

Article

MOCVD Growth of Relaxed, Crack-Free AlGa_N on Tiled GaN Substrates

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Abstract

Al_xGa_{1-x}N layers with up to 53% Al composition and 1.3 μm total thicknesses were grown utilizing step-graded layers on dense arrays of 10 × 10 μm² GaN tiles. While the layers were fully relaxed and crack-free, the higher Al composition sample showed increased X-ray line width, which did not correlate with a lack of increased dislocation density (as measured by TEM).

Keywords: AlGa_N; elastic relaxation; deep UV crystal; high Al content

1. Introduction

AlGa_N has a wide tunable bandgap range from 3.4 eV for GaN to 6.1 eV for AlN. While utilizing the full bandgap range is challenging, even an Al content of 0.7 provides a large bandgap of 5.1 eV [1–3]. This can be used to obtain light emitting diodes (LEDs) and laser diodes (LDs) across the ultraviolet (UV) wavelength range [4–10], as well as to fabricate high-power, high-frequency, and power switching devices [11,12]. The large bandgap also makes it viable for use in high-resolution optical lithography [13,14]. It is also being explored for use in UV photonics systems for potential on-chip optical communication [15–19].

AlGa_N epilayers with low dislocation densities are necessary for these applications, especially when moving to high Al content. The first challenge is to obtain good-quality, large-area AlGa_N substrates, which, at present, are not readily available. As such, various techniques are being developed, utilizing either GaN or AlN as the starting substrate. Lateral overgrowth of buried cracks was used to plastically relax AlGa_N layers with 20% Al composition on GaN templates [20], while overgrowth on serrated GaN templates yielded AlGa_N layers with up to 25% Al composition [21]. Sidewall growth of AlGa_N on porous GaN and AlN base layers resulted in an air bridged structure with reduced dislocation densities with Al composition of 24% [22] and 98% [23], respectively, as did AlGa_N layers grown via epitaxial lateral overgrowth on deep-grooved SiC substrates [24]. AlN on Sapphire [25] and SiC substrates are used to obtain AlGa_N epilayers. These were used to grow UV LEDs [26,27], while LDs were demonstrated on sapphire substrates using a semipolar orientation [28]. Several research groups are also using a sacrificial AlGa_N layer with up to 50% Al composition for flip chip UV LEDs [29,30].

Previously, we have had success using porous GaN pseudo-substrates (PSs) to grow crack-free, elastically relaxed AlGa_N [31,32]. In this work, we have extended the growth method and deposited AlGa_N directly on tiled GaN templates in an attempt to demonstrate a simpler method of obtaining relaxed AlGa_N.



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2. Materials and Methods

All layers in this study were grown epitaxially utilizing metal–organic chemical vapor deposition (MOCVD) using standard precursors trimethylgallium (TMGa), trimethylaluminum (TMAI), and ammonia (NH_3) in a single wafer Thomas Swan (Aixtron) close-coupled showerhead reactor. Initially, 2.8 μm thick GaN was grown on c-plane sapphire substrates using standard template growth conditions [33]. The samples were first patterned and dry etched with a 100 W BCl_3/Cl_2 etch for 600 nm, generating $10 \times 10 \mu\text{m}^2$ square tiles 2 μm apart, resulting in tiled substrates with a fill factor of $\sim 83\%$. The mutually orthogonal GaN [1-100] and GaN [11-20] were identified by substrate orientation (and verified later with x-ray diffraction), and the sidewalls of the tiles were aligned along them for easier characterization.

On top of the tile arrays, AlGaIn layers were grown by MOCVD at a temperature of 1155 $^\circ\text{C}$, in a predominantly hydrogen ambient environment at a pressure of 100 Torr and a NH_3 flow rate of 45 mmol/min. Metalorganic flow rates were set based on prior-grown fully strained AlGaIn on GaN calibration samples with TMGa between 8.8 and 14.6 $\mu\text{mol}/\text{min}$ and TMAI in the range of 1.0–2.4 $\mu\text{mol}/\text{min}$, respectively. The regrowth conditions were mostly similar to the optimized parameters from our previous work on porous GaN pseudo-substrates [32].

The AlGaIn layers were regrown in steps, and the samples were characterized after every regrowth step. Between each regrowth, the samples were cleaned with acetone and isopropanol to remove contaminants, which was followed by a buffered HF dip to remove the oxide layer and finished with a deionized water rinse. The surface morphology was characterized using an Asylum MFP-3D atomic force microscope (AFM) from Oxford Instruments. High-resolution x-ray diffraction (XRD) measurements were performed in a X'Pert MRD diffractometer equipped with a Pixcel3D detector (Panalytical) to obtain rocking curves to evaluate the film quality [34]. Area scans were also taken to generate reciprocal space maps (RSMs) around the GaN (11–24) reflection to determine composition and relaxation [35]. In addition, the samples were characterized by transmission electron microscopy. The TEM samples were first prepared via the Focus Ion Beam (FIB) technique using a Helios Dualbeam Nanolan 600 instrument (FEI); then, measurements were performed at 200 kV in the ThermoFisher Talos G2 200X system.

3. Experiment and Results

The schematic of the full epitaxial layer structure of the first samples grown on the tiled GaN is shown in Figure 1a. The first regrowth consisted of a 10 nm GaN transition layer, followed by an 180 nm thick step-graded AlGaIn layer, with each grade being 30 nm thick and with nominal Al compositions (x_{Al}) of 0.05, 0.09, 0.13, 0.17, 0.21, and 0.25, respectively. The regrowth was continued with a 330 nm thick AlGaIn with nominal $x_{\text{Al}} = 0.30$. Two further regrowths were performed, each with 400 nm AlGaIn and with a decreased nominal $x_{\text{Al}} = 0.28$ for easier relaxation, bringing the total thickness of the AlGaIn layer to 1.3 μm .

The final samples were crack-free. RSM evaluation of the first regrown ~ 500 nm thick AlGaIn stack showed $\sim 35\%$ Al composition, with a relaxation of $\sim 82\%$. The measured Al composition was higher than the nominal x_{Al} of 0.30, likely due to the composition pulling effect (the increased Al or In uptake from the gas phase with decreasing lattice mismatch) [36–38]. After depositing another 400 nm AlGaIn, which brought the total regrowth thickness to ~ 900 nm, the AlGaIn was $\sim 90\%$ relaxed with a composition of $\sim 32\%$ Al. The final regrowth resulting in a total $\sim 1.3 \mu\text{m}$ thick AlGaIn structure caused the layer to be fully relaxed with Al composition, staying the same at $\sim 32\%$ (Figure 1b) due to the enhanced Al uptake from the gas phase on the 90% relaxed $\text{Al}_{0.32}\text{Ga}_{0.68}\text{N}$.

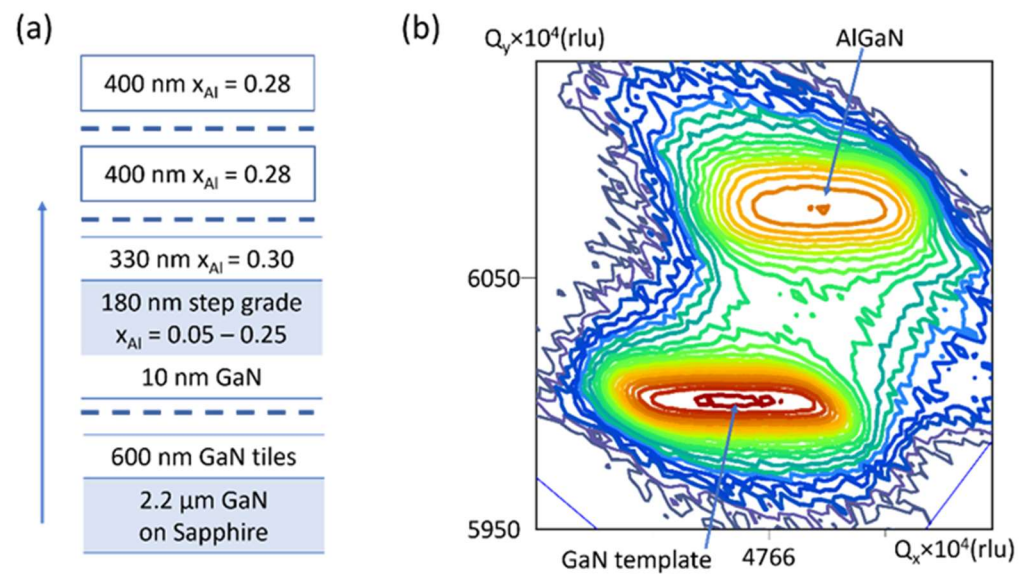


Figure 1. (a) Schematic of the first AlGaIn structure grown on tiled GaN. The vertical arrow represents the growth direction, and the dashed lines indicate regrowth interfaces. The x_{Al} values are nominal derived from the growth of strained AlGaIn on GaN test samples. (b) RSM obtained from the XRD of the final structure, with the GaN and AlGaIn peaks marked.

Rocking curve measurements with pseudo-Voigt fitting were used to obtain the ω -FWHM of the (0002) and (2-201) reflexes, which were 436 and 554 arc seconds, respectively. This is on par with good quality hetero-epitaxial growth of AlN on SiC [39]. The surface morphology was smooth after the first regrowth step, with a root mean square (RMS) roughness of 1.5 nm, as observed with AFM (Figure 2). The further regrowth steps increased the roughness to 2.3 and 2.7 nm, respectively. The rms increase resulted from the formation of growth spirals, a process which has also been observed for fully planar AlGaIn layers [40,41].

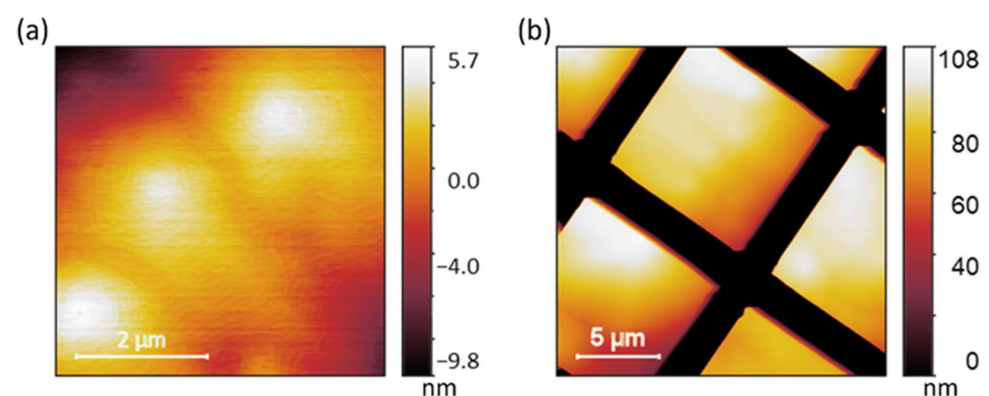


Figure 2. (a) $5 \times 5 \mu\text{m}^2$ AFM height image of the AlGaIn layer stack after final regrowth. (b) $20 \times 20 \mu\text{m}^2$ AFM image shows minimal sidewall growth on the tiles. The Al composition in the top layer was 32%.

With tiled GaN templates working well to grow relaxed AlGaIn with a composition of 32%, a higher Al composition was attempted. As before, a step-graded layer was utilized, this time with a steeper grading to obtain higher Al content. The layer consisted of 7 steps of 25 nm each, with nominal $x_{\text{Al}} = 0.05, 0.11, 0.17, 0.23, 0.29, 0.35$, and 0.41 , respectively. It was followed by a 325 nm AlGaIn layer with a nominal x_{Al} of 0.47 (Figure 3a). Two further regrowths were performed with an adjusted x_{Al} of 0.44 of 400 nm, each bringing the total AlGaIn stack thickness to almost 1.3 μm . Evaluation of RSMs was promising, with the first

stack showing 88% relaxation and 58% Al content. After the second regrowth the AlGa_N was fully relaxed and stayed so after a further regrowth, with a composition of ~53% Al (Figure 3b). As observed for the first sample, a significant increase in the Al incorporation was observed on the relaxed tiles.

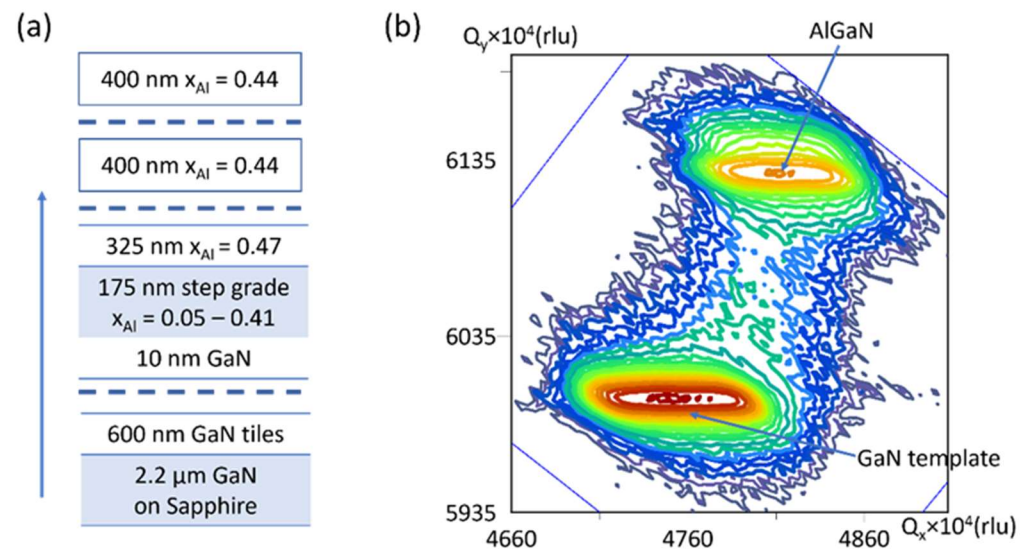


Figure 3. (a) Schematic of the higher Al content structure grown on tiled GaN. The vertical arrow represents the growth direction, and the dashed lines indicate regrowth interfaces. The x_{Al} values are nominal, derived from the growth of strained AlGa_N on GaN test samples. (b) RSM obtained from XRD of the final structure with the GaN and AlGa_N peaks marked.

Rocking curve measurements showed ω -FWHM values of 432 and 1008 arc seconds for the (0002) and (2-201) reflexes, respectively. In comparison with the previous sample, with a final Al content of 32%, a sharp increase in the off-axis value was observed. Again, the layers remained crack-free. AFM images showed the RMS roughness increasing from 1.2 nm for the first regrowth to 2.3 and 2.5 nm for the subsequent regrowth steps (Figure 4), similar to the observations made for the lower Al composition samples. As seen for the Al_{0.32}Ga_{0.68}N sample, the Al_{0.53}Ga_{0.47}N growth preferentially occurred on the top surface of the mesa, and no significant sidewall deposition was observed.

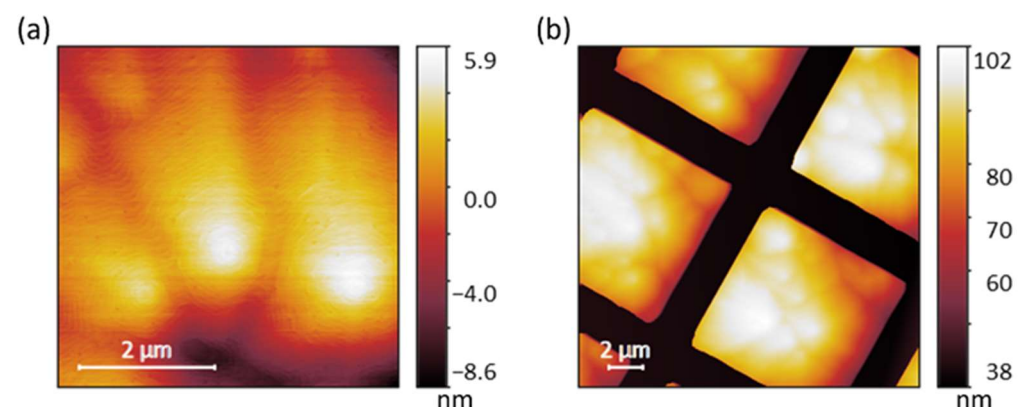


Figure 4. (a) $5 \times 5 \mu\text{m}^2$ AFM height image of the AlGa_N layer after final regrowth. (b) $20 \times 20 \mu\text{m}^2$ AFM image showing preferential regrowth on top only. The Al composition in the top layer was 53%.

To better understand the origin of the increased (2-201) ω -FWHM of the higher Al composition sample, high-angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) observation was performed. The visible vertical lines observed

in the STEM images are not related to dislocations and originate from the curtaining effect when performing cross-sectional milling. Horizontal white lines are visible at the base of the step-graded layer, which were associated with pure edge component dislocations (Figure 5). A few of these planar pure edge component dislocations were also observed in our work on partially relaxed InGaN grown by molecular beam epitaxy (MBE) on tiled porous GaN pseudo-substrates [42]. Plane-view TEM was also performed to obtain a count of dislocation density. Starting with the assumption that the dislocations go straight upward to the surface of the prepared sample and finding the dislocations per unit area gave us a dislocation density of $0.9\text{--}1.1 \times 10^9 \text{ cm}^{-2}$ for both the samples.

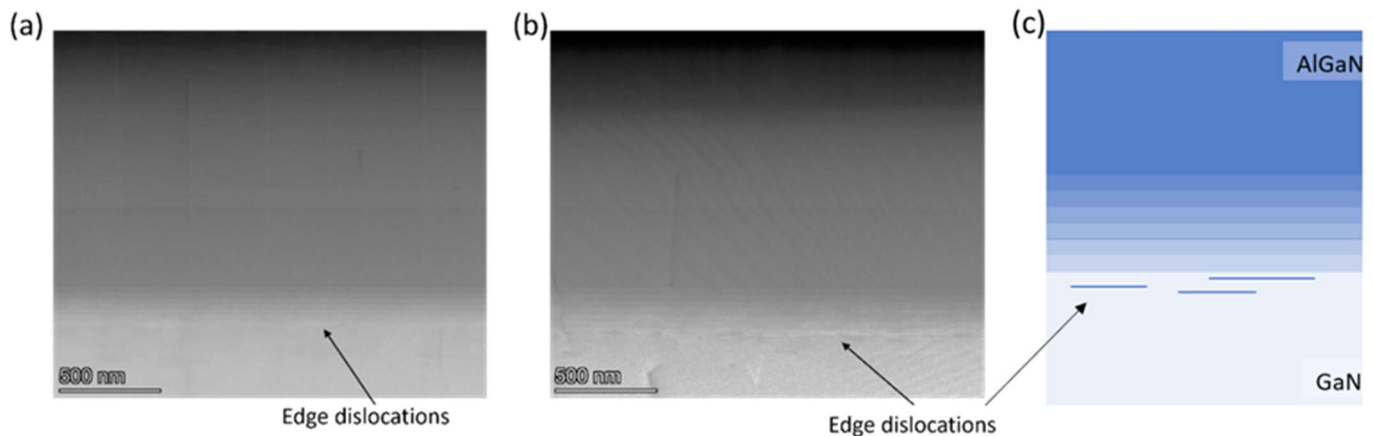


Figure 5. HAADF-STEM images showing edge dislocations in the GaN below the AlGaIn stack for sample with (a) 32% Al content and (b) sample with 53% Al content. (c) Schematic showing the presence of edge dislocations in the GaN template at the AlGaIn/GaN interface.

The results of the TEM investigation suggest that the increased (2-201) ω -FWHM of the higher Al composition sample is caused by reasons other than a higher dislocation density. It could be due to composition fluctuations across the tile [43], resulting from different rates of relaxation when comparing center to edge. Further investigations are needed to understand these findings. Moreover, in the experiments discussed here, the thickness of the graded AlGaIn layer was held constant, leading to a steeper grade for the higher Al composition sample. Further optimization of the thickness of the graded AlGaIn layer, as well as other process parameters, is expected to result in improved properties of the regrown AlGaIn.

4. Conclusions

Fully relaxed, crack-free $1.3 \mu\text{m}$ thick AlGaIn layers with 32% and 53% Al content were formed on dense $10 \times 10 \mu\text{m}^2$ GaN tile arrays. The epilayers were smooth, with a reasonable dislocation density of $\sim 10^9 \text{ cm}^{-2}$. The results show the potential of using dense tile arrays for the epitaxy of lattice mismatched materials.

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