substrate.

There is an increasing interest in PAMBE growth of GaN NWs on amorphous Al$_2$O$_3$ (a-Al$_2$O$_3$) buffer layers. As we have reported the structural and optical properties of GaN NWs grown on a-Al$_2$O$_3$ buffers are comparable to those obtained on silicon [29]. There are also indications that a-Al$_2$O$_3$ buffer efficiently blocks diffusion of silicon from the substrate to GaN NWs [29]. This could facilitate the NW growth at high temperatures without incorporating any impurities [62], potentially leading to exceptional luminescence properties. Furthermore, a-Al$_2$O$_3$ layers can be produced by atomic layer deposition (ALD) at temperatures below 100 °C on various surfaces [63]. This paves a new way for growing high-quality GaN NWs on a large variety of bulk substrates, which can substantially widen the range of applications based on GaN NWs.

In this work we show that, in addition to the advantages mentioned above, amorphous Al$_2$O$_3$ nucleation layer can be efficiently used for selective area formation of GaN NWs by PAMBE on GaN/sapphire substrates with stripes of a-Al$_2$O$_3$ on top. We show that the a-Al$_2$O$_3$ stripes induce formation of NWs while a compact GaN layer is formed outside the stripes. Our calculations verified by dedicated growth experiments prove that the ratio of the NW length to the thickness of the neighboring GaN compact layer can be controlled by varying the N/Ga flux ratio during the growth. We also show that during the NW incubation period gallium diffused out of the a-Al$_2$O$_3$ stripe, which delayed nucleation onset and reduced length of GaN NWs in the vicinity of the stripe edge, simultaneously enhancing the compact layer growth on the GaN/sapphire substrate. The effect allowed us to estimate Ga diffusion length on a-Al$_2$O$_3$ surface.

### 2. Experiments

The samples used in this study were fabricated by plasma-assisted molecular beam epitaxy (PAMBE) using a radio frequency N$_2$ plasma source and a solid-source effusion Ga cell. GaN growth was performed on commercially available GaN/sapphire substrates. 12 μm wide stripes of a-Al$_2$O$_3$ with 500 μm wide spacing were formed on all substrates by standard photolithography and lift-off processes from 20 nm thick a-Al$_2$O$_3$ buffer layer deposited by ALD at the temperature of 85 °C [63]. In the growth chamber, the substrates were heated to the required growth temperature $T_g$ of 814 °C, after which the N source was ignited and the Ga and N shutters were opened simultaneously to start the growth. The growth duration was $t = 120$ min for all samples. Ga and N fluxes $\Phi_Ga$ and $\Phi_N$ used for growth were calibrated in GaN-equivalent growth rate units (nm min$^{-1}$) [64]. After growth surface morphology of the samples was examined by scanning electron microscopy (SEM). Table 1 contains the list of samples used in this study and presents values of Ga and N fluxes $\Phi_Ga$ and $\Phi_N$ used during the growths.

### 3. Results and discussion

Figure 1 shows bird’s (a) and plan (b), (c) view SEM micrographs of sample A grown with gallium and nitrogen fluxes of 4.5 nm min$^{-1}$ and 8.3 nm min$^{-1}$, respectively. As seen, well-organized GaN NWs were formed on a-Al$_2$O$_3$ stripes (figure 1(c)) whilea rough compact GaN layer was obtained on bare, crystalline parts of GaN/sapphire substrate (figure 1(b)). Surface morphology of the compact layer is typical for GaN grown by PAMBE under N-rich conditions at

| Sample | $\Phi_Ga$ (nm min$^{-1}$) | $\Phi_N$ (nm min$^{-1}$) |
|--------|------------------------|------------------------|
| A      | 4.5                    | 8.3                    |
| B      | 4.5                    | 11.5                   |
| C      | 4.5                    | 14.5                   |
| D      | 2.5                    | 11.5                   |
| E      | 2                      | 8.3                    |
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Figure 1. Bird’s (a) and plan (b), (c) view SEM micrographs of sample A grown on GaN/sapphire substrate with 12 μm wide stripes of a-Al₂O₃. (b) and (c) show zoomed plan view of GaN compact layer and GaN NWs, respectively. Scale bars correspond to 2 μm.

Figure 2. Contour plot of the value of the aspect ratio AR as a function of nitrogen Φ_N and gallium Φ_Ga fluxes calculated from equation (2) for the growth temperature of 814 °C and growth duration t of 120 min. Stars mark growth conditions for samples A–E while the labels give experimental values of the aspect ratio measured for these samples.

moderate substrate temperatures. This example clearly shows that the a-Al₂O₃ layer efficiently induces selective area formation of GaN NWs.

Although GaN crystallization on GaN substrates outside the a-Al₂O₃ stripes cannot be completely avoided the ratio of GaN compact layer thickness d and the length of GaN NWs h can be tailored by controlling the growth conditions. To discuss in detail we notice that under N-rich conditions the growth of GaN NWs may take place there is a number of reports showing that, despite of overall N-rich conditions, the growth of GaN NWs may take place outside the a-Al₂O₃ stripe

\[ \text{AR} = \frac{h}{d} = \frac{\Phi_N \times (t - t_{inc})}{\Phi_{Ga} \times t} \]  

(2)
can be calculated as a function of the growth parameters.

Figure 2 presents the value of the aspect ratio AR as a contour plot with a linear scale as a function of nitrogen and gallium fluxes calculated from equation (2) for the growth temperature of 814 °C and the growth duration of 120 min. The map serves as a convenient guide for controlling the AR value for various growth conditions. As seen, for fixed value of Ga flux Φ_Ga, the aspect ratio strongly increases with the increase of the nitrogen flux Φ_N. This is due to the faster N-limited axial growth of NWs as well as to much shorter NW incubation time, which means shorter delay of NW nucleation onset at higher N fluxes [31]. For fixed nitrogen flux larger values of the aspect ratio are expected for lower gallium fluxes, mainly because then the Ga-limited growth rate of the compact GaN layer becomes smaller.

In order to verify these theoretical predictions samples A–E were grown for 120 min under various Ga and N fluxes at the growth temperature of 814 °C. Figure 3 presents cross-section SEM micrographs of these samples. Position of stars in figure 2 correspond to the growth conditions (Φ_Ga and Φ_N) of the samples while the labels contain experimental values of the aspect ratio measured for these samples.

For sample A grown at gallium and nitrogen fluxes of 4.5 nm min⁻¹ and 8.3 nm min⁻¹, respectively, the value of the aspect ratio of 1.45 is found from figure 3(a). As seen in figure 2, the measured AR value agrees very well with results of calculations. Importantly, an increase on the nitrogen flux from 8.3 to 11.5 nm min⁻¹ for the same Φ_Ga in sample B leads to an increase of the aspect ratio as expected. Specifically, the NW growth rate increases indicating locally Ga-rich conditions, while the growth rate, and consequently the

\[ t_{inc} = C \times \Phi_N^{1.78} \times \Phi_{Ga}^{0.68} \times \exp(-4.79[\text{eV}] / k_B T_Gr) \]  

(1)

where C is a constant and \( k_B \) the Boltzmann constant. Using equation (1) the value of the aspect ratio AR that is the ratio of GaN NW length h to the thickness d of compact GaN layer

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thickenss, of the compact layer grown under N-rich conditions remains the same. The AR value equal to 2.1 for sample B (figure 3(b)) again agrees well with calculated data as shown in figure 2. Unfortunately, further increase of the nitrogen flux to 14.5 \text{ nm min}^{-1} (sample C) leads to a decrease of the aspect ratio value to 1.7 while for those growth conditions the calculated value of 3 can be inferred from figure 2. Similar disagreement is found when decreasing the impinging gallium flux at 4.5 to 2.5 \text{ nm min}^{-1} for \( \Phi_N = 11.5 \text{ nm min}^{-1} \) (sample D). Although the thickness of the compact layer is reduced accordingly to the reduced Ga flux (see figure 3(d)), the NWs are not as long as expected and the resulting value of the aspect ratio is only 2.2 instead of 3.8.

In order to explain this discrepancy we note that an important contribution to NW elongation is due to an efficient diffusion of the Ga adatoms on the NW sidewalls and gallium accumulation on the NW tip [66–69], eventually leading to locally Ga-rich conditions there. Consequently, the axial growth rate becomes N-limited and is not influenced by further increase of the impinging gallium flux [69, 70]. Therefore a decrease of \( \Phi_{Ga} \), as for sample D, should result in a reduction of the axial growth rate which turns to be Ga-limited. Such transition from N- to Ga-limited NW growth regimes with decreasing the impinging gallium flux was indeed observed experimentally in a single GaN NW via a marker technique [67, 69] or by using QMS for analysis of GaN NW growth kinetics [70]. The same effect of transition from N- to Ga-limited NW growth is responsible for the aspect ratio reduction in sample C. This time, however, the nitrogen flux is increased so much that despite Ga diffusion to the NW tip gallium concentration there cannot reach that of nitrogen, so again the axial growth rate becomes Ga-limited. Consequently, for these two samples (C and D) their actual growth conditions are not well described by equation (2) that assumes N-limited growth regime, thus predicting longer NWs and higher value of the aspect ratio than observed experimentally. To underline this disagreement positions of those two samples on the map in figure 2 are distinguished in red.

To further confirm the above explanation sample E was grown at the same temperature of 814 °C for low gallium flux \( \Phi_{Ga} = 2 \text{ nm min}^{-1} \) and \( \Phi_N = 8.3 \text{ nm min}^{-1} \). Cross-section SEM micrograph of the sample is shown in figure 3(e). The NW incubation time for that growth conditions is 43 min. Thus the effective growth time of 630 nm long NWs was 77 min, which corresponds to the NW axial growth rate of 8.2 \text{ nm min}^{-1} being close to the nitrogen flux and indicating N-limited NW growth conditions. On the other hand, the growth rate of the compact layer in sample E, as determined from the layer thickness measurement in figure 3(e), equals to 1.95 \text{ nm min}^{-1}, i.e. very close to the \( \Phi_{Ga} \) value. This example clearly shows that, in agreement with our explanations, by decreasing the gallium flux at fixed \( \Phi_N \) the N-limited growth conditions can be recovered. The measured value of the aspect ratio in sample E is 2.7 that agrees well with the results of calculations shown in figure 2.

It is noteworthy that the thickness of the compact layer increases, while the length of NWs decreases close to the edge of the a-Al\(_2\)O\(_3\) stripe (figure 3(e)). Enhanced growth at the edge of mesa structure grown by SAG has been observed already [41, 71, 72] and has been explained by diffusion of adatoms out of the mask material. In our case this corresponds to diffusion of Ga from the a-Al\(_2\)O\(_3\) stripe where GaN nucleation is delayed relatively to that on the bare GaN surface. Since the GaN layer grows in the Ga-limited regime this process locally enhances planar GaN growth close to the stripe edge. On the other hand, this surface diffusive flux produces a zone with reduced surface Ga concentration on the a-Al\(_2\)O\(_3\) area in the vicinity of the stripe edge. Consequently, as predicted by equation (1), delay of NW nucleation onset increases in that zone and NWs grow shorter than in the middle of the stripe as seen in figure 3(e). Practical implication of these edge effects is that the thickness \( d \) of the compact GaN layer as well as the length of NWs \( h \) should be measured far away from the stripe edge to get the correct
value of the aspect ratio. The AR values for samples A–E in this work have been determined just in that way. On the other hand, it is expected that for the stripes narrower than double diffusion length $\lambda$ of Ga on a-Al$_2$O$_3$ surface GaN NWs should have nearly uniform length across the stripe. As seen from figure 3 NW length reduction takes place for $\sim$500 nm from the edge of the stripe, so this should correspond to the value of $\lambda$. This value is similar to 400–500 nm reported for Ga diffusion on SiN$_x$ surface [73, 74]. Consequently, the stripes much wider than 1 $\mu$m are needed to see the impact of Ga surface diffusion on GaN NW nucleation, which explains dimensions of the stripes we have chosen in this work.

Finally, let us note that the edge effect is best visible for low Ga fluxes (sample E). While some decrease of NW lengths at the stripe edge is noticeable for all samples, the excess planar growth is most evident for sample E. The reason is that for smaller Ga NW incubation time increases (see equation (1)). Thus there is more time for gallium adatom transfer out of the a-Al$_2$O$_3$ stripe. Moreover, for low $\Phi_{Ga}$ values planar GaN growth rate is lower and therefore relative contribution of excess surface Ga flux to the growth is more noticeable.

4. Conclusions

The application of a-Al$_2$O$_3$ nucleation layer deposited by ALD is an efficient way of controlling spatial distribution of self-assembled GaN NWs grown on GaN substrates by PAMBE. Amorphous Al$_2$O$_3$ stripes induce formation of NWs while a compact GaN layer is formed outside the stripes. Although growth of planar GaN layer on uncovered parts of GaN substrate cannot be avoided the ratio of NW length $h$ to the thickness of the compact layer $d$ can be tailored by adjusting impinging gallium and nitrogen fluxes. Calculations of the $h/d$ aspect ratio were performed taking into account dependence of NW incubation time on the growth parameters. The results were used as a guidance for controlling the AR value at various growth conditions. In agreement with calculations we found that the value of $h/d$ ratio can be increased by increasing the $\Phi_N/\Phi_{Ga}$ flux ratio in the way that the N-limited growth regime determines NW axial growth rate while growth of compact layer remains Ga-limited. This ensures the highest value of the $h/d$ aspect ratio. Local modification of GaN growth kinetics caused by surface diffusion of Ga adatoms through the boundary separating the a-Al$_2$O$_3$ stripe and the substrate is discussed. It is shown that during the NW incubation period gallium diffused out of the a-Al$_2$O$_3$ stripe leading to delayed nucleation onset and reduced length of GaN nanowires in the vicinity of the stripe edge. At the same time the growth on the GaN/sapphire substrate was locally enhanced, so the planar GaN layers adopted a typical edge shape of mesa structures grown by SAG. Ga diffusion length on a-Al$_2$O$_3$ surface of $\sim$500 nm is estimated from our results.

It is apparent that pure SAG of GaN NWs leading to growth of position controlled single nanowires can be obtained with the use of a-Al$_2$O$_3$ nucleation layer if the substrate is covered by a mask that prohibits GaN nucleation outside the seeds (e.g. SiN mask) and nucleation site size is significantly reduced, e.g. by using e-beam lithography for substrate patterning. However, this subject is outside the scope of this work and will be discussed elsewhere.

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