Reducing the grain density in semipolar (11-22) AlGaN surfaces on m-plane sapphire substrates

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The growth mechanisms during metalorganic vapor phase epitaxy (11-22) oriented Al$_{x}$Ga$_{1-x}$N with $x \approx 0.80$ on m-plane sapphire are studied with the intention of mitigating the expansion of misoriented grains, composed of the (1-10-3) crystal orientation and achieving a flat surface with only the (11-22) orientation. An increase in reactor pressure, metalorganic supply, and V/III ratio led to a decrease in the grain density from $1.0 \times 10^{7}$ cm$^{-2}$ to $1.5 \times 10^{6}$ cm$^{-2}$. By comparing different growth regimes, we found that the main factor suppressing the growth of the (1-10-3) orientation and decreasing the grain density in the AlGaN layers is the growth rate, which decreased with increasing reactor pressure, growth conditions with higher total flow and lower TMAl flow were chosen, yielding low growth rates of $0.13 \mu$m h$^{-1}$ and a grain density of $3.0 \times 10^{6}$ cm$^{-2}$ at an aluminum mole fraction of 84%. To allow the growth of thick LED heterostructures we demonstrated that such a buffer can be overgrown with higher growth rate AlGaN, yielding a low grain density of $1.0 \times 10^{6}$ cm$^{-2}$ and a smooth morphology with a rms roughness of 2.5 nm by avoiding misoriented crystal propagation during nucleation. © 2019 The Japan Society of Applied Physics

1. Introduction

Light emitting diodes (LEDs) in the very deep ultraviolet (UV) spectral range are interesting for environmental and industrial sensing applications. However, below 240 nm, the external quantum efficiency of UV LEDs drops below 0.15%. LEDs grown on c-plane sapphire substrates suffer from a change of the optical polarization from dominant transverse electric polarized emission to dominant transverse magnetic polarized emission around 240 nm, leading to a reduced light extraction efficiency. Investigating semipolar growth provides a new approach for deep UV emitters since it overcomes the poor light extraction efficiency. Additionally, semipolar MQWs do not suffer from internal polarization fields therefore thus having a high electron and hole wavefunction overlap and high radiative recombination rate. The (11-22) orientation is promising as it exhibits stability during growth, can be easily nucleated on (10-10) m-plane sapphire, and has been successful for InGaN devices.

AlGaN and AlN layers with different semipolar orientations have been reported on m-plane sapphire. For GaN, crystal orientations of (1-10-3), (11-22) and (10-10) were reported. The crystal orientation depends mainly on the nitridation and nucleation conditions. There has additionally been reports of using a (11-22) AlN template or GaN microrods on m-plane sapphire before AlGaN deposition. The surface morphology of (11-22) AlGaN grown on m-plane sapphire with high aluminum mole fraction is often adorned by grains, composed of the (1-10-3) crystal orientation and achieving a flat surface with only the (11-22) orientation. An increase in reactor pressure, metalorganic supply, and V/III ratio led to a decrease in the grain density from $1.0 \times 10^{7}$ cm$^{-2}$ to $1.5 \times 10^{6}$ cm$^{-2}$. By comparing different growth regimes, we found that the main factor suppressing the growth of the (1-10-3) orientation and decreasing the grain density in the AlGaN layers is the growth rate, which decreased with increasing reactor pressure, growth conditions with higher total flow and lower TMAl flow were chosen, yielding low growth rates of $0.13 \mu$m h$^{-1}$ and a grain density of $3.0 \times 10^{6}$ cm$^{-2}$ at an aluminum mole fraction of 84%. To allow the growth of thick LED heterostructures we demonstrated that such a buffer can be overgrown with higher growth rate AlGaN, yielding a low grain density of $1.0 \times 10^{6}$ cm$^{-2}$ and a smooth morphology with a rms roughness of 2.5 nm by avoiding misoriented crystal propagation during nucleation. © 2019 The Japan Society of Applied Physics

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GaN and AlN layers with different semipolar orientations have been reported on m-plane sapphire. For GaN, crystal orientations of (1-10-3), (11-22) and (10-10) were reported. The crystal orientation depends mainly on the nitridation and nucleation conditions. There has additionally been reports of using a (11-22) AlN template or GaN microrods on m-plane sapphire before AlGaN deposition. The surface morphology of (11-22) AlGaN grown on m-plane sapphire with high aluminum mole fraction is often adorned by grains, increasing the surface roughness. These grains were identified as crystallites of the (1-10-3) or (10-10) orientation, and have been observed by transmission electron microscopy. Furthermore, the grains prevent a smooth morphology, which is critical for the growth of quantum wells in a device.

In this manuscript, the growth mechanisms for metalorganic vapor phase epitaxy (MOVPE) (11-22) Al$_{x}$Ga$_{1-x}$N, at compositions of $x = 80\% \pm 5\%$, were studied with the intention of mitigating the formation of these grains. Growth parameters such as the reactor pressure, metalorganic (MO) supply, and V/III ratio are investigated and their effect on the grain density is studied.

2. Experiment

The AlGaN layers were grown in a close-coupled showerhead MOVPE reactor using the standard precursors trimethylaluminum (TMAI), trimethylgallium (TMGa), and ammonia (NH$_3$) on 2" m-plane (10-10) sapphire substrates. The sapphire substrates underwent 600 s of nitridation before growth at a temperature of 1120 °C, at reactor pressure of 200 hPa, and an NH$_3$ flow of 30 Pa. The growth conditions, i.e. reactor pressure, MO supply, and V/III ratio were varied systematically to identify which parameters influenced the surface morphology and grain density. The Al composition of the layers was held constant at 80% ± 5% Al by varying the growth temperature. The film thickness varied between 0.25-0.4 μm. The TMAI/ (TMGa + TMAI) ratio in the gas phase was held constant at 0.87. The carrier gas flow into the reactor was 8 standard liters per minute (slm) of hydrogen.

In the first sample series, the reactor pressure was varied from 200 hPa to 500 hPa, while holding the growth temperature, V/III ratio, and MO supply constant at 1080 °C ± 5 °C, 660, and 88 μmol min$^{-1}$ TMAI and 13 μmol min$^{-1}$ TMGa, respectively. The increase in the reactor pressure led to a subsequent increase in the partial pressure of the TMAI and TMGa, where $P_{TMAI} + P_{TMGa}$ increased from 5.70 Pa at a reactor pressure of 200 hPa to 14.20 Pa at a reactor pressure of 500 hPa.

In the second sample series, the partial pressure of TMAI and TMGa ($P_{TMAI} + P_{TMGa}$) was increased from 3.5 Pa to 19.0 Pa. The reactor pressure was held constant at 500 hPa. In order to hold the V/III constant at 660, the NH$_3$ partial pressure was varied from 2300 to 12 500 Pa. To maintain an Al content at 80% ± 5% Al, the temperature ranged from 1120 °C to 1110 °C.

Finally in the third sample series, the V/III ratio was varied from 280 to 1320. This was accomplished by increasing the NH$_3$...
flow from 2310 to 12 500 Pa, while holding the \( P_{\text{TMAl}} + P_{\text{TMGa}} \) constant at 14.2 Pa (\( \text{TMAl/ (TMGa + TMAl)} = 0.87 \)). The temperature and pressure were held constant at 1100 °C ± 10 °C and 500 hPa, respectively.

The surface morphology was investigated using an atomic force microscope (AFM) in contact mode. The root-mean squared (rms) roughness, grain density, grain height, and grain width were calculated from scans from three different areas on each sample (average as well as standard deviation). The aluminum composition in the AlGaN layers was determined using transmission spectroscopy at room temperature. High-resolution X-ray diffraction was utilized to verify the (11-22) growth orientation in the AlGaN layers with an omega-two-theta scan using an open detector in double axis configuration with a four-bounce monochromator in the incidence beam. The growth rate and thickness of the AlGaN layers was determined using in situ spectroscopic reflectance using a Laytec EpiCurve TT system.

3. Results and discussion

3.1. Series 1: variation of growth pressure

In the first sample series the reactor pressure was investigated, as Ref. 11 showed that it had an effect on the grain density in AlN. Figure 1 displays three representative 10 \( \mu \)m \( \times \) 10 \( \mu \)m AFM images of the AlGaN surface at a reactor pressure of 200, 400, and 500 hPa. As the reactor pressure is increased the grain density is reduced, accompanied with a smoother morphological surface with an rms roughness of 13.5, 2.7, and 1.6 nm, respectively. The semipolar AlGaN morphology contains triangular features with grains (dot like structures) as described by Ref. 25 for an aluminum content of 70% and higher. The amount of grains in each image is clearly reduced with increasing reactor pressure. Figure 2(a) displays the grain density and growth rate for all AlGaN layers grown in sample series 1. The reactor pressure leads to a reduction in the grain density on the AlGaN surface from \( 5.2 \times 10^8 \) cm\(^{-2} \) at 200 hPa to \( 3.9 \times 10^7 \) cm\(^{-2} \) at 500 hPa. In comparison, the grain density from Ref. 28 was calculated to be \( 8.6 \times 10^7 \) cm\(^{-2} \) (determined independently from AFM images published in Ref. 28). Figure 2(b) displays the grain height and grain width as a function of reactor pressure. Both parameters decrease from 50 nm and 275 nm at 200 hPa to 21 nm and 184 nm at 500 hPa, respectively. The growth rate of AlGaN [Fig. 2(a)] also steadily decreases with increasing reactor pressure from 1.00 \( \mu \)m h\(^{-1} \) at 200 hPa to 0.40 \( \mu \)m h\(^{-1} \) at 500 hPa. Increasing the pressure in the reactor increases the partial pressure of the precursors but also reduces the gas flow velocity, thus increasing the likelihood of TMAl and NH\(_3\) interacting with each other in the gas phase before reaching the substrate and forming particles which will not be incorporated. However, this reduction in growth rate was also accompanied by a reduced grain density, height, and width, indicating that this method also suppressed the propagation.
and formation of the (1-10-3) orientation, as the (11-22) orientation had a faster growth rate and overgrew the (1-10-3) orientation.

Figure 3 displays the omega-two-theta scan of the AlGaN layer grown at 200 hPa. The sapphire (30-30) peak is centered at 34.35°, the AlGaN (11-22) peak is to the right at 35.65°. (1-10-3) AlGaN would appear on the left hand side of the (30-30) sapphire peak. However, as shown by Ref. 26 the intensity of the (1-10-3) peak reduces with increasing aluminum mole fraction in spite of the occurrence of grains. Therefore, in Fig. 3 the (1-10-3) AlGaN is only slightly visible and a correlation of XRD peak intensity and grain density is not suitable. All ω/2θ scans for AlGaN, regardless of growth conditions in this manuscript, look similar to Fig. 3.

3.2. Series 2: variation of MO-supply
The influence of the MO supply is investigated by varying the sum of TMGa and TMAI from $P_{\text{TMGa}} + P_{\text{TMAI}} = 3.5$ to $P_{\text{TMGa}} + P_{\text{TMAI}} = 19.0$ Pa at a constant TMAI/ (TMGa + TMAI) ratio of 0.87 and using a constant pressure of 500 hPa. Figure 4 displays three 10 μm × 10 μm AFM images of the AlGaN surface at $P_{\text{TMGa}} + P_{\text{TMAI}}$ of 3.5, 14.2, and 19.0 Pa, respectively. As the MO supply is increased, the reduction in grains is clearly visible leading to a smoother morphological surface with a rms roughness of 13.0, 5.6, and 3.0 nm, respectively. Figure 5(a) displays the grain density and growth rate for all AlGaN layers grown in the MO supply investigation. The grain density decreased with increasing MO supply by more than one order of magnitude from $9.3 \times 10^8$ cm$^{-2}$ at 3.5 Pa to $3.5 \times 10^7$ cm$^{-2}$ at 19.0 Pa. Figure 5(b) displays the grain height and grain width as a function of MO supply. Both parameters decrease from 64 nm and 305 nm at 3.5 Pa to 33 nm and 200 nm at 19.0 Pa, respectively. The growth rate of AlGaN initially increased, but after 12.0 Pa the growth rate decreased to a minimum value of 0.44 μm h$^{-1}$ at 19.0 Pa.
Without pre-reactions a constant increase in growth rate with MO supply would be expected. However, due to pre-reactions, TMAl and TMGa is consumed before reaching the surface. Increasing the MO supply from 3.5 to 12.0 Pa (and along the NH3 supply) increased the growth rate, however not by a proportional amount. After 12.0 Pa, the growth rate decreased due to the higher MO supply as well as the increased NH3 supply and the already increased pressure (500 hPa). However, this was accompanied by reduced grain density, height, and width, indicating that this method also suppressed the formation and propagation of the (1-10-3) orientation.

3.3. Series 3: variation of V/III ratio

Increasing the pressure and MO supply led to grain density reduction due to a suppression of the (1-10-3) orientation. In order to investigate the influence of the V/III ratio on the formation of grains, the V/III ratio was varied between 280

![Fig. 6.](image)

Fig. 6. (Color online) 10 μm × 10 μm AFM images of the AlGaN surface at a V/III ratio of 280, 860, and 1320, with an rms roughness of 13.2, 2.9, and 2.5 nm, respectively.

![Fig. 7.](image)

Fig. 7. (Color online) (a). Grain density and growth rate of AlGaN as a function of V/III ratio. (b). Grain height and width as a function of V/III ratio.

![Fig. 8.](image)

Fig. 8. (Color online) Overview of grain density as a function of growth rate for all studies, demonstrating a decreasing trend.

![Fig. 9.](image)

Fig. 9. Schematic showing (a) competition for nucleation and growth of (11-22) and (1-10-3) AlGaN orientation on m-plane sapphire, (b) growth and coalescence during high grain density conditions and (c) growth and coalescence during low grain density conditions.
and 1320 by varying the NH3 supply and keeping the MO supply and reactor pressure constant at 14.2 Pa and 500 hPa, respectively. Figure 6 displays three 10 μm × 10 μm AFM images of the AlGaN surface at a V/III ratio of 280, 860, and 1320. Increasing the V/III ratio led to a lower grain density and a smoother surface with an rms roughness of 13.2, 2.9, and 2.5 nm, respectively. Figure 7(a) plots the grain density and growth rate of AlGaN as a function of V/III ratio. Increasing the V/III ratio leads to a reduction in the grain density on the AlGaN surface from 3.0 × 10^8 cm^{-2} at V/III = 280 to 1.6 × 10^7 cm^{-2} at V/III = 1320. Figure 7(b) displays the grain height and grain width as a function of V/III ratio. Grain height and width decreased from 60 nm and 350 nm at V/III = 283 to 39 nm and 305 nm at V/III = 1320, respectively. The growth rate [Fig. 7(a)] decreases from 0.58 μm h^{-1} at V/III = 280 to 0.25 μm h^{-1} at a V/III = 1320. Increasing the V/III ratio via NH3 uptake increases the likelihood of ammonia and TMAl/TMGa to pre-react. The subsequential decrease in the (11-22) AlGaN growth rate, however, is accompanied by a reduction in grain density, height, and width, indicating that this method also allowed for the (11-22) orientation to overgrow and suppress the propagation of the (1-10-3) orientation.

### 4. Discussion of the growth model

The three sample series all showed a strong influence of reactor pressure, MO-supply, and V/III ratio on the appearance of (1-10-3) oriented grains and the lowest grain density was always observed for the lowest growth rate. Figure 8 sums up the grain density of all three series investigated over the growth rate. Obviously, a decrease in growth rate is associated with a decrease in the grain density.

To understand the effect of the growth rate on the grain density we propose the following model as depicted in Fig. 9. The grain density depends on the density of (1-10-3) oriented crystallites during nucleation of the layer and their evolution during the overgrowth. Initially the (11-22) and (1-10-3) oriented crystallites nucleate (or are formed during nitridation) on the surface of m-plane sapphire as shown in Fig. 9(a) thereby setting the upper limit of the grain density. The nucleation depends on the nitridation and nucleation layer conditions.21,22) During the overgrowth, the size (and surface area) of the crystallites change. In the worst case scenario, the undesirable crystallites expand leading to polycrystalline material as shown in Ref. 27. In this paper—even under high grain density growth conditions—the (11-22) crystallites expand laterally and vertically such that they fully coalesce, as can be seen by the underlying undulations in AFM throughout the study. However the (1-10-3) oriented grains also expand vertically and protrude out of the sample, adorning the surface and increasing the surface roughness, as shown in Fig. 9(b). Under low grain density conditions [Fig. 9(c)], the growth of the (1-10-3) orientation is suppressed, such that the (11-22) orientated material coalesces and overgrows the (1-10-3) oriented grains, shown via AFM by a reduced grain density and lower rms roughness. The growth of the (1-10-3) oriented grains can only be suppressed when the relative growth rate of the (11-22) is higher than that of the grains, i.e., when adatoms do not diffuse from the (11-22) surface to the (1-10-3) oriented grains. As the diffusion length of Al adatoms is smaller than that of Ga, we attribute the dependence of the grain density on the growth conditions mainly to the diffusion of gallium.21) This is supported by previous investigations, where grains were only observed for high Al contents >70%.25) The experiments show that the overgrowth of (1-10-3) oriented grains is more likely when the overall growth rate of the layer is decreased. The growth conditions investigated here could also impact the nucleation of the different crystal orientations.

#### Table I. Growth parameters for optimized AlGaN structure.

| Parameter        | AlGaN buffer layer | AlGaN overgrowth layer |
|------------------|--------------------|------------------------|
| P_{TMGa} (Pa)    | 1.10               | 0.75                   |
| P_{TMAl} (Pa)    | 0.40               | 3.70                   |
| P_{NH3} (Pa)     | 400                | 1250                   |
| Thickness (nm)   | 240                | 1000                   |
| Growth rate (μm h^{-1}) | 0.13               | 0.66                   |

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**Fig. 10.** Optimized AlGaN structure grown on m-plane sapphire, consisting of a low growth rate/grain density buffer layer and a high growth rate/grain density overgrowth layer.

**Fig. 11.** (Color online) 10 μm × 10 μm AFM images of the (a) buffer layer, (b) overgrowth layer, and (c) the full optimized structure (shown in Fig. 10).
orientations; however the change in growth conditions appears moderately affect the nucleation, which will be the subject of a further investigation.

5. Growth of two-step buffer layer

As the reduction in growth rate was caused by pre-reactions due to increasing the pressure (pressure study), increasing the MO supply beyond 12.0 Pa (increasing MO in principle should increase the growth rate, as initially observed), and increasing the V/III ratio (due to increase in ammonia flow), low grain densities should also be possible for growth conditions with low growth rate and low pre-reactions. Therefore to mitigate this, the carrier gas flow into the reactor was increased from 8 to 20 slm, in order to increase the flow velocity and reduce the possibility of the precursors interacting with each other in the gas phase before reaching the susceptor. Assuming that a low growth rate (<0.20 μm h⁻¹) is necessary to achieve a low grain density the precursors were reduced and the TMGa flow adjusted to reach an aluminum mole fraction of (80 ± 5)% in the layer.

To prove the applicability of a low growth rate buffer layer for growth of heterostructures, thick AlGaN overgrowth, with a layer of higher growth rate and without a deterioration of layer quality, has to be proven so that the growths of UV LED structures don’t last an inordinate amount of time. The optimized structure is shown in Fig. 10 and Table I shows the growth parameters for the buffer to be grown first and the subsequent overgrowth layer. The V/III ratio, temperature, carrier gas flow, and pressure were held constant at 280, 1090°C ± 10 °C, 20 slm, and 500 hPa, respectively.

Figure 11 shows the 10 μm × 10 μm AFM images of the (a) buffer layer, (b) overgrowth layer and (c) the full optimized structure (shown in Fig. 10). The AFM images corresponding the buffer and overgrowth layer where measured from the single layer growths directly on m-plane sapphire and then used in the optimized structure, resulting in the AFM image shown in Fig. 11(c). The buffer layer [Fig. 11(a)] was grown thin (240 nm), using a low MO supply (1.5 Pa) to achieve a low grain density/growth rate (0.20 μm h⁻¹, 3.0 × 10⁸ cm⁻², respectively). The overgrowth layer [Fig. 11(b)] was grown thick (1000 nm), using an increased MO supply compared to the buffer (4.45 Pa) to achieve a high grain density/growth rate (0.66 μm h⁻¹, 2.3 × 10⁹ cm⁻², respectively). The resulting structure [Fig. 11(c)] exhibited a grain density of 1.0 × 10⁶ cm⁻², and a rms roughness of 2.5 nm. However, due to the layer thickness or the unfavorable overgrowth conditions the grain height and width increased to 125 nm and 560 nm, respectively.

The experiment shows that once a low grain density surface is established, this can be maintained independent of the growth conditions. These results are favorable when growing full structures, as they have a low grain density, a smooth surface, high Al content, and are not plagued by the low growth rates. It is speculated that by optimizing the nucleation conditions one dominating (11-22) orientation without any grains can be achieved to create a buffer layer without grains that is preferred when growing thick, semipolar (11-22) AlGaN.

6. Conclusions

In conclusion, the growth of (11-22) AlGaN by MOVPE was performed, where the reactor pressure, MO supply, and V/III ratio were investigated to suppress the formation of grains, composed of the (1-10-3) crystal orientation. It was shown that increasing the mentioned parameters reduced the grain density and rms roughness, leading to a smooth surface morphology, while also decreasing the growth rate due the formation of pre-reactions. It was concluded that the reduced growth rate was the main factor for overgrowing misoriented grains. In order to allow for low grain density layers at reduced pre-reactions, the total gas flow into the reactor was increased. A low grain density AlGaN buffer layer was developed with a smooth morphological surface that was successfully overgrown to a thick low grain density layer.

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