Metalorganic vapor phase epitaxy and characterizations of nearly-lattice-matched AlInN alloys on GaN/sapphire templates and free-standing GaN substrates

Guangyu Liu, Jing Zhang, Xiao-Hang Li, G.S. Huang, Tanya Paskova, Keith R. Evans, Hongping Zhao, Nelson Tansu

1. Introduction

III-nitride based semiconductors are of great importance for various device technologies. Recently, III-nitride semiconductors have been widely employed for energy-efficiency device technologies, including light-emitting diodes (LEDs) for solid state lighting [1–19], visible diode lasers for both display and biosensing [20–26], photovoltaics and solar energy conversion [27–29], and thermoelectric heat conversion and active cooling materials [30–39]. In designing structures in nitride-based devices for photonics and electronics applications, the most-widely studied heterostructures have been primarily limited to AlGaN/GaN and InGaN/GaN configurations. The use of ternary AlInN alloy has attracted significant interest as an alternative to InGaN/GaN and AlGaN in III-nitride based applications for LEDs [40–42] and laser diode (LD) [20–26], solar-blind photodetector [43], short wavelength Distributed Bragg Reflector (DBR) [44,45], and near-infrared optoelectronic devices based on intersubband transition [46–50]. The use of AlInN alloy [51,52] has also been explored for high electronic mobility transistor as the alternative to AlGaN attributing to its larger band offset and reduced lattice mismatch strain. Prior works related to the first theoretical and experimental demonstration of high channel conductivity of AlInN based FETs had also been reported [53–56]. Recently, lattice-matched AlInN alloys have also been reported as a promising material candidate for thermoelectric applications [32–34].

The availability of large bandgap lattice-matched AlInN material is of great importance for optoelectronic, power electronic, and thermoelectric applications. The availability of large bandgap AlInN material lattice-matched to GaN will also be useful as cladding layer in laser structure. Specifically, AlInN material with In-content ~17% is of great interest for achieving lattice matching condition to GaN substrate/template, which in turn leads to elimination of lattice-mismatch strain. The lattice-matching condition of AlInN can also potentially lead to a reduction in threading dislocation and cracking, as well as the elimination of strain-driven piezoelectric polarization field, in the material.

In comparison to the studies performed for AlGaN and InGaN alloys, the metalorganic vapor phase epitaxy (MOVPE) optimization studies of the AlInN alloy are still relatively lacking. The challenges in the growth of high quality AlInN by MOVPE can be attributed to the large contrast of the optimized growth conditions for AlN and InN alloys. The large immiscibility and
differences in the thermal stability between AlN and InN lead to phase separation and composition inhomogeneities. The optimized growth temperatures of AlN (~1100 °C) and InN (~550 °C) are very distinct for these two binary materials grown by MOVPE [42,57], which leads to further challenges in achieving optimized growth conditions for AlInN alloy [42].

Up to today, the growths of AlInN thin films have been carried out by reactive radio-frequency (RF) magnetron sputtering [58–60], molecular beam epitaxy (MBE) [61–63], and MOVPE [33–35,64–69] on the c-plane sapphire substrates. However, comprehensive studies on the effect of the growth conditions for MOVPE of lattice-matched AlInN thin film have not been performed. In addition, the detailed comparison of the properties of AlInN thin films grown on GaN/sapphire templates and on free-standing GaN substrates has not been extensively studied and compared yet [70–72]. In addition, recent works by MOVPE has also been reported for the growth of AlInN on GaN template on Si substrate [73]. The understanding on the growths and optical properties of AlInN alloy will have important impact on the development of the nitride-based devices including such layers.

In this work, the growths of Al_{1−x}In_{x}N materials with indium-contents (x) ranging from x=0.38% to x=25.3% were carried out by employing MOVPE. Two different types of templates were employed, as follow: 1) MOVPE-grown 3 μm-thick unintentionally-doped GaN (u-GaN) templates grown on (0001) sapphire substrates with a dislocation density in the order of ~1 × 10^8 cm^−2, and 2) 470 μm free-standing GaN native substrates grown by hydride vapor phase epitaxy (HVPE) with a typical defect density in the order of 10^6 cm^−2 (provided by Kyma Technologies) [17]. The growth temperature, reactor pressure and V/III ratio were optimized with the goal of obtaining lattice-matched AlInN on GaN templates/substrates. The X-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM) characterizations were carried out to characterize the indium content, material quality, and surface morphology of AlInN thin film for various indium contents.

The organization of this paper is presented as follow. Section 2 discusses about the growth conditions for AlInN alloys by MOVPE. Section 3 focuses on the characterization of the AlInN alloys with various In-contents by XRD. Section 4 focuses on the growth condition optimization studies of AlInN alloy on GaN/sapphire templates, as well as the characterizations of the films by SEM and AFM. In Section 5, the electrical properties of the AlInN thin film grown on GaN/sapphire templates are presented. Section 6 focuses the MOVPE of AlInN thin film grown on GaN substrate, as well as the comparison of the characteristics of the nearly-lattice-matched AlInN thin film growths on GaN substrates and GaN/sapphire templates. The potential applications of AlInN alloy for thermoelectric and high power LEDs applications are also discussed in Section 7.

2. Growth conditions of AlInN alloy with various In-contents

All the AlInN thin films studied here were grown using a vertical-type VEECO P-75 MOVPE reactor. Trimethylindium (TMIn) and trimethylaluminum (TMAI) were used as the group III precursors, and ammonia (NH3) was employed as the group V precursor. Purified N2 was used as carrier gas in the growth of AlInN alloy. In the growth of the AlInN film, the rotation speed of the sample was 1500 rpm.

The AlInN films were grown on top of GaN/sapphire template. The u-GaN/sapphire template and the AlInN thin film growths were carried out separately. For the growth of 3 μm thick GaN templates on the c-plane sapphire substrates, the etch-back and recovery process with 30 nm low temperature buffer layer was employed [16], and the growths of high-temperature u-GaN layers were carried out at a growth temperature of 1075 °C. For the AlInN thin film growth, an additional 210 nm-thick high-temperature u-GaN layer was grown to remove any surface defects prior to the AlInN material growth. To control the In/Al ratio of the AlInN thin film for achieving lattice-matching condition, the growth temperature was decreased from 860 °C to 750 °C resulting in increased In-content in the film, and independently the molar ratio [TMIn]/[TMAI + TMAl] was varied from 0.34 up to 0.64 to further enhance the indium incorporation in the film. After obtaining the growth condition for the AlInN alloy with various compositions, the optimization of the growth conditions for the nearly-lattice-matched alloy was carried out.

3. Characterizations of AlInN alloys with various In-contents

In order to characterize the AlInN alloys, various characterizations were carried out. The crystal quality and In-content of the AlInN epilayers were analyzed using high-resolution X-ray diffraction (HRXRD) using a Philips triple axis diffractometer. The measurements were performed in a triple axis geometry giving a resolution of 36 arcsec at (2θ=30–40°). The scans were performed under ω−2θ (omega−2 theta) method around the (022) reflection of the GaN. The use of scanning electron microscopy (SEM) [Hitachi 4300] was employed to analyze the surface morphology of the samples, and atomic force microscopy [AFM] [Veeco Dimension 3000] was employed for analyzing the surface roughness of the grown film. The root mean square (RMS) surface roughness was measured over a scanning area of 1 μm x 1 μm.

Fig. 1 shows the XRD ω−2θ scans [(0002) direction] of the AlInN alloy grown on GaN/sapphire templates at a growth pressure of 20 Torr for various growth temperatures and [TMIn]/[III] molar ratios. From our studies, by reducing the growth temperature and increasing the [TMIn]/[III] molar ratio, the In-contents in AlInN thin film could be increased from 0.38% up to 21.4%. As shown in Fig. 1, the peaks of the XRD plots for AlInN epitaxial layers with increasing In-content were observed to approach that of GaN. The full width at half maximum (FWHM) of the ω-scans for GaN layers were estimated be in the range of 31–49 arcsec. The nearly-lattice-matched layer was found for growth temperature of 780 °C, [TMIn]/[III] ratio of 0.64, and growth rate of 0.15 μm/h. The angle separation (Δθf) of the AlInN epilayer and GaN peaks of 1044 arcsec was determined for the nearly-lattice-matched layer, which corresponded to In-content of ~16.44%.
The XRD peak intensity and the corresponding FWHM data for the investigated AlInN layers were listed in Table 1. The AlInN thin films with In-content below ~8% were grown with thicknesses exceeding the critical thickness, and the strain would have been released by the formation of dislocations in the material. The determination of the In-contents on cracked films (In-content < 8%) may be less accurate due to the partial relaxation in the films. The cracks in the low In-content AlInN films may result in less accurate compositional measurements in the samples specifically with In-contents of ~0.38% up to ~8%. In contrast, the thicknesses of AlInN films with In-contents ~11%–21% are well maintained below the corresponding critical thicknesses [76], which result in minimal error in the determination of the lattice parameters. The narrow FWHM in the range of ~110–140 arcsec from the optimized pseudomorphically-grown AlInN films indicated the high quality of the AlInN epilayer. The strongest AlInN XRD peak intensity was obtained from that of the nearly-lattice-matched layer with the narrow FWHM of 134 arcsec.

Fig. 2 shows the lattice mismatch ratio of AlInN grown on GaN templates along the c-axis and a-axis as a function of the In-contents. The insert shows the wurtzite crystal structure of nitride material system.

The XRD rocking curve FWHM and intensity for AlInN thin films on GaN/sapphire templates are shown in Table 1. Table 1: XRD rocking curve FWHM and intensity for AlInN thin films on GaN/sapphire templates.

<table>
<thead>
<tr>
<th>In-content (x) of Al_{1-x}In_{x}N</th>
<th>Thickness of Al_{1-x}In_{x}N (μm)</th>
<th>Peak intensity (arb. units)</th>
<th>FWHM (arcsec)</th>
</tr>
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<tr>
<td>0.0038</td>
<td>0.36</td>
<td>7958</td>
<td>81.8</td>
</tr>
<tr>
<td>0.0725</td>
<td>0.21</td>
<td>4198</td>
<td>199</td>
</tr>
<tr>
<td>0.0798</td>
<td>0.2</td>
<td>3291</td>
<td>191</td>
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<tr>
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<td>7191</td>
<td>137</td>
</tr>
<tr>
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<td>0.2</td>
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<td>0.2127</td>
<td>0.2</td>
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<tr>
<td>0.2134</td>
<td>0.2</td>
<td>377</td>
<td>360</td>
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The XRD peak intensity and the corresponding FWHM data for the investigated AlInN layers were listed in Table 1. The AlInN thin films with In-content below ~8% were grown with thicknesses exceeding the critical thickness, and the strain would have been released by the formation of dislocations in the material. The determination of the In-contents on cracked films (In-content < 8%) may be less accurate due to the partial relaxation in the films. The cracks in the low In-content AlInN films may result in less accurate compositional measurements in the samples specifically with In-contents of ~0.38% up to ~8%. In contrast, the thicknesses of AlInN films with In-contents ~11%–21% are well maintained below the corresponding critical thicknesses [76], which result in minimal error in the determination of the lattice parameters. The narrow FWHM in the range of ~110–140 arcsec from the optimized pseudomorphically-grown AlInN films indicated the high quality of the AlInN epilayer. The strongest AlInN XRD peak intensity was obtained from that of the nearly-lattice-matched layer with the narrow FWHM of 134 arcsec.

Fig. 2 shows the lattice mismatch ratio of AlInN grown on GaN templates along the c-axis and a-axis with all the In-contents studied here. The lattice-matching condition corresponds to In-content of 17% in AlInN alloy on GaN [75], thus our growths of AlInN alloys with In-content ~16.44% on GaN template resulted in nearly-lattice-matched thin film. Note that the XRD ω–2θ scans were obtained by Bragg diffraction along (0002) direction, which was measured along the growth direction of the c-axis in wurtzite structure. The lattice constant along the c-axis (c) was calculated from the ω–2θ scans based on the following relation [74]:

$$\frac{c_{GaN} - c_{AlInN}}{c_{GaN}} = -\Delta \cot \theta_{GaN}$$

After the parameter c was obtained, the In-content and lattice constant along the a-axis (a) could be described by the linear interpretation as follow:

$$c_{Al_{1-x}In_{x}N} = x_{D_{InN}} + (1-x)c_{AlN}$$

$$a_{Al_{1-x}In_{x}N} = xa_{AlN} + (1-x)a_{InN}$$

Please note that lattice constant (a) corresponds to the lattice parameter that determines the lattice matching condition in the growth of the c-plane wurtzite semiconductors, specifically AlInN on the c-plane GaN template. The strain field between Al_{1-x}In_{x}N and GaN varied from tensile (x=0.38%) to compressive (x=25.32%) with lattice-matched condition obtained at Δa/a = −0.18%, which corresponded to a nearly-lattice-matched condition (x=16.44%).

To further illustrate the effect of strain field in AlInN alloy on the sample surface morphology, the microscope images of the tensile and nearly-lattice-matched AlInN alloys grown on GaN/sapphire templates were compared in Figs. 3(a) and (b). As shown in Fig. 3(a), the surface morphology of the tensile-strained AlInN with In-content of ~0.38%, which was grown at 860 °C with the thickness of 0.3 μm, is full of cracks from the tensile strain.
driven strain relaxation. In contrast, as shown in Fig. 3(b), the nearly-lattice-matched AlInN alloy with thickness of 0.2 μm shows crack-free layer.

4. Growth optimizations of nearly-lattice-matched AlInN on GaN/sapphire templates

The growth condition optimization for the lattice-matched AlInN thin film was investigated for samples grown on GaN/sapphire template. Specifically, the effects of various growth parameters on the surface morphology of the nearly-lattice-matched AlInN samples were investigated. In our studies, the growth parameters investigated include growth pressure, V/III ratio, and growth rate. The growth pressure was decreased from 100 Torr down to 20 Torr, while maintaining the V/III ratio of 9314. The V/III ratio was increased afterwards from 5205 up to 9314 with a constant growth pressure of 20 Torr.

Fig. 4 shows the effect of the growth pressure on the growth rate and indium incorporation of the AlInN alloys. In this study, the total flow rate was kept the same for the AlInN thin film growths under different growth pressures. The corresponding surface morphology of the AlInN thin film was characterized by SEM measurements, as shown in the insets of Fig. 4. By increasing the growth pressure in the reactor from 20 Torr to 100 Torr, the growth rate of the films was increased from 0.15 μm/h to 0.3 μm/h. The use of higher growth pressure and growth rate also led to a higher indium incorporation in the AlInN thin film. Our results indicated that the indium content in the thin films increases from 16.44% to 25% by increasing the growth pressure from 20 Torr up to 100 Torr. The SEM image of the nearly-lattice-matched AlInN with In-content = 16.44%, which was grown at 20 Torr, showed the smoothest surface morphology. In contrast, the growth of AlInN with In-content = 25% performed at higher growth pressure exhibited a grain-like surface profile.

To further confirm the growth pressure effect, atomic force microscopy (AFM) was carried out to measure the AlInN surface roughness, as shown in Fig. 5. The thicknesses of AlInN thin films studied ranged from 0.2 μm up to 0.26 μm over a scanning area of 1 μm × 1 μm. As the growth pressure was reduced from 100 Torr (50 Torr) to 20 Torr, the root mean square (RMS) surface roughness of AlInN alloys grown on GaN/sapphire template varied from ~5.61 nm (~6.84 nm) to ~1.01 nm. Thus, both AFM and SEM measurement results confirm that the use of low growth pressure leads to a reduction in the surface roughness of the AlInN film.

Fig. 6 shows the effect of V/III ratio on the growth rate and surface morphology of the AlInN thin films. In this particular study, the AlInN layers were grown at 780 °C with constant growth pressure of 20 Torr, and the V/III ratio of the epitaxy was increased from 5205 to 9314. The insets in Fig. 6 show the corresponding SEM images of the surface morphologies for the AlInN films. By increasing the V/III ratios from 5209 to 9314, the growth rate of the films was decreased from 0.28 μm/h to 0.15 μm/h. While the use of higher growth pressure and growth rate was found to lead to higher indium incorporation in the AlInN thin film, this study showed that the indium content in the AlInN thin films decreased as the V/III ratio increased. The smoothest surface morphology was also observed for the nearly-lattice-matched AlInN film with In-content of 16.44%, which was grown with V/III ratio of 9314, as compared to those measured for AlInN films grown with lower V/III ratios.

Fig. 7 shows the AFM measurement results for nearly-lattice-matched AlInN thin film grown at different V/III ratios. The thicknesses of AlInN thin films studied ranged from 0.2 μm up to 0.26 μm with a scanning area of 1 μm × 1 μm. As the V/III ratio was increased from 5205 (7122) to 9314, the root mean square
Table 2 shows the use of high V/III ratio and low growth pressure in the epitaxy of tron mobility, and carrier concentration of Al templates.

Specifically, the Hall measurements are less reliable for the AlInN templates on GaN/sapphire substrate (Kyma Technologies) for comparison purpose. Fig. 8 shows the effect of growth pressure and V/III ratio on the material quality of AlInN thin film on GaN native substrates. In Fig. 8(a), the growth pressure was reduced from 100 Torr (50 Torr) to 20 Torr, which is analogous to the studies discussed in Figs. 4 and 5. The surface roughness of AlInN alloys on GaN substrates decreased from 5.93 nm (6.36 nm) to only 0.89 nm. The insets exhibit the sample surface morphology characterized by SEM measurements. The comparison of the SEM images indicates that the use of low growth pressure leads to a smoother surface of the AlInN thin film as compared to those grown under higher growth pressure, which is in good agreement with the finding from the growths on GaN/sapphire template.

Fig. 8(b) shows the AFM RMS roughness and SEM images as a function of V/III ratio to illustrate the surface morphology of the AlInN layers grown on GaN native substrate. As the V/III ratio increased from 5205 (7122) to 9314, the roughness for AlInN alloys on GaN substrates decreased from 6.5 nm (4.75 nm) and 0.89 nm. Both the AFM and SEM results indicate that the use of higher V/III ratio resulted in improved surface morphology of the AlInN films.

6. Growth optimizations of lattice-matched AlInN alloys on GaN native substrates

The growth optimization studies were also carried out of nearly-lattice-matched AlInN alloys on GaN free-standing substrate. As shown in Fig. 8(b), the V/III ratio was increased from 5205 (7122) to 9314, the roughness for AlInN alloys on GaN substrates decreased from 6.5 nm (4.75 nm) and 0.89 nm. Both the AFM and SEM results indicate that the use of higher V/III ratio resulted in improved surface morphology of the AlInN films.

Table 2: Hall measurement results of Al$_{1-x}$In$_x$N alloy on GaN/sapphire template.

<table>
<thead>
<tr>
<th>In-content (x) of Al$_{1-x}$In$_x$N</th>
<th>Thickness of Al$_{1-x}$In$_x$N (µm)</th>
<th>Sheet resistivity (Ω/cm$^2$)</th>
<th>Hall mobility (cm$^2$/(V·s))</th>
<th>Bulk concentration (cm$^{-3}$)</th>
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<td>1.6E+18</td>
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<tr>
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<td>0.208</td>
<td>341</td>
<td>400</td>
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5. Electrical characteristics of AlInN alloys on GaN/sapphire templates

The electrical characteristics, including sheet resistivity, electron mobility, and carrier concentration of Al$_{1-x}$In$_x$N alloys on GaN templates on sapphire substrates were characterized by employing Van der Pauw Hall method [33]. Table 2 shows the summary of the Hall measurement data of four AlInN thin films with various indium contents. As shown in Table 2, the sheet resistivity of the AlInN film decreases from $\sim 7 \times 10^4$ Ω/cm$^2$ to 341 Ω/cm$^2$, as the In-content was increased from 0.4% to 21%. In our finding, the electron mobilities for the AlInN films show increasing trend for higher In-content films. However, the nearly-lattice-matched AlInN alloy with In-content of 17% exhibits the highest electron mobility among all measured samples, presumably attributed to the improved material quality leading to a reduction in scattering processes in the film. The bulk n-type concentrations are measured in the range of $\sim 10^{18}$ cm$^{-3}$ for AlInN with In-contents in the range of 11%–21%, while the n-type background concentration of $\sim 10^{17}$ cm$^{-3}$ was measured for the AlInN film with In-content of 0.4%. Further improvement of the material quality is still required in order to achieve higher carrier mobility and lower background carrier concentration. Note that the low In-content Al$_{1-x}$In$_x$N thin film has cracks resulting from strain relaxation. Specifically, the Hall measurements are less reliable for the Al$_{1-x}$In$_x$N with x=0.399% since conduction paths within the film are broken.

Fig. 8. Surface roughness as a function of: (a) growth pressure and (b) V/III ratio for AlInN grown on free-standing GaN substrate on sample surface roughness with the insets show the corresponding SEM images for the respective growth pressures and V/III ratio.
AllInN grown on GaN native substrate, which is in good agreement with the finding for the material grown on GaN/sapphire template.

Fig. 9 shows the comparison of the SEM images of 0.2 μm thick nearly-lattice-matched AllInN thin film grown on GaN substrate on sapphire substrate (sample #1804) and on GaN native substrate (sample #1805) under the identical optimized growth conditions with growth temperature of 780 °C, growth pressure of 20 Torr, and V/III ratio of 9314. The SEM images exhibited very similar surface morphology for both AllInN samples, marked by the existence of V-defects on the film similar to the finding reported in the literature [64, 66, 68]. The V-defect densities were estimated as ~4 × 10⁶ cm⁻² and ~3 × 10⁸ cm⁻² on sample #1804 (on GaN/sapphire template) and sample #1805 (on GaN native substrate), respectively. The AFM measurements indicated a slightly smaller RMS roughness of about 0.89 nm on the nearly-lattice-matched AllInN thin film grown on GaN substrate (sample #1805), in comparison to the RMS roughness of 1.01 nm for the sample grown on GaN/sapphire template (sample #1804). Thus, both AFM and SEM measurements showed that the use of GaN native substrate led to the growths of AllInN thin film with improved surface morphology. The surface morphologies of the AllInN thin films are very similar to those reported in the literature [65–71].

Fig. 10 shows the XRD ω–2θ scans for samples #1804 and #1805 around the (002) reflection. The angle separations of AllInN epilayer and GaN peaks were 1044 arcsec and 900 arcsec for sample #1804 and #1805, which corresponded to In-contents of 16.44% and 18.05%, respectively. The discrepancy in the In-content for the two samples grown under the same growth conditions could be attributed to the different thermal conductivity and wafer thicknesses of these two substrates (GaN/sapphire template and GaN native substrate). The GaN native substrate has higher thermal conductivity and thicker substrate thickness in comparison to those of sapphire in the literature [65–71]. In addition, future works using transmission electron microscopy (TEM) are of great importance to characterize the dislocations in the AllInN epitaxial thin films grown on GaN/sapphire templates and GaN free standing substrates. The electrical characterizations using capacitance–voltage (C–V) method are also of interest specifically to understand the origin of high carrier concentration and carrier mobility observed from the experiments.

Fig. 10. XRD ω–2θ scans for nearly-lattice-matched AllInN on GaN(0001) template (#1804) and on GaN free-standing substrate (#1805).
suitability of AlInN alloy as a strong material candidate for solid state cooling and solid state lighting applications, with the thermoelectric figure of merit ($ZT$) value of nearly lattice-matched AlInN obtained as high as 0.532 at room temperature ($T = 300$ K) [32–34]. Several recent works have identified the potential of employing large bandgap AlInN barrier materials to suppress the carrier leakage process in the InGaN QW LEDs [40, 41], which potentially will lead to suppression of efficiency droop in nitride LEDs. The use of lattice-matched (In-content $\pm 1\%$) or slightly-tensile-strained (In-content $\pm 10\%$ up to $16\%$) AlInN alloys are of great interest for integrating as barrier materials in InGaN QW LEDs.

8. Summary

In summary, MOVPE and characterizations of AlInN alloys grown on both GaN/sapphire templates and free-standing GaN substrates were performed and discussed. The growth optimizations were performed aiming to obtain the lattice matching conditions for AlInN alloys. The effects of growth pressure, growth temperature, and V/III ratio were studied for the epitaxy of AlInN thin films on GaN/sapphire and GaN substrates. The optimization studies of nearly-lattice-matched AlInN films on both GaN/sapphire template and GaN substrate indicated that the use of lower growth pressure and higher V/III ratio led to an improvement in surface morphology and a reduction in RMS roughness as characterized by SEM and AFM measurements. The AlInN thin film grown on GaN substrate was also shown to have lower surface roughness in comparison to that grown on GaN/sapphire template, indicating the advantage from the use of GaN substrate. In addition, various applications of AlInN for thermoelectric and light-emitting diodes applications were also discussed.

Acknowledgment

This work is supported by DARPA and US National Science Foundation (ECCS #0701421, ECCS #1028490, DMR # 0907260), and in part by Class of 1961 Professorship Fund.

References
