Epitaxial growth and characterization of non-polar $a$-plane AlGaN films with MgN/AlGaN insertion layers

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Abstract

The MgN/AlGaN insertion layers were applied for the first time in the growth of non-polar $a$-plane AlGaN epi-layers by metal organic chemical vapor deposition technology. The full-width-at-half-maximum value of X-ray rocking curve for the $a$-plane AlGaN epi-layer was decreased by approximately 50.6% and the root-mean-square value of the surface was reduced by 74% by inserting the MgN/AlGaN insertion layers with optimized number of insertion pairs, which revealed that the compressive strain within the $a$-plane AlGaN epi-layers was effectively reduced, leading to significant improvements in crystalline quality and surface morphology, which is very helpful to fabricate high quality AlGaN-based ultraviolet light-emitting-diodes.

Keywords: non-polar $a$-plane AlGaN; MgN/AlGaN insertion layers; crystalline quality; surface morphology.
1. Introduction

AlGaN-based ultraviolet light emitting diodes (UV-LEDs) with emission wavelength between 210 and 365 nm are in demand for a wide range of applications, such as sterilization, water/air purification, and medical treatment.\(^1\) \(^2\) Currently, most of the AlGaN-based UV-LEDs have been fabricated based on polar (0001)-oriented \(c\)-plane AlGaN epi-layers because there is less difficulty to grow relatively high quality AlGaN epi-layers on polar \(c\)-plane sapphire substrate than on other orientation sapphire substrates. However, it is well known that the light emission efficiency of the polar \(c\)-plane AlGaN-based UV-LEDs can be strongly reduced by the quantum confined Stark effect (QCSE) induced by the spontaneous and piezoelectric polarization fields along [0001] \(c\)-direction.\(^3\) In contrast, there is no spontaneous polarization-induced built-in electric field in a non-polar AlGaN epi-layer and the QCSE is completely suppressed in non-polar AlGaN-based multiple quantum wells (MQWs).\(^4\) Hence, high light emission efficiency can be expected for the non-polar AlGaN-based UV-LED. Unfortunately, the hetero-epitaxial growth of non-polar (11\(\bar{2}\)0)-oriented \(a\)-plane AlGaN films on semi-polar (1\(\bar{1}\)02)-oriented \(r\)-plane sapphire substrates has been proven to be a very challengeable task. Because of the large mismatch both in lattice constant and thermal expansion coefficient between the \(r\)-plane sapphire substrate and the non-polar AlGaN epi-layer, high density of the misfit dislocations, undulating surface morphology, and strong crystallographic anisotropy are usually generated for the non-polar AlGaN epi-layers during the epitaxial growth process. Therefore, the epitaxial growth of high quality non-polar AlGaN films plays a crucial role in the fabrication of AlGaN-based UV-LEDs.

In order to improve the crystalline quality of the non-polar GaN epi-layers, various kinds of intermediate layers\(^5\)\(^-\)\(^8\) and patterned sapphire substrate (PSS) with different patterns\(^9\)\(^-\)\(^12\) were applied for the epitaxial growth of the non-polar GaN films. However, up to date there were only few reports on the approaches to improve the crystalline quality and the surface morphology for the non-polar AlGaN epi-layers. Even though the first attempt to grow the
non-polar \( a \)-plane AlGaN films under the two-dimensional growth mode was made in 2003,\(^{13}\) it is still indispensable to further improve the crystalline quality and the surface morphology of non-polar \( a \)-plane AlGaN epi-layers, especially those with high Al composition (> 0.3) for meeting the needs to fabricate deep UV-LEDs with wavelength less than 320 nm.

In this study, the MgN/AlGaN insertion layers were applied for the first time in the epitaxial growth of non-polar \( a \)-plane AlGaN films on semi-polar \( r \)-plane sapphire substrates by metal organic chemical vapor deposition (MOCVD) technology. X-ray diffraction (XRD), transmission electron microscopy (TEM), Raman spectroscopy, photoluminescence (PL) spectroscopy, and atomic force microscopy (AFM) were employed to characterize the structural and optical properties of the grown non-polar \( a \)-plane AlGaN epi-layers. The characterization results demonstrate that high crystalline quality non-polar \( a \)-plane AlGaN epi-layers with low dislocations density, smooth surface morphology, low compressive strain, and strong PL intensity have been achieved with the optimized MgN/AlGaN insertion layers.

2. Experimental

![Fig. 1 The schematic layer structures for the non-polar (11\( \bar{2} \)0)-oriented \( a \)-plane AlGaN samples grown with various number (\( n = 0, 1, 2, 3, 4 \)) of pairs of MgN/AlGaN insertion layers.](image)

All the non-polar \( a \)-plane AlGaN epi-layer samples used in this study were grown on semi-polar \( r \)-plane sapphire substrates in a low pressure (40 Torr) MOCVD system.
Trimethyl-aluminum (TMAI), trimethyl-gallium (TMGa), bis(cyclopentadienyl)-magnesium (CP2Mg), and ammonia (NH3) were used as the precursors for Al, Ga, Mg, and N, respectively, and hydrogen (H2) was used as the carrier gas. Similar to the epitaxial growth process described in detail in our recently published paper,14) for the epitaxial growth of the non-polar $a$-plane AlGaN film without MgN/AlGaN insertion layer which was named as sample A, the semi-polar $r$-plane sapphire substrates were firstly heated up to 1,060 °C in H2 ambience to remove the surface contamination. A 20 nm-thick low-temperature (LT)-AlN nucleation layer was then deposited on the semi-polar $r$-plane sapphire substrate at a lower temperature of 600 °C. Subsequently, the temperature was raised to 1,100 °C to grow a high-temperature (HT)-AlN buffer layer. Finally, an 800 nm-thick non-polar $a$-plane AlGaN epi-layer was grown on the HT-AlN buffer layer. The epitaxial growth processes and the layer structures for samples B, C, D, and E are basically the same as that for sample A with the exception that 1, 2, 3, and 4 pairs of MgN/AlGaN composite layers were inserted in between the HT-AlN buffer layer and the top AlGaN layer, respectively, as schematically shown in Fig. 1. For the purpose of comparison, the thickness for one pair of the MgN/AlGaN composite layers was fixed at 250 nm throughout this study, and the averaged layer thickness of the MgN film was estimated to be less than 10 nm.

The crystal orientation and the Al composition for the grown non-polar $a$-plane AlGaN epi-layers were determined by means of high resolution (HR)-XRD 2θ-ω scans. In specific, the X-ray rocking curves (XRCs) were measured in two directions that are perpendicular to each other for evaluating the crystalline quality for the non-polar $a$-plane AlGaN epi-layers. The blocking of the threading dislocations (TDs) by the MgN/AlGaN insertion layers was confirmed with TEM measurement. Moreover, the surface morphology for the non-polar $a$-plane AlGaN epi-layers was characterized by AFM measurement. The structural and optical properties for the grown non-polar $a$-plane AlGaN epi-layers were further investigated with Raman and PL spectroscopies conducted at room temperature (RT).

3. Results and discussions
Fig. 2 The HR-XRD $2\theta$-\(\omega\) scanning curve for the non-polar \(a\)-plane AlGaN epi-layer sample A (a), the XRC FWHM values as a function of the number of pairs of the MgN/AlGaN insertion layers (b).

The HR-XRD $2\theta$-\(\omega\) scanning curve for sample A was shown in Fig. 2(a). It was demonstrated unambiguously that the peaks located at $2\theta=52.56^\circ$, $58.40^\circ$ and $59.35^\circ$ were the XRD diffraction peaks from the semi-polar (2204)-oriented \(r\)-plane sapphire substrate, the non-polar (1120)-oriented \(a\)-plane AlGaN and AlN epi-layers, respectively.$^{15, 16}$ Extremely similar results were observed for samples B, C, D, and E although they are not shown in Fig. 2(a). Moreover, the Al compositions were determined to be 0.40 for all the five samples A-E by fitting the XRD spectra with an analysis software called “PANalytical X’Pert Epitaxy”. Considering the structural anisotropy of the non-polar AlGaN epi-layers, the measurements of the XRCs were performed at azimuth angles of $\varphi=0^\circ$ parallel to the [0001] direction and $\varphi=90^\circ$ parallel to the [1100] direction, respectively to characterize the crystalline quality of the grown non-polar \(a\)-plane AlGaN epi-layers. The full width at half maximum (FWHM) values of the XRCs for the five non-polar \(a\)-plane AlGaN epi-layer samples A-E that have varied number of pairs of the MgN/AlGaN insertion layers were depicted in Fig. 2(b). It was evident that the FWHM values of the XRCs measured along both the [0001] and the [1100] directions decreased remarkably with the increase in the number of pairs of MgN/AlGaN insertion layers, indicating a significant improvement in crystalline quality for the non-polar \(a\)-plane AlGaN epi-layer samples that were inserted with
MgN/AlGaN composite layers. In fact, the FWHM values of the XRCs for sample D with three pairs of the MgN/AlGaN insertion layers were found to be 49.1% less along the [0001] direction and 50.6% less along the [1100] direction, respectively than those for sample A without any MgN/AlGaN insertion layers. However, as shown clearly in Fig. 2(b), the FWHM value of the XRC decreased slowly and tended to saturate when the number of pairs of the MgN/AlGaN insertion layers increased further from three for sample D to four for sample E.

![Fig. 3](image_url)

Fig. 3 The growth models of the non-polar (1120)-oriented a-plane AlGaN epi-layer samples with one pair (a) and two pairs (b) of the MgN/AlGaN insertion layers as well as the bright field image of cross-sectional TEM for sample D (c).
The growth models for the non-polar $a$-plane AlGaN epi-layer samples with one and two pairs of MgN/AlGaN insertion layers as well as the bright field image of the cross-sectional TEM for sample D with three pairs of the MgN/AlGaN insertion layers were demonstrated in Figs. 3(a), 3(b), and 3(c), respectively. It was inferred that the improvement in crystalline quality could be attributed to the blocking of the TDs formed during the growth process of the LT-AlN nucleation layer and at the initial growth stage of the HT-AlN buffer layer by the extra hetero.Interfaces associated with the MgN/AlGaN insertion layers.18,19) As shown schematically in Fig. 3(a), the TDs in the non-polar $a$-plane AlGaN epi-layer sample with one pair of MgN/AlGaN insertion layers are partially blocked and the defect-free areas with less TDs are created. Moreover, as illustrated in Fig. 3(b), the TDs are further blocked and the defect-free areas are increased markedly in the non-polar $a$-plane AlGaN epi-layer sample with two pairs of MgN/AlGaN insertion layers. On the other hand, as demonstrated clearly by the TEM measurement results shown in Fig. 3(c), the TDs in sample D were indeed effectively blocked or merged with the introduction of the MgN/AlGaN insertion layers, reaching the surface in the form of bundles. As a result, large defect-free areas were observed in the TEM image shown in Fig. 3(c) for sample D, indicating a relatively good coincidence with the models shown in Figs. 3(a) and 3(b).

Fig. 4 The RT Raman spectra for the five non-polar $a$-plane AlGaN epi-layer samples A-E measured under the backscattering configuration of $x(−, −)x$ (a), the phonon frequency and the FWHM value of GaN-like $E_2$ (high) mode-related transition for the non-polar $a$-plane.
AlGaN epi-layer samples A-E as a function of the number of pairs of the MgN/AlGaN insertion layers (b).

Figure 4(a) shows the RT Raman spectra for the non-polar $a$-plane AlGaN epi-layer samples A-E measured under the backscattering configuration of $x(\overline{1}-,\overline{1})\bar{x}$ with particular attentions paid on the behavior of GaN-like $E_2$ (high) mode. It was well known that under such scattering configurations, $E_2$ phonon transition related to the strain- and crystalline quality was allowed. Furthermore, it was found that $E_2$ (high) modes for the non-polar AlGaN samples included the features of both GaN-like and AlN-like modes. As shown in Fig. 4(a), two peaks located at 418 cm$^{-1}$ and 646.1 cm$^{-1}$, respectively, appeared in the Raman spectra for all the samples, corresponding to the $A_{1g}$ mode for the semi-polar $(11\bar{2}0)$-oriented $r$-plane sapphire substrates and the AlN-like $E_2$ (high) mode for the non-polar $(11\bar{2}0)$-oriented $a$-plane AlGaN epi-layers, respectively. The phonon frequencies and the FWHM values of GaN-like $E_2$ (high) mode for samples A-E were plotted in Fig. 4(b) to analyze the effects of the MgN/AlGaN insertion layers on the characteristics of GaN-like $E_2$(high) mode. It is apparent that as the number of the inserted MgN/AlGaN pairs was increased from 0 to 4, the GaN-like $E_2$ (high) phonon peak was red-shifted monotonously from 592.0 to 579.8 cm$^{-1}$. Since the GaN-like $E_2$ (high) phonon peak for the unstrained $Al_{0.40}Ga_{0.60}N$ film was known to be located at 579.8 cm$^{-1}$, it is reasonable to treat this value as the critical point value between compressive strain and tensile strain. In other words, lower or higher values than the critical point value implies the existence of a tensile strain or a compressive strain within the epitaxial layers, and the frequency shift from the critical point is proportional to the magnitude of the strain. Therefore, it could be concluded that samples A-E were all suffering from a compressive strain. This compressive strain, however, decreased monotonously from sample A to sample E, indicating an effective relaxation of the compressive strain within the non-polar $a$-plane AlGaN epi-layers with the increase in the number of pairs of MgN/AlGaN insertion layers from 0 to 4. Moreover, as shown in Fig. 4(b), the FWHM values of the GaN-like $E_2$ (high) mode-related peaks decreased evidently from sample A to sample E. Because the FWHM value reflects the crystalline quality, the
significant reduction in the FWHM value indicates an improvement in crystalline quality of the grown non-polar $a$-plane AlGaN epi-layers with the insertion of the MgN/AlGaN composite layers.\textsuperscript{25)} As mentioned above, the reduction in the dislocation density could be ascribed to the MgN/AlGaN insertion layers-induced enhancement in the opportunity for the dislocations to annihilate, resulting in the improvement in crystalline quality.\textsuperscript{26)} In particular, as shown in Fig. 4(a), the GaN-like $E_2$ (high) peak for sample E was located at 579.8 cm$^{-1}$ which was consistent with the critical point value of 579.8 cm$^{-1}$. This fact implies that the compressive strain within sample E is nearly completely compensated, or sample E has the lowest dislocation density among the five non-polar $a$-plane AlGaN epi-layer samples A-E. In addition, it was found that both the phonon frequency and the FWHM value of the GaN-like $E_2$ (high) mode decreased slowly and tended to saturate as the number of pairs of the MgN/AlGaN insertion layers increased further from three for sample D to four for sample E.

Fig. 5 The PL spectra for the non-polar $a$-plane AlGaN epi-layer samples A-E (a), the FWHM values of the PL emission peaks as a function of the number of pairs of the MgN/AlGaN insertion layers (b).

The PL spectra and the FWHM values of the near band edge (NBE) transition peaks for the grown non-polar $a$-plane AlGaN epi-layer samples as a function of the number of pairs of the MgN/AlGaN insertion layers are plotted in Fig. 5. As shown in Fig. 5(a), the NBE transition peaks for samples A-E were slightly red-shifted from 293.5 to 295.0 nm as the number of the inserted MgN/AlGaN pairs was increased from 0 to 4, indicating clearly the
effective relaxation of the compressive strain within the non-polar $\alpha$-plane AlGaN epi-layers.\textsuperscript{27} It was demonstrated clearly in Fig 5(b) that the FWHM value of the PL emission peak decreased monotonously with increasing the number of pairs of the MgN/AlGaN insertion layers from 0 to 4. This result indicates once again that significant improvements in both optical property and crystalline quality could be achieved for the non-polar $\alpha$-plane AlGaN epi-layer samples that were inserted with the MgN/AlGaN composite layers.\textsuperscript{28}

Fig. 6 The AFM images for the non-polar $\alpha$-plane AlGaN epi-layer samples A-E with a detection area of 10×10 μm$^2$ (a-e), the RMS values as a function of the number of pairs of the MgN/AlGaN insertion layers (f).

The three-dimensional (3D) view AFM images and the root mean square (RMS) values for samples A-E are shown in Fig. 6. The RMS values obtained from the AFM measurements for samples A-D decreased gradually as the number of pairs of the MgN/AlGaN insertion layers increased from 0 for sample A to 3 for sample D, whereas the RMS value increased abruptly as the number of pairs of the MgN/AlGaN insertion layers further increased to 4 for
sample E, as shown clearly in Fig. 6(f). This phenomenon demonstrates clearly that the surface morphology for the non-polar $a$-plane AlGaN samples could definitely be improved by inserting the MgN/AlGaN composite layers with an appropriate number of pairs of the MgN/AlGaN insertion layers. Generally, the amelioration in surface morphology could be ascribed to the improvement in both the crystalline quality and the relaxation of the residual compressive strain within the non-polar $a$-plane AlGaN epi-layers.\textsuperscript{29} In fact, the RMS value for sample D with three pairs of the MgN/AlGaN insertion layers was 74\% less than that for sample A without any MgN/AlGaN insertion layers, showing the smoothest surface morphology among all the samples. Nevertheless, the RMS value for sample E with four pairs of MgN/AlGaN insertion layers was much larger than that of sample D, which could be explained in terms of such a theory that the growth mode tends to transform from two-dimensional (2D) to 3D growth mode when the thickness of the insertion layers is increased too much, resulting in a degradation in surface morphology.\textsuperscript{30} On the other hand, the surface morphology for the AlGaN epi-layers could also be degraded with the increase in Mg incorporation into the non-polar $a$-plane AlGaN epi-layers.\textsuperscript{31}

4. Conclusions

The MgN/AlGaN insertion layers were applied for the first time in the epitaxial growth of the non-polar $a$-plane AlGaN films on semi-polar $r$-plane sapphire substrates by MOCVD. The FWHM value of the XRC for the non-polar $a$-plane AlGaN epi-layer sample with three pairs of the MgN/AlGaN insertion layers was reduced to approximately a half of that for the sample without any MgN/AlGaN insertion layers. The smallest RMS value obtained from the AFM measurement was also achieved with the non-polar $a$-plane AlGaN epi-layer sample with three pairs of the MgN/AlGaN insertion layers, which was found to be 74\% less than that for the sample without any MgN/AlGaN insertion layers. Furthermore, the characterization results of Raman spectroscopy showed that the in-plane compressive strain within the non-polar AlGaN epi-layer samples could be compensated effectively by inserting the MgN/AlGaN composite layers with optimized number of insertion pairs. These results
revealed that the defects and misfit dislocations within the grown non-polar $a$-plane AlGaN epi-layers could be significantly reduced with the insertion of the MgN/AlGaN composite layers. The remarkable improvements in optical property, crystalline quality, and surface morphology achieved in this study are very helpful to fabricate high quality non-polar AlGaN-based UV-LEDs.

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