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Strain compensated superlattices on m-plane gallium nitride by ammonia molecular beam epitaxy

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The results of tensile strained AlN/GaN, AlGaN/GaN, and compressive strained InGaN/GaN superlattices (SLs) grown by Ammonia MBE (NH₃-MBE) are presented. A combination of atom probe tomography and high-resolution X-ray diffraction confirms that periodic heterogeneous structures of high crystallographic quality are achieved. Strain induced misfit dislocations (MDs), however, are revealed by cathodoluminescence (CL) of the strained AlN/GaN, AlGaN/GaN, and InGaN/GaN structures. MDs in the active region of a device are a severe problem as they act as non-radiative charge recombination centers, affecting the reliability and efficiency of the device. Strain compensated SL structures are subsequently developed, composed of alternating layers of tensile strained AlGaN and compressively strained InGaN. CL reveals the absence of MDs in such structures, demonstrating that strain compensation offers a viable route towards MD free active regions in III-Nitride SL based devices. Published by AIP Publishing. [http://dx.doi.org/10.1063/1.4991417]

INTRODUCTION

Light Emitting Diodes (LEDs) and Laser Diodes (LDs) in the III-Nitride system have received significant research attention and realized major technological progress over the past two decades.¹ Heterostructures of III-Nitrides offer larger band offsets than conventional arsenide, phosphide, or antimonide semiconductors. The band gaps of the ternary InGaN and AlGaN span the entire visible range and regions of the ultraviolet range, enabling light emission over a wide range of wavelengths.²⁻⁵ Optoelectronic devices perform optimally on non-polar or semi-polar orientations.⁶ These non-basal orientations minimize the Quantum Confined Stark Effect (QCSE) by reducing the strength of the polarization fields inherent to wurtzite nitride heterostructures.⁷

At the same time, strain-induced defects, particularly misfit dislocations (MDs) at heterointerfaces, act as non-radiative recombination centers on such non-basal orientations. MDs reduce the stress in the film and thus the strain energy, but at the expense of the line energy from the dislocation. Beyond a certain critical thickness, it will be energetically favorable to form MDs to relieve the misfit stress in a thick, coherently strained layer.⁸ MDs are particularly problematic to optoelectronics, as they act as non-radiative charge trapping centers near the heterostructure, often in the intrinsic region of a device.⁹ MD formation energy, strain energy, and therefore the critical thickness, however, are highly dependent on the crystal structure and orientation. During on-axis c-plane growth, there is no resolved shear stress available to glide threading dislocations (TDs) on the (0001) c-plane and leave basal slip plane MDs; the same holds true for slip on the {1100} m-planes, which would leave behind prismatic slip MDs. Rather than relieving strain via MDs, c-plane films must therefore relax by either cracking in the tensile state, or succumb to morphological instabilities resulting from thick layers in compression.¹⁰ On semipolar orientations, shear stress on both the (0001) c-plane and the {1010} m-planes is possible, and both basal and prismatic MDs are observed.¹¹⁻¹⁶ In m-plane oriented strained layers, there is no shear stress on the vertically oriented (0001) plane, but there is shear stress on the inclined {1010} prismatic m-planes for strained layers, meaning only prismatic MDs are observed.⁵,⁶,¹⁶ The manner through which strain is relieved is a major issue in III-Nitride heterostructures as there are large lattice mismatches between the binary endpoints of the system; −2.4% and 11.2% for AlN and InN, respectively, on GaN along the [1120] a-direction and −3.9% and 10.0% for AlN and InN, respectively, on GaN along the [0001] c-direction. Ultimately, the critical thicknesses place limits on the compositions and thicknesses of AlGaN and InGaN layers attainable in optoelectronic devices.

Success at mitigating strain has been achieved with homogeneous lattice matched In₉₉.₈₈Al₀₉₉.₈₂N to GaN, primarily for lateral devices.¹⁷,¹⁸ Fixing the alloy composition, however, fixes the bandgap of the InAlN. This paper therefore investigates a strain compensated superlattice (SL) approach, which can simultaneously suppress misfit stress-induced MDs while retaining the ability to tune confined states in a SL. Strain compensated m-plane SLs may be of great use in the development of nitride intersubband photodetectors,¹⁹ and may lead to the possibility of more exotic optoelectronic devices such as quantum cascade lasers²⁰ and polariton lasers.²¹

EXPERIMENTAL

Ammonia Molecular Beam Epitaxy (NH₃-MBE) was used for all samples studied. NH₃-MBE affords a large and stable nitrogen-rich growth regime.²²,²³ All samples were grown using a Veeco 930 MBE system, modified with a custom NH₃ shower head injector. Dual-filament Knudsen effusion cells provided the metal species. The substrate temperature was
monitored by optical pyrometry calibrated to samples that were Indium bonded to a Silicon wafer, enabling thermal coupling to the substrate heater. InGaN and AlGaN growths were calibrated by growing 20–30 nm strained films at the fixed metal flux ratios, and subsequently determining composition and film thicknesses by analysis of the peak separation from the GaN and thickness fringe pattern in ω-2θ scans in high resolution x-ray diffraction (HRXRD), assuming coherent strained films.

The substrates used were n-type (1010) m-plane bulk substrates, provided by Mitsubishi Chemical Corporation, with the TD density in the ∼10^7 cm^-2 range. All samples were miscut 4° towards the a-direction from the (1010) plane, which aided the morphology in the low temperature regime, as will be discussed. The samples were degreased in standard solvents (acetone, methanol, and isopropanol) before loading, and baked at 400°C for 1 h under vacuum prior to growth. All structures began with a ~100 nm buffer GaN layer to bury any contamination at the initial growth interface. Standard NH3-MBE growth conditions at 820°C under 200 sccm of NH3 were used for bulk GaN layers.23 AlN layers were grown at reduced temperatures of 585–620°C, which have been shown to minimize Ga impurities.24 InGaN layers were grown at reduced temperatures of 585–620°C depending on the desired target indium composition.

Triple axis high resolution x-ray diffraction (HRXRD) scans were performed on a Pananalytical MRD PRO Thin Film Diffractometer. Additional characterization to identify and locate defects in the heterostructures was performed using panchromatic cathodoluminescence (CL) spectroscopy using a Gatan MonoCL4 system on an FEI Inspect S scanning electron microscope (SEM). For the CL experiments, the excitation voltages used were in the range 5–10 keV range which corresponds to electron penetration depths in GaN on the order of 150–500 nm.

Atom probe tomography (APT) was subsequently done on representative SL samples. An additional 200 nm of GaN was regrown on the samples to protect the regions of interest during the APT specimen preparation. Sharp tips were prepared with a FEI Helios 600 dual beam Focused Ion Beam (FIB) instrument following the standard procedure with the final FIB voltage down to 2 kV to minimize Ga induced damage.25 To allow for the evaporation of the complete SLs and avoid fractures of the APT specimen, the FIB preparation is optimized to obtain tips with low shank angles. The increase of the applied voltage to evaporate the samples and consequently the increase of the probability of tip fractures was reduced. APT analyses were performed with a Cameca 3000X HR Local Electrode Atom Probe (LEAP) operated in the laser-pulse mode (13 ps pulse, 532 nm green laser, and 10 µm laser spot size) with a sample based temperature of 40°K. The laser pulse energies were different between each sample but were kept below 0.5 nJ. A detection rate of 0.005 atoms/pulse was set during the analysis. The APT 3D reconstruction was carried out using the commercial software IVAS™™. The reconstruction is optimized to obtain flat interfaces between the layers in the SLs and to respect the region of interest’s thicknesses obtained from XRD measurements.26

**STRAINED SUPERLATTICES**

Initial structures consisted of strained SLs, to determine both the structural quality and the upper limits of the number of periods that could be grown before the onset of extended defect formation. A series consisting of 5, 10, and 20 periods of 0.9 nm of AlN and 5.5 nm GaN was first explored. Such fully strained AlN/GaN structures would have calculated strains of −2.4% along the [1120] a-direction and −3.9% along the [0001] c-direction. Atomic Force Microscopy (AFM) scans over 5 µm x 5 µm areas showed a smooth morphology, indicating that the growth remained in a stable two-dimensional growth mode, with sub-nanometer RMS roughness. Symmetric ω-2θ HRXRD scans of the series are shown in Figs. 1(a)–1(c). The evolution of both the SL(0) peak to the right of the GaN substrate peak, as well as the SL peak to the left of the GaN substrate peak is observed as the number of periods increases. Clear Pendellösung fringing indicates good interfacial quality through the SLs. Panchromatic CL scans of the same series in Figs. 1(d)–1(f), however, indicate MD formation along the [0001] c-direction, even on the samples with the fewest number of periods. The MDs have a spacing of ~10 µm. This is consistent with the prismatic MD formation.14,15 Multiplying the projection of the Burger’s vector along the [1120] a-direction by this spacing gives an estimate of the plastic relaxation, in this case merely ~0.0016% (plastic relaxation εp = 1.6 × 10^-5). By the 20-period sample, cracking was observed on (0001) planes (crack traced along the [1120] a-direction). As the prismatic MD density is too low to allow for efficient plastic relaxation, cracking occurs to relieve the accumulated strain energy beyond a certain number of periods. Therefore, while the onset of prismatic MD occurs rapidly for pure AlN/GaN SLs on the m-plane orientation, it is inefficient at effectively relieving strain energy, and further increasing the number of periods will eventually lead to cracking.

Further investigation of the SLs with both ternary alloys, Al0.5Ga0.5N and In0.1Ga0.9N were explored. One key difference between these samples is the strain sign difference, indicating tensile versus compressive strain. A 10 period 1.5 nm Al0.5Ga0.5N/4 nm GaN sample (with an additional AlGaN 11th barrier for confinement) with a 20 nm GaN cap, and a 10 period 6.1 nm In0.1Ga0.9N/4 nm GaN sample with a 10 nm GaN cap were grown. While compositions, number of periods and layer thicknesses were chosen for subsequent spectroscopic measurements,27 these Al0.5Ga0.5N/GaN and In0.1Ga0.9N/GaN SLs had unbalanced average strains of −1.2% and 1.1% in the SL, respectively. Figures 2(a)–2(c) show HRXRD, CL, and AFM scans of the Al0.5Ga0.5N/GaN sample, while Figs. 2(d)–2(f) show the same scans of the In0.1Ga0.9N/GaN sample. Once again, clear Pendellösung fringing and SL peaks were visible in HRXRD, indicative of coherent growth and a high crystal quality. Prismatic MDs were once more seen by CL along the [0001] c-direction.14,15 Even lower MD linear densities of 2.0 × 10^9 cm^-1 and 1.8 × 10^9 cm^-1, for the InGaN/GaN and AlGaN/GaN SLs, respectively, indicate that the sign of the average SL strain did not affect the MD formation. Additionally, while AFM RMS roughness of both samples was again sub-nanometer, a
meandering morphology was evident in the In_{0.10}Ga_{0.90}N/GaN SL, a consequence of the lower temperature required for higher composition InGaN growth. Although cracking or other defects are not seen on these 10 periods samples, once more the prismatic MD densities are too low to efficiently reduce the misfit stress, and we predict cracking or other defects to similarly occur as in the AlN/GaN case for larger numbers of periods.

Further investigation of the strained AlN/GaN and AlGaN/GaN SL structures by APT was undertaken. A 20 x 20 x 48 nm$^3$ 3D volume of six AlN/GaN periods is shown in Fig. 3(a). The volume is extracted in the center of the global SL volume where the reconstruction is optimized. From this volume, Al atoms are only detected in the AlN layers. Figure 3(b) reports the variation of the Al/(Al+Ga) and Ga/(Al+Ga) ratios along the growth axis and within the volume on Fig. 3(a). The peak Al values within the Al layers range from 83% to 95% with a 4% deviation, and are listed in Table I. Thicknesses of the AlN layers are measured at 10% of the peak Al value, again listed in Table I. Thicknesses of the AlN layers are reported as 10% of the peak Al value, again reported in Table I. From the concentration profiles on Fig. 3(b), it is not possible to conclude which interface (GaN on AlN or AlN on GaN) is the sharpest, however, as the AlN layers are only approximately 4 monolayers thick. From the APT data, we can conclude that the layers are nearly pure AlN, for reasons to be explained shortly.

Similarly, a 20 x 20 x 48 nm$^3$ 3D volume of six periods of an AlGaN/GaN SL sample is presented in Fig. 3(d). Again, the volume is extracted in the center of the SL reconstruction. Figure 3(e) reports the variation of the Al/(Al+Ga) and Ga/(Al+Ga) ratios along the growth axis and within the volume in Fig. 3(d). The average Al fractions in the AlGaN layers ranged from 44 to 53% with a 4% deviation, and are listed in Table I. Thicknesses of the AlGaN layers are measured between 10% of the plateau Al value, again listed in Table I. Taking the interface length as the distance between 10% and 90% relative concentrations between the plateau values, the bottom interface (AlGaN on GaN) is sharper than the top interface (GaN on AlGaN). The average thickness of the bottom interface is 0.9 ± 0.2 nm while the top interface is 1.6 ± 0.3 nm. In the case of the AlGaN/GaN SL, the extracted APT AlGaN compositions agree with the HRXRD fit value of 53%, while the thicknesses, removing the interfacial lengths agree more closely with the HRXRD fit AlGaN thickness of 1.8 nm.

The difficulty in quantifying the interfaces on APT samples with high Al content AlGaN or nominally pure AlN interlayers is that it may violate the assumption of homogeneous evaporation from the tip surface. In practice, AlN is much more difficult to evaporate than GaN. During the evaporation through a GaN/AlN/GaN interface, the top GaN layer is preferentially evaporated leading to a modification...
of the hemispherical tip shape. The shape of the tip flattens, leading to local variations of the projection law of the ions and local magnification artifacts. These artifacts are responsible for the Al under-density observed at the GaN/AlN interfaces in Figs. 3(a) and 3(d) and prevent accurate depth reconstruction. An ideal method to obtain accurate depth reconstruction would require real time knowledge of these variations in the tip morphology, which would in turn depend on knowing the AlN layer thickness and interface sharpness a priori. Such an analysis is beyond the scope of this study.

This inherent difficulty of APT reconstruction and positioning of interfaces in such thin AlN samples leads to a larger discrepancy between the APT extrapolated high Al content AlGaN or nominally pure AlN thicknesses and the thicknesses extracted from HRXRD fitting of SL fringing. In both the AlN/GaN and AlGaN/GaN SL case, the HRXRD fitted values more closely matched the expected layer thicknesses from growth calibrations. Subsequent APT analysis on SL containing layers of lower composition Al$_{0.25}$Ga$_{0.75}$N in the Strain Compensated Superlattices section do not suffer from this discrepancy.

**STRAIN COMPENSATED SUPERLATTICES**

The Strained Superlattices section illustrated that while high quality, strained SL structures can be grown on $m$-plane orientations, and that prismatic MDs in such SLs form within a few number of periods, their density is too low to relieve any significant misfit strain. For any of the possible applications discussed in the introduction, SL active regions will need to consist of tens, if not hundreds, of periods. Even if sufficient regions of such SL structures might remain optically active, the effects of MDs, or eventual cracking, on electrical injection at the required current densities on such strained superlattice structures are a significant concern. There remains, however, the possibility of combining InGaN and AlGaN into a single period to balance the misfit. One first chooses a fixed magnitude of misfit strain, and then calculates the AlGaN and InGaN compositions grown pseudomorphically on GaN, respectively, corresponding to the negative tensile strain and positive compressive strain of that chosen magnitude. One could then grow a SL consisting of periods of AlGaN and InGaN layers of equal thickness; the average stress over the period would be zero, assuming relaxation did not occur within either individual layer. In principle, such a SL could be repeated for many periods without the onset of MDs.

However, determining the best method to create such structures is a growth challenge. Optimal growth of In containing nitrides requires lower growth temperatures, due to the higher In vapor pressure, while the converse holds for Al containing compounds. Furthermore, the In incorporation properties by NH$_3$-MBE are orientation dependent. Rather
than attempting to grow structures in each layer’s optimal growth regime with long growth interrupts for temperature ramping (which itself might introduce interfacial roughening and unintended impurities), a constant growth temperature route was chosen. A structure consisting of a 0.6% strain corresponds to Al$_{0.25}$Ga$_{0.75}$N and In$_{0.06}$Ga$_{0.94}$N was chosen, with 3.6 nm layers of each. Two 20 period structures were grown, with n-type Si doping in either the AlGaN barriers or InGaN wells, and an additional 21st AlGaN barrier to provide electron confinement for subsequent spectroscopic measurements. For this In composition on the $m$-plane, a constant growth temperature of 620°C was used. AFM and HRXRD in Figs. 4(b), 4(e), 4(a), and 4(d) did not show any noticeable degradation of the structure from AlGaN being grown at such low temperature. The 0th order SL peak position of the HRXRD ω-2θ scans in Figs. 4(a) and 4(d), which provides information about the out of plane lattice constant of the epitaxial layer averaged over one period of the SL, is nearly aligned with the GaN substrate peak. This is further evidence that the strain is compensated in each SL period. The miscut, the thinness of the individual layers, and the alternating SL structure all likely contributed to maintaining the morphological stability. CL images of both well and barrier doped structures in Figs. 4(b) and 4(e) revealed a lack of MD, with only pre-existing TDs from the substrate. We take this as evidence that strain compensated AlGaN/InGaN SLs can be grown, and that such SL structures prevent MD stress relaxation.

TABLE I. Tabulated values of layer thicknesses and when applicable, alloy percentages of AlN, AlGaN, and InGaN in the strained AlN/GaN and AlGaN GaN SLs and an InGaN/AlGaN strain compensated SL mentioned in the text.

<table>
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<th>AlN/GaN SL</th>
<th>AlGaN/GaN SL</th>
<th>InGaN/AlGaN SL</th>
<th>InGaN/AlGaN SL</th>
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</table>
Further APT analysis on an InGaN/AlGaN strain compensated SL was also performed. A $20 \times 20 \times 53$ nm$^3$ 3D volume of 6 InGaN/AlGaN periods is presented in Fig. 5(a). The volume is extracted in the center of the SL reconstruction. Figure 5(b) reports the variation of the Al/(Al+Ga+In), In/(Al+Ga+In) and Ga/(Al+Ga+In) ratios along the growth axis and within the volume on Fig. 5(a). The plateau Al fraction in the AlGaN layers, listed in Table I, ranges from 17% to 21%, with a 3% deviation. We note that although the Al content was slightly lower than designed, the structure contained an additional 21st AlGaN barrier. The plateau In fraction is similar for each InGaN layer at 0.07±0.02. The average thickness of the AlGaN layers is 4.1±0.4 nm while the average thickness of the InGaN layers is 4.1±0.3 nm. These values are in decent agreement with the HRXRD fitted values of 4.2 nm $\text{Al}_{0.23}\text{Ga}_{0.77}\text{N}$ barriers, with 5.0 nm $\text{In}_{0.06}\text{Ga}_{0.94}\text{N}$ wells.

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FIG. 5. (a) APT of an AlGaN/InGaN SL structure, with (b) accompanying line profile of Ga, Al, and In concentration. The distribution of (c) In content and (d) Al content in the respective InGaN and AlGaN layers is shown to be binomially distributed, indicative of a random alloy without clustering.
Statistical distribution analysis (SDA) is used to highlight alloy fluctuations in both the AlGaN and the InGaN. Two 20 × 20 × 2 nm³ sampling volumes are extracted in an AlGaN layer and an InGaN layer. The sampling volumes were divided into 100 atoms bins and the Al and In fractions were calculated. Figures 5(c) and 5(d) show the experimental distribution of the binned Al fraction and In fraction, respectively. The experimental distributions of compositions are compared to the binomial distributions that would be expected of a random alloy [dotted curve of Figs. 5(c) and 5(d)]. A χ² test is used to quantitatively compare the two distributions. Both the presented AlGaN and InGaN alloy distributions passed the χ² test with p-values of 0.91 and 0.34, respectively. The same test was repeated on sampling volumes composed of 50, 100, and 150 atoms, taken from several separate InGaN and AlGaN layers. All tested volumes passed this χ² test, confirming the random alloy nature of the individual layers of the SL.

CONCLUSIONS

Several SL structures were grown on (1010) m-plane substrates by NH₃-MBE. A series of tensile strained AlN/GaN SL with the number of periods increasing from 5 to 20 had sharp interfaces as observed by APT, SEM, and HRXRD. Further investigation by CL revealed prismatic MDs oriented along the [0001] c-direction on the thinnest 5 period SL sample, and ultimately cracking along the 11_2 plane to incorporate larger fractions of compressively strained InGaN/GaN SL. However, the evidence of cracking on the thinnest AlN/GaN strained SL sample, as well as the low density of prismatic MDs on all strained SL samples indicated that prismatic MDs alone are insufficient to relieve misfit strain in such structures.

By contrast, strain compensated SLs, consisting of periods of compressively strained InGaN wells and AlGaN barriers, performed better. A narrow NH₃-MBE growth window and the ability of the m-plane to incorporate larger fractions of In as compared to the c-plane allowed for a growth regime near 620 °C, which allowed for controlled growth of InGaN and AlGaN without temperature ramping or growth interrupts. HRXRD showed high quality SL fringing equal to that of the optimally grown, strained SL samples, indicating minimal loss of crystal quality; morphology was likewise not compromised. No prismatic MDs were observed by CL on two such 20 period samples, which differed only in the location of Si doping (wells versus barriers). Further development of such prismatic MD free, strain compensated SLs with even greater numbers of periods and of varying thickness and composition are ongoing, and may be of great use to the further development of m-plane optoelectronic devices.

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