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ABSTRACT

We report on the absence of strain relaxation mechanism in $Al_{0.6}Ga_{0.4}N$ epilayers grown on (0001) AlN substrates for thickness as large as 3.5 μ m, three-orders of magnitude beyond the Matthews–Blakeslee critical thickness for the formation of misfit dislocations (MDs). A steady-state compressive stress of 3–4 GPa was observed throughout the AlGaN growth leading to a large lattice bow (a radius of curvature of 0.5 m⁻¹) for the thickest sample. Despite the large lattice mismatch-induced strain energy, the epilayers exhibited a smooth and crack-free surface morphology. These results point to the presence of a large barrier for nucleation of MDs in Al-rich AlGaN epilayers. Compositionally graded AlGaN layers were investigated as potential strain relief layers by the intentional introduction of MDs. While the graded layers abetted MD formation, the inadequate length of these MDs correlated with insignificant strain relaxation. This study emphasizes the importance of developing strain management strategies for the implementation of the single-crystal AlN substrate platform for III-nitride deep-UV optoelectronics and power electronics.

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Al-rich (>50% Al) AlGaN alloys are the most promising compound semiconductors for deep-UV optoelectronics thanks to their direct, ultra-wide bandgap that can be tuned from 4.4 to 6.1 eV.^{1,2} AlGaN alloys are also poised to enable next-generation power electronic devices, given their superior Baliga's figure of merit as compared to SiC, GaN, and Ga₂O₃.^{3,4} AlGaN heterostructures have been grown typically on non-native substrates, such as SiC, sapphire, and Si(111). Given the heteroepitaxial nature of growth, threading dislocation (TD) densities of $\sim 10^9$ cm⁻² are the norm. In light-emitting diodes and photodetectors, TDs with a screw component increase the reverse-bias leakage current⁵ while those with an edge component have been found to suppress the radiative recombination efficiencies.⁶ In high electron mobility transistors, screw dislocations impede the off-state performance by enhancing the leakage current across the Schottky gate contact. This yields lower breakdown voltages, power loss due to leakage, and poor device reliability. Growth of thick AlGaN epilayers with lower dislocation density is, therefore, necessary for next-generation devices. The commercialization of single-crystal AlN substrates has propelled the development of Al-rich AlGaN epilayers with a drastically lower density of TDs $(<10^3 \text{ cm}^{-2})$.²⁹ This, in addition to the high thermal conductivity^{10,11} of the AlN substrates, is expected to enable compelling improvement to device performance and reliability. However, the increasing lattice mismatch with increasing Ga-content in the alloy engenders large compressive stress of several GPa, resulting in large wafer bow for the growth of thick AlGaN epilayers on AlN substrates.

For highly strained systems with very low TD density (<10³ cm⁻²), such as Al_{0.6}Ga_{0.4}N growth on AlN substrates (lattice mismatch of 1%), it is expected that beyond a certain critical thickness (CT), plastic strain relaxation occurs via the formation of misfit dislocations (MDs) that relax the in-plane strain of the epilayer. This mechanism typically results in a significant increase in threading dislocation density at the relaxation interface^{12–14} and has been observed in multiple lattice-mismatched systems.¹⁵ In the cubic (001) zinc blende structure, the relaxation primarily occurs by glide in the $\langle 0-10 \rangle$ {111}slip system as there exists a finite component of shear stress. For these structures, the critical thickness corresponding to the onset of misfit glide and plastic relaxation can be accurately predicted by the Matthews–Blakeslee (M–B) model.¹⁶ Similarly, for the case of semipolar, wurtzite III-nitrides, there exists a non-vanishing resolved shear

stress on the basal plane and the predictions of the M–B model have been proven to be quite accurate for MDs originating from the basalplane slip for InAlGaN layers grown on native GaN substrates.¹² In the polar (0001) wurtzite structure, however, the resolved shear stresses in both the basal and prismatic (90° with respect to the growth plane) planes vanishes, thereby leading to a geometrical limitation on the operative slip systems for strain relaxation.^{16,17} Of all the slip systems in (0001)-oriented III-nitrides, a finite driving force for dislocation glide exists only on the $\langle 11-23 \rangle \{11-22\}$ and $\langle 11-23 \rangle \{1-101\}$ systems, making them the only favorable systems for the glide of MDs.^{17,18} Despite accounting for these systems, the M-B model predicts critical thicknesses one to two orders of magnitude lower than the values obtained experimentally for polar AlGaN/AlN and InGaN/ GaN heterostructures.^{7,19–22}

Multiple reports on the growth of InGaN on native GaN substrates or low dislocation density GaN templates have discussed the observation of periodic arrays of MDs at the heterointerface as the strain relaxation mechanism.^{18,23,24} Iwaya et al. showed that the critical thickness for the introduction of MDs in the InGaN epilayer had a dependency on the TD density in the underlying GaN layer.²³ The critical thickness for pseudomorphic growth of polar AlGaN has been observed to be orders of magnitude higher than InGaN even for compositions with a comparable lattice mismatch.²⁰ Of the few reports on AlGaN growth on native AlN substrates, Dalmau et al. observed ~8% relaxation for a 400 nm thick $Al_{0.65}Ga_{0.35}N$ layer.⁹ Grandusky et al. observed pseudomorphic growth of Al_{0.6}Ga_{0.4}N until a thickness of $0.5 \,\mu m$.⁷ At larger thicknesses, they observed relaxation and an increase in TD density, which possibly formed via the interaction between MDs. Ren et al. reported the use of superlattice buffer layers for strain relief and found an inverse relationship between the degree of relaxation and surface roughness.8 An alternate mechanism of strain relaxation has been observed for polar InGaN/GaN^{21,25} and has been suggested to extend to AlGaN/GaN heterostructures.²⁰ It involves the generation of V-shaped dislocation half-loops on the growth surface followed by their penetration to the lower interface of the strained epilayer by the climb in the m-plane. Recently, Rudinsky et al. developed a quasi-thermodynamic growth model by accounting for the effect of material decomposition during dislocation half-loop formation on the critical thickness value and found a reasonable quantitative agreement with the literature data.20

In general, the onset of stress relaxation phenomena and the formation of MDs are not well understood in polar AlGaN layers with low TD density. The first part of this Letter aims to determine the pseudomorphic limit of $Al_{0.6}Ga_{0.4}N$ epilayers grown on single-crystal (0001) AlN substrates in light of various strain relaxation mechanisms. The lack of strain relaxation can lead to substantial bowing of the AlN substrate, which puts a limitation on its use for device processing and calls for strain management strategies. The second part of this Letter investigates the applicability of compositionally graded AlGaN as a potential strain relief layer while concomitantly maintaining the low TD density.

All samples in this study were grown on single crystal AlN substrates in a vertical, cold-walled, low-pressure metalorganic chemical vapor deposition system with RF heating. The 1-in., (0001) AlN substrates were 500 μ m thick and had an average TD density of $<10^3$ cm⁻², a miscut of $0.2^{\circ} \pm 0.1^{\circ}$ toward the m-direction, a radius of curvature greater than 50 m, and as-received surface roughness of <1 nm. The AlN substrates were processed from AlN boules grown by physical vapor transport.^{26,27} Pre-epitaxy acid-based AlN surface preparation, hydrogen annealing, and nitridation are described elsewhere.²⁸ Three series of samples were grown as shown schematically in Fig. 1. In the first series, $0.5 \,\mu m$ homoepitaxial AlN was deposited followed by the growth of Al_{0.6}Ga_{0.4}N with three thicknesses: 0.95, 1.8, and 3.5 μ m [Fig. 1(a)]. As has been reported previously, surface kinetics play a significant role in governing the morphology and composition of AlGaN layers grown on AlN substrates.²⁹ A growth rate of \sim 500 nm/h was achieved at the growth temperature of 1100 °C by flowing 0.013 mol/min of ammonia (NH₃), 6.7 µmol/min of trimethylaluminum (TMA), and 21 µmol/min triethylgallium (TEG) at a total pressure of 20 Torr to achieve AlGaN composition of 60% Al, following the model discussed by Washiyama et al.³⁰ In the second series, a compositionally graded layer was introduced as a potential strain relief layer [Fig. 1(b)]. The grading was achieved by linearly varying the TEG flow rate throughout the growth of the AlGaN film from 4 to 24 µmol/min and keeping all other growth conditions constant to vary the nominal composition from 90% Al to 60% Al. For the third series, a step-graded buffer layer was implemented [Fig. 1(c)]. The composition gradient in the linearly graded film was confirmed using electron dispersive spectroscopy in a transmission electron microscope (TEM). The AlGaN film thicknesses were determined using Z-contrast cross-



FIG. 1. Epi-stacks with (a) varied thickness of Al_{0.6}Ga_{0.4}N epilayer grown directly on homoepitaxial AIN (b) Al_{0.6}Ga_{0.4}N epilayer grown using a linearly graded AlGaN buffer layer (c) Al_{0.6}Ga_{0.4}N epilayer grown using a triple-heterostructure step-graded AlGaN buffer layer.

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FIG. 2. (a) (00.4) and (b) (10.5) RSMs of 1.8 μ m Al_{0.6}Ga_{0.4}N film showing a strict vertical q_x alignment to AlN, indicative of a fully strained layer; (c) (top) AFM surface morphology of the 3.5 μ m thick Al_{0.6}Ga_{0.4}N epilayer showing smooth morphology with bilayer steps (bottom) a line scan showing average step height and terrace width of 0.25 and 70 nm, respectively.

sectional scanning electron microscopy. The radius of curvature of the substrate was measured using the rocking curve method³¹ in a Philips X'Pert Materials Research Diffractometer. The strain state of the AlGaN epilayers was characterized using reciprocal space mapping (RSM) in a triple crystal configuration. The surface morphology was acquired in a tapping mode using Asylum Research MFP-3D atomic force microscope (AFM). The possible formation of dislocations in the graded layer stacks was investigated by cross-sectional TEM in weak beam conditions at an acceleration voltage of 300 kV.

In general, during an epitaxial growth, stress generation can have various sources. These may include grain coalescence,³² lattice mismatch, the inclination of a-type TDs,³³ and thermal expansion mismatch during cooldown. The grain coalescence stress can be ruled out as the AlN homoepitaxial growth does not proceed via nucleation but rather via a step flow.³⁴ The inclination of TDs would be expected to generate negligible stress because the density of TDs is low.³⁵ The low thermal expansion coefficient mismatch of AlN and Al_{0.6}Ga_{0.4}N will lead to insignificant cooldown stress.³⁶ Therefore, only the predominant lattice mismatch stress should be related to the generation of large compressive stress during the epitaxy. The potential stress relaxation mechanisms that can operate against the generated compressive stress are nucleation (and glide) of misfit dislocations,¹⁹ generation of V-shaped dislocation half-loops as discussed by Rudinsky et al.,²⁰ surface roughening³⁷ and buckling, and delamination of the AlGaN film. As evidenced below, none of these mechanisms were found to operate in this study.

First, the strain state of the AlGaN layers was investigated using reciprocal space mapping. The strict alignment of q_x reciprocal space vector of the Al_{0.6}Ga_{0.4}N layer to AlN, indicative of a pseudomorphic growth, was observed for all the samples with the representative RSM for the 1.8 μ m sample shown in Figs. 2(a) and 2(b) for the (00.4) on-axis and (10.5) off-axis planes, respectively. Table I summarizes the curvature data from the three samples. Compressive growth stress of 3.3–3.8 GPa can be extracted by using the Stoney equation for the case of spherical curvature (Table I).³⁸ The large stress value confirms that the lattice mismatch stress in the AlGaN layer dominates the stress generation and supports the absence of strain relaxation in these samples. Step-flow surface morphology³⁴ with bilayer steps was observed for all the samples with an rms roughness of 0.1–0.15 nm. The representative morphology is shown in Fig. 2(c) for the 3.5 μ m thick Al_{0.6}Ga_{0.4}N epilayer. Average step height and terrace width of 0.25 and 75 nm

TABLE I. Measured value of substrate radius of curvature and calculated value of stress for samples with varying $Al_{0.6}Ga_{0.4}N$ thickness.

Thickness	Radius of curvature	Stress
0.95 μm	6 m	-3.8 GPa
$1.8\mu{ m m}$	3.4 m	-3.6 GPa
3.5 µm	2 m	-3.3 GPa

corresponding to the substrate miscut of 0.15° can be extracted from the line scan as shown in Fig. 2(c) (bottom). This confirms that the bilayer step flow mechanism was retained throughout the AlGaN growth. Surface roughening, which can be associated with strain relaxation, was not observed. Relaxation by gliding of misfit dislocations is typically associated with a surge in the density of TDs.^{12,13} The long straight steps in the AFM image [Fig. 2(c)] indicate that they are not pinned by TDs, therefore suggesting their absence.

Figure 3 shows the estimations of the critical thickness for MD nucleation in the $\langle 11-23 \rangle \{ 11-22 \}$ slip system from models by Matthews-Blakeslee, People-Bean, and Holec.³⁹ Experimentally measured data points for pseudomorphic growth of the three samples in this study and values reported by Dalmau et al.9 and Gradusky et al.7 are shown alongside. A large discrepancy between the experimental results and theoretical predictions is observed with the experimental thickness of 3.5 µm exceeding the predicted critical thickness values by more than two orders of magnitude for the Al_{0.6}Ga_{0.4}N layer. These results suggest either a large barrier for the nucleation of MDs or a significantly larger gliding path that is pinned by intrinsic point defects or impurities, suppressing the glide.²⁰ The former might be caused by an insufficient resolved shear stress for overcoming the Peierls barrier in the basal plane.²⁰ Following Rudinsky et al.'s model, plastic relaxation via dislocation half-loops is not expected for Al content >55% for a growth temperature of 1100 °C.²⁰ Finally, neither surface roughening nor buckling were observed even for the $3.5\,\mu m$ thick film. All these observations confirm the absence of strain relaxation in the studied Al_{0.6}Ga_{0.4}N films.

The large compressive stress led to an extreme wafer curvature of 0.5 m^{-1} in the $3.5 \,\mu\text{m}$ thick AlGaN sample. Such wafer bowing is considered unworkable for large-area device processing. This makes necessary a strain management strategy that promotes strain relief in the epitaxial stack. One particular approach is the engineered, compositionally graded layer, as described by Xie.⁴⁰ Using this approach, dislocation half-loops are nucleated in the graded layer so that the strain in the structure is predominantly relieved by the misfit segments of the



FIG. 3. Critical thickness as a function AIGaN composition following different models. Experimentally measured data points of pseudomorphic AIGaN films obtained in this study and those reported by Dalmau *et al.*⁹ and Grandusky *et al.*⁷ are plotted alongside.

dislocation half-loops that lie parallel to the surface. It is to be noted that the threading segments of the half loop make no contribution to the relaxation of the strain since it is the discontinuity at the interface caused by the in-plane Burgers vector component of the misfit dislocation that relieves the epilayer strain. As a consequence of the compositional grading (or step grading), the strain energy builds up gradually in the layers such that when it reaches a critical value, only a few dislocation half-loops are needed to relax the mismatch as shown in Fig. 4 (right). Therefore, the threading segments of the loops can glide over long distances (no pinning through dislocation entanglement), resulting in long lengths of strain-relieving misfit dislocations. As the growth progresses, this process is repeated at various thicknesses, resulting in long misfit dislocations that are distributed over the layer thickness and few threading dislocations at the film surface, as shown in Fig. 4 (right). Without strain management, a large number of half-loops must nucleate simultaneously to accommodate the rapid buildup in strain energy, providing for a larger threading dislocation density [Fig. 4 (left)] than the one arising from a graded layer. It is important to realize that once copious threading components are generated, they are not only difficult to reduce but they also inhibit strain relaxation via misfit elongation due to entanglement. Thus, the strain must be controlled throughout the growth process.

Figure 5(a) shows a weak-beam dark-field cross-sectional TEM image for the $Al_{0.6}Ga_{0.4}N$ sample grown with a linearly graded AlGaN strain relief layer with the epi stack depicted in Fig. 1(b). As expected, spatially separated MDs are formed throughout the graded layer. Minimal interaction between the MDs is evident with a single TD observed in the entire image (top right). Figure 5(b) shows the measured (00.2) omega-2theta scan (red curve) for the epi stack and the simulated scan (blue curve). Surprisingly, the simulated scan considering fully strained graded AlGaN and $Al_{0.6}Ga_{0.4}N$ layers showed excellent matching to the measured scan, indicating that despite the strain relief layer, the AlGaN is still fully strained.

To conclusively confirm the strain state, (00.2) on-axis and (10.5) off-axis RSMs were measured for the compositionally graded structure and are shown in Figs. 6(a) and 6(b), respectively. A strict vertical alignment along the q_x axis was observed, which again points to the absence of strain relaxation in this structure. This result suggests that



FIG. 4. Nucleation of dislocation half-loops: abrupt strain relief by copious short misfit segments resulting in many threads at the surface (left), and gradual strain relief by misfit elongation and low density of threads at the surface (right).



FIG. 5. (a) Cross-sectional dark field TEM image of the linearly graded AlGaN stack shown in Fig. 1(b) with g = (1-210). Horizontal MDs are seen only in the graded AlGaN layer. (b) Comparison of measured (00.2) omega scan for the graded layer stack and the simulated scan for fully strained state of the linearly graded and bulk Al_{0.6}Ga_{0.4}N layers.

the cumulative length of MDs formed in the graded layer is possibly insufficient^{35,41} to engender any observable relaxation. Figure 6(c) shows the (10.5) off-axis RSM measured for the step graded structure depicted in Fig. 1(c). A strict vertical alignment along the q_x axis was observed not only for the $Al_{0.85}Ga_{0.15}N$, $Al_{0.80}Ga_{0.20}N$, and $Al_{0.70}Ga_{0.30}N$ step graded buffer layers but also for the top $Al_{0.60}Ga_{0.40}N$ layer confirming the inefficient strain relaxation in this structure. In conclusion, although the formation of misfit dislocations was observed, neither composition grading layers nor step grading was effective in stress relaxation in thick Al_{0.6}Ga_{0.4}N epilayers grown on the single-crystal AlN substrate. The large strain was found to be accommodated primarily by wafer bowing. These results point to the presence of a large barrier for the nucleation of MDs in Al-rich AlGaN epilayers, therefore limiting their density. The associated large wafer bowing calls for the design of an effective strain management strategy





that will also retain the low TD density. This is of critical importance in realizing the consummate promise of the single-crystal AlN substrate platform for III-nitride deep-UV optoelectronic and power electronic applications.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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