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<td><strong>Author(s)</strong></td>
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Strain states of AlN/GaN-stress mitigating layer and their effect on GaN buffer layer grown by ammonia molecular beam epitaxy on 100-mm Si(111)

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The effect of strain states of AlN/GaN-stress mitigating layer (SML) on buried crack density and its subsequent influence on the residual stresses in GaN buffer layers grown using ammonia-molecular beam epitaxy on 100-mm Si(111) substrate has been investigated. Different stages involved in the formation of buried cracks, which are crack initialization, growth of relaxed AlN layer, and subsequent lateral over growth, are identified using in-situ curvature measurements. While the increase of GaN thickness in AlN/GaN-SML enhanced its compressive strain relaxation and resulted in reduced buried crack spacing, the variation of AlN thickness did not show any effect on the crack spacing. Moreover, the decrease in the crack spacing (or increase in the buried crack density) was found to reduce the residual compression in 1st and 2nd GaN layers of AlN/GaN-SML structure. The higher buried crack density relaxed the compressive strain in 1st GaN layer, which further reduced its ability to compensate the tensile stress generated during substrate cool down, and hence resulted in lower residual compressive stress in 2nd GaN layer. © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4822031]

I. INTRODUCTION

GaN growth on Si substrate presents greater advantages in the fields of electronics and optoelectronics. However, a large thermal mismatch of 52% and a lattice mismatch of 17% between GaN and Si lead to high tensile strain generation, which cracks epi-layers during cool down from the growth temperature. AlGaN/GaN high electron mobility transistor (HEMT) structures on crack free GaN layers were demonstrated on 100-mm Si(111) substrate using plasma assisted molecular beam epitaxy (PA-MBE) growth process.1,2 Moreover, Aidam et al.3 have recently demonstrated crack free GaN layers up to 4.2 µm thickness on 100-mm Si(111) by optimizing the V/III flux ratio during the initial stages of PA-MBE growth of GaN layers. Alternatively, the growth of GaN layers by ammonia-MBE offers distinctive advantages such as wider growth window, reproducible Ga-polarity surface, and possibly improved device uniformity. However, the higher growth temperatures employed in ammonia-MBE necessitate the usage of stress mitigating layers (SMLs) to grow crack free thick GaN layers. Several types of SMLs are studied in the literature for the growth of GaN on Si.4–10 In our previous work, we have investigated the effect of AlN/GaN-SML and AlGaN-SML on the structural and morphological properties of GaN on 100-mm Si substrates using ammonia-MBE.11 AlN/GaN-SML was found to be effective in obtaining compressive GaN layers with good crystalline quality due to its higher lattice mismatch induced compression and annihilation of dislocations at the two subsequent AlN/GaN interfaces.

The layer structures grown using AlN/GaN-SML intrinsically contains buried cracks at the interface between AlN and GaN layers in the SML. Bethoux et al.12 have observed the formation of buried cracks in metal organic chemical vapor deposition (MOCVD) grown AlGaN layers and ammonia-MBE grown AlN layers on GaN templates. They explained that the brittle relaxation through crack formation occurs in nitride semiconductors due to the inability of forming misfit dislocation by Mathew-Blakeslee mechanism.13 MOCVD grown AlGaN layers with good structural quality were demonstrated by the method of overgrowth of cracked AlGaN layers. In the structures grown using AlN/ GaN-SML, cracks are formed to relax the tensile strain in AlN layer and are overgrown to produce smooth surface morphology. The overgrown AlN layer acts as a buffer layer and induce high compression in the final top GaN layer, which helps in overcoming the tensile strain during the cool down.8,9 The buried cracks in the structure are accompanied by the presence of voids in the GaN layer of AlN/GaN-SML. Tang et al.14 have attributed high temperature growth of AlN layer for the development of voids in the GaN layer. Furthermore, they have accounted buried cracks in AlN/ GaN-SML structure to the stress compensation mechanism during the cool down. Thus, the buried cracks play an important role in GaN heterostructures and determine the strain states and structural quality of the epilayers on Si substrate. However, their variation in density and their effect on overall strain states have not been reported. In this work, we have studied the formation of buried cracks in detail and reported effect of strain states of AlN/GaN-SML on the density of buried cracks and their consequent effect on the residual stress in GaN buffer layers, grown using ammonia-MBE on 100-mm Si(111) substrate.
II. EXPERIMENTAL PROCEDURE

A typical epilayer structure with AlN/GaN-SML, grown using ammonia-MBE, on 100-nm Si(111) substrate is shown in Fig. 1. Growth temperatures of AlN and GaN epilayers were maintained at 920 and 800 °C, respectively. AlN and GaN epilayers were grown at a rate of 0.13 µm/h and 0.65 µm/h, respectively. The complete details of the growth process can be found elsewhere.9

To understand the variation in strain states of AlN/GaN-SML and their effect on the density of buried cracks, five structures were grown with varied thicknesses of 2nd AlN/1st GaN layers. The thickness of 1st AlN and 2nd GaN were kept constant at 50 and 500 nm, respectively, for all the samples. Sample A was grown with the thinner (2nd AlN/1st GaN) layer combination of 150/120 nm and sample E was grown with the thicker layer combination of 370/470 nm. Samples B, C, and D were grown with different 1st GaN thicknesses of 120, 250, and 350 nm, respectively, while the thickness of 2nd AlN layer was kept constant at 250 nm. Strain states of epilayers were determined in-situ by laser-reflection based curvature measurements and ex-situ by confocal micro-Raman spectroscopy, equipped with a thermally cooled charge-coupled device detector. For Raman measurements, a green laser with 532 nm wavelength was used as an excitation source and a 100× objective (Numerical Aperture: 0.9) was used to focus the sample. A typical spatial resolution of <350 nm was achieved using the confocal configuration of microscope. Surface morphology and the buried cracks in the structure were observed using an optical microscope operating in differential interference contrast (DIC) mode. The cross sectional transmission electron microscopy (TEM) was performed in the bright field mode along (1120) zone axis on selected samples to observe the voids and dislocations in the grown structures.

III. RESULTS AND DISCUSSION

The streaky reflection high energy electron diffraction (RHEED) patterns observed during the growth of different epilayers in samples A to E indicated that the growths proceeded in two-dimensional mode for all the samples. In-situ curvature data obtained during the growth of a typical structure with AlN/GaN-SML (with thicknesses of 250/250 nm) are shown in Fig. 2.

![FIG. 1. AlN/GaN-SML structure with 500 nm thick 2nd GaN layer and 50 nm thick 1st AlN layer.](image)

![FIG. 2. In-situ curvature plot of a typical AlN/GaN-SML structure with 250/250 nm 2nd AlN/1st GaN layers as a function of growth time. Positive and negative slopes of the curvature represent tensile and compressive strains, respectively.](image)

The slope of the curvature becomes positive during AlN growth and negative during GaN growth, indicative of tensile and compressive stresses, respectively. Further, the increment in curvature is found to be steeper for 2nd GaN compared to 1st GaN layer indicative of its higher residual stress and slower relaxation rate. Raman measurements were also performed on samples A to E at room temperature to measure the strain states of 2nd GaN layer by observing the shift in GaN-E2 phonon mode. The focus of confocal microscopy in Raman measurements was adjusted on to the sample surface (2nd GaN layer) to collect the signal. As the peak of phonon modes in Raman measurements follows the Lorentzian profile, the exact peak positions of E2 modes were determined by Lorentzian fit. Further, the stresses in GaN layers were estimated using the relation between biaxial stress and Raman shift: $\Delta \omega = 4.3 \sigma_{xx} cm^{-1} GPa^{-1}$. Table I lists the Raman shift in E2 peak position and the corresponding stress for samples A to E. A red shift in E2 peak position was observed from samples A to E with decreased compression in 2nd GaN layer.

### A. Density of buried cracks in AlN/GaN SML

Surface morphology of samples A to E, observed using an optical microscope, presented crack free surface for all

<table>
<thead>
<tr>
<th>Sample</th>
<th>2nd AlN/1st GaN thicknesses (nm)</th>
<th>Raman shift in E2 peak (cm⁻¹)</th>
<th>Compressive stress in 2nd GaN (MPa)</th>
<th>Average crack spacing of buried cracks (µm)</th>
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<tr>
<td>A</td>
<td>150/120</td>
<td>569.14</td>
<td>498</td>
<td>1.55</td>
</tr>
<tr>
<td>B</td>
<td>250/120</td>
<td>568.93</td>
<td>449</td>
<td>1.58</td>
</tr>
<tr>
<td>C</td>
<td>250/250</td>
<td>568.43</td>
<td>332</td>
<td>1.37</td>
</tr>
<tr>
<td>D</td>
<td>250/350</td>
<td>568.00</td>
<td>233</td>
<td>1.25</td>
</tr>
<tr>
<td>E</td>
<td>370/470</td>
<td>567.06</td>
<td>014</td>
<td>1.04</td>
</tr>
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</table>
the samples except E. The cracking of sample E coincides with the observation of nearly relaxed 2nd GaN in Raman measurements, where the tensile stain generated in GaN layer might have relaxed by cracking. Observation of buried cracks in samples requires shifting the focus of the microscope from the sample surface to the interface of 2nd AlN and 1st GaN layers. Figures 3(a) and 3(b) shows the buried crack features of samples A and E, respectively. As can be seen, the buried cracks are oriented in several crystallographic directions, and the crack density is higher in sample E compared to sample A. Quantification of the buried crack density is difficult as the cracks are oriented in different crystallographic directions. However, a numerical representation of the buried crack density was obtained by taking the statistical average of crack spacing between two parallel cracks in different directions from the microscope image. Table I lists the average spacing of buried cracks for samples A to E.

From Table I, samples B, C, and D show that the average crack spacing of buried cracks decreases with the increase of 1st GaN layer thickness, while the thickness of 2nd AlN layer was kept constant at 250 nm. However, for a constant 1st GaN layer thickness of 120 nm, the crack spacing was found to be almost independent of the thickness of 2nd AlN when its thickness was varied from 150 to 250 nm (samples A and B). These observations suggest that the density of buried cracks respond more to the thickness of 1st GaN layer than to the thickness of 2nd AlN layer. Buried cracks are formed at the interface between 2nd AlN and 1st GaN layers to relax the lattice mismatch induced strain during the growth. In-situ stress measurements offer critical details of the strain states of the epilayer during the growth. Hence, the formation of buried cracks and their behavior was observed using these measurements. Figure 4 shows a close-up look of the in-situ curvature data during the growth of 2nd AlN/1st GaN layers with thicknesses of 250/250 nm. As can be seen, the curvature data are divided into 6 segments from point 1 to 7. The segment between “1” to “2” indicates the in-situ wafer curvature during the growth of 1st AlN layer. The segments between “2” and “4” and “4” and “7” indicate the curvature during the growth of 1st GaN and 2nd AlN layers, respectively. The overall in-situ curvature measured is a result of stresses induced and simultaneous relaxation processes during the growth. Moreover, the slope of the curvature becomes negative from point “2” to “3” of GaN growth, indicating the development of net compressive stress. However, the curvature changes its sign, and its slope becomes positive at point “3”, signifying that the relaxation process overtakes the compression at this point. Based on the curvature data obtained from repeated growths, the typical thickness found for the relaxation is around 110–130 nm.

The relaxation of GaN layer occurs through dislocation bending and looping as described by Cantu et al.\textsuperscript{17} and modeled by Romanov and Speck.\textsuperscript{18} Plastic relaxation ($\bar{\varepsilon}_{pl}$) through dislocation bending and looping follows Eq. (1), where $b$ is the magnitude of the burger vector, $\rho_{TD}$ is the threading dislocation density, $l$ is the layer thickness of dislocation looping, and $x$ is the inclination angle

$$
\bar{\varepsilon}_{pl} = \frac{1}{4} b \rho_{TD} h \tan x.
$$

In our previous study on GaN grown using AlGaN-SML, it was observed that the compressive stress relaxation of GaN grown in 2D growth mode was mainly due to the dislocation looping mechanism.\textsuperscript{19} Similarly, as shown in Fig. 2, the steeper increment in the curvature of 2nd GaN layer with thickness can be attributed to its lower compressive stress relaxation due to the lesser dislocation density and their consequent annihilation compared to 1st GaN layer. To understand the relaxation process of 1st GaN layer, the dislocation behavior in the 1st GaN layer was observed using cross-sectional TEM images of Fig. 5. As indicated by the arrow marks in Fig. 5, clear bending and looping of dislocations occur in samples C and E. However, sample A showed relatively lesser looping as most of the dislocations appeared to be travelling to the top layer. The layer thickness of dislocation looping, $h$, is found to be around 110 nm, which is well matched with the critical thickness of relaxation from the curvature data of Fig. 4. Hence, the sudden relaxation of 1st GaN layer in the curvature measurements can be attributed to the intense bending and looping of dislocations around this thickness. However, as can be seen Figs. 5(b) and 5(c), dislocation activity continues in the 1st GaN layer throughout its thickness to further relax the residual compressive strain. Sahonta et al.\textsuperscript{20} have also observed similar behavior of compressive stress relaxation in GaN layers with

FIG. 3. Microscopic images of the buried cracks of sample A (a) and sample E (b). Sample E with the thicker 1st GaN/2nd AlN layer combination showed higher crack density compared to sample A with thinner layer combination.

FIG. 4. In-situ curvature plot of 1st GaN and 2nd AlN layers with the thicknesses of 250 nm as a function of growth time. The inset shows the TEM image of a typical buried crack and its lateral growth phenomenon.
increased thickness and attributed it to dislocation bending and looping mechanism. The increase in relaxation of 1st GaN layer increases its lattice mismatch with 2nd AlN, leading to higher tensile strain generation. The higher tensile strain in AlN layer further favors the formation of high density of cracks to relax the strain. Thus, the observed trend of decreased crack spacing with the increased 1st GaN thickness in samples B to D can be attributed to the enhanced relaxation of GaN layer with thickness. Moreover, the highest crack spacing observed in sample B with 1st GaN thickness of 120 nm might be due to its partially relaxed GaN layer, resulted from the lesser dislocation looping.

The curvature data also provide an insight into the effect of 2nd AlN layer on the density of buried cracks. As shown in Fig. 4, the positive slope of curvature at point “4” signifies the growth of tensile strained 2nd AlN on 1st GaN layer. The curvature subsequently becomes flat at point “5” and saturates with a slight negative slope towards point “6,” indicating the relaxation through the formation of cracks and growth of relaxed AlN layer, respectively. Here, the negative slope observed from point 5 to 6 might be due to the induced compression in the underlying GaN layer, which is explained in detail in Sec. III.C. After the initial relaxation through cracking, development of tensile strain is observed at point “6” of AlN layer growth. As shown in the TEM image for the sample C of Fig. 4, point “6” corresponds to the AlN thickness of ~70 nm, where the lateral overgrowth of cracked regions initiates. The process of lateral overgrowth might have resulted in tensile strain generation in AlN layer. Thus, from the in-situ curvature measurements, different stages of 2nd AlN growth such as the initial tensile strain generation followed by the relaxation through buried crack formation and the lateral overgrowth can be observed. Furthermore, it can also be seen that the relaxation of AlN through crack formation is not observed beyond point “5.” This signifies that the cracking and formation of buried cracks is not observed anymore during 2nd AlN layer growth up to 250 nm of its thickness. This justifies the observation of similar crack spacing for samples A and B with AlN layer thickness of 150 and 250 nm, respectively. Thus, the density of buried cracks is found to be independent of 2nd AlN layer thickness (up to 250 nm) and depends primarily on the relaxation of 1st GaN layer and hence on its epilayer thickness.

B. Density and size of voids in AlN/GaN SML

AlN/GaN-SML structure contains voids in 1st GaN layer along with the buried cracks in the structure as shown in the cross-sectional TEM images of Fig. 5. Cracks in the 2nd AlN layer propagates to the 1st GaN layer due to the strain field that develops at the crack edge of 2nd AlN layer.12 Tang et al.14 have reported that the formation of voids in GaN layer occurs due to the decomposition of GaN in cracked regions and diffusion along the crack opening at high growth temperature of AlN. As shown in Fig. 5, no voids were observed in TEM image of sample A with thinner AlN/GaN layers, indicating very low void density. However, samples C and E clearly showed the presence of voids with their density and size increasing from sample C to E. The increased growth time of 2nd AlN layer from sample A to C to E led to the continuous decomposition of GaN in the cracked regions, which enhanced the probability of void formation and increased the size of the existing voids. The material decomposed during this process fills in the buried cracks of 2nd AlN layer and the upper portion of voids in the 1st GaN layer. Bethoux et al.12 have observed GaN domains in the buried crack regions by energy dispersive X-ray (EDX) analysis, which confirms the mass transport. Further, they have also reported that the depth of the crack in GaN layer, over which AlGaN layer was grown, increases as its thickness is increased. The increased crack depth in GaN might have also exposed more cracked area to higher temperature during the subsequent AlN growth leading to higher decomposition and increased void dimensions. Thus, the thicknesses of both 2nd AlN as well as 1st GaN layers might play a critical role in the variation of density and size of voids in AlN/GaN-SML structure.

C. Effect of buried cracks on the residual stress in GaN buffer layers

The relaxation of 2nd AlN layer due to cracking can be visualized from the in-situ curvature measurements. However, the curvature measurement does not show the effect of cracking on the strain states of 1st GaN layer. Two samples namely F and G, shown as insets in Fig. 6, were grown to study the effect of cracking on 1st GaN layer. Sample G with the growth sequence of 2nd AlN/1st GaN/1st AlN showed the presence of buried cracks in the microscope image, whereas sample F with 1st GaN/1st AlN was found to be completely crack free (microscope images are not presented here). Fig. 6 shows the normalized GaN-E2 phonon peaks obtained from both the samples. A high spatial
resolution of <0.35 μm of the confocal microscope allowed us to obtain Raman spectra from the close vicinity of the crack and from the center of the two parallel cracks of sample G, as represented in Fig. 6 as G-1 and G-2, respectively. The solid vertical line at 567 cm\(^{-1}\) in the Raman spectra indicates the \(E_2\) position of free standing GaN.

The red shift in GaN \(E_2\) peak of sample F with respect to the free standing GaN \(E_2\) peak indicates its tensile nature, whereas the blue shift of GaN \(E_2\) (G-2) peak of sample G indicates its compressive nature. This observation clearly suggests that the growth of 2nd AlN layer induces compression in 1st GaN layer, changing its strain state from tensile to compression. Further, the slight negative slope of curvature observed during the growth of the relaxed AlN layer from point 5 to 6 in Fig. 4 can be attributed to the induced compression in 1st GaN layer by the growth of 2nd AlN layer. Steude \textit{et al.} have also observed similar behavior, where the growth of AlGaN layers was found to induce compression in the bottom GaN layer.\textsuperscript{23} Moreover, the separation of GaN \(E_2\) peaks at positions G-1 and G-2 in Fig. 6 shows that the formation of buried cracks partially relaxes the induced compression in GaN layer. Thus, the growth of 2nd AlN layer induces compression in the 1st GaN layer, which gets partially relaxed through crack formation at the interface.

The variation in the crack spacing of samples A to E (Table I) indicates different compression states of the 1st GaN layer. To determine the effect of strain states of the 1st GaN layer on the 2nd GaN layer, Raman measurements were performed on the 1st and 2nd GaN layers of samples A to E. The focus of the confocal microscope was adjusted to the sample surface and to the surface of the 1st GaN layer to obtain the spectra from the 2nd and 1st GaN layers, respectively. In the case of 1st GaN layer, Raman spectrum was collected from the center of the two parallel cracks to determine its average compression. A typical plot of normalized GaN \(E_2\) peaks of 1st and 2nd GaN layers obtained from sample E is shown in Fig. 7. The separation of GaN \(E_2\) peak positions indicates the different strain states of these two layers.

Stresses obtained from Raman measurements of GaN layers in samples A to E are plotted as a function of buried crack spacing in Fig. 8. It can be seen that the residual compression in the 1st and 2nd GaN layers decreases with the decrease of the buried crack spacing. As discussed, relaxation of the 1st GaN layer through crack formation was observed in Raman measurements on sample G at positions G-1 and G-2 as shown in Fig. 6. Hence, the decrease in the residual compression in 1st GaN with the decrease of crack spacing can be attributed to the increased relaxation through cracking. However, the formation of buried cracks at the interface of 1st GaN/2nd AlN layer may not directly affect the strain states of the 2nd GaN layer. Using variable temperature high resolution X-ray diffraction measurements, Tang \textit{et al.} have shown that AlN/GaN-SML compensates the thermal strain during cool down and attributed it to the buried cracks in the structure.\textsuperscript{14} But our observation from Fig. 8 indicates that the increase in the buried crack density is found to decrease the residual compression of the 2nd GaN layer. Moreover, a trend of decreased compression of 2nd GaN layer with the decrease in compression of 1st GaN layer is observed.

These two observations suggest that the stress compensation observed in AlN/GaN-SML structure during cool
down may be related to the compressive nature of 1st GaN layer. The compressive stress in 1st GaN acts as a compensating mechanism for the tensile strain during cool down and reduces the thermal mismatch strain transferred from Si to 2nd GaN layer. Hence, the increase in the buried crack density increases the relaxation of 1st GaN layer, which reduces its stress compensation ability leading to lower compression of 2nd GaN layer. Thus, the buried cracks are found to affect the strain states of 1st GaN layer, which further affects the strain states of 2nd GaN layer.

IV. CONCLUSION

Buried crack formation in AlN/GaN-SML structures and their density dependence on strain states of 2nd AlN/1st GaN layers was studied using in-situ curvature measurement technique during the ammonia-MBE growth of GaN layers on 100-mm Si(111) substrate. Further, the effect of density of buried cracks on the strain states of 1st and 2nd GaN layers was also investigated. Using in-situ curvature measurements, different stages of buried crack formation such as crack initialization, the growth of strain free AlN layer followed by lateral overgrowth of the cracked AlN layer was clearly identified. The density of the buried cracks was found to depend upon the relaxation of 1st GaN layer prior to the growth of 2nd AlN layer. Increased thickness of 1st GaN layer enhanced its relaxation and has led to higher buried crack density in order to relax the tensile strain in AlN layer. In addition, the in-situ curvature measurements showed that the density of buried cracks is independent of the thickness of 2nd AlN layer up to a thickness of 250 nm. However, the increased thickness of 2nd AlN layer increased the density and size of voids in 1st GaN layer. Higher buried crack density was found to reduce the residual compression in the 1st and 2nd GaN layers. The increased relaxation through the enhanced buried crack formation is found to be the reason for the reduction of compression in the 1st GaN layer. Compression in the 1st GaN layer acts as a stress compensating mechanism for the tensile stress generated during cool down and hence reduces the residual compression in the 2nd GaN layer with reduction in compression of 1st GaN. Thus, the strain states of GaN layer during the growth of AlN/GaN-SML influence the density of buried cracks, which further affect the residual strain in GaN buffer layers. These results point out that AlN/GaN-SML not only helps in inducing higher compression during the growth of GaN buffer layer but also compensates the thermal strain generated due to GaN heteroepitaxy on Si during cool down, where the compressive strain in 1st GaN layer acts as compensation mechanism for the thermal strain.

ACKNOWLEDGMENTS

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