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Improved Quality Non-polar III-Nitride
Heteroepitaxial Films and Devices

A Dissertation submitted in partial satisfaction of the
requirements for the degree of

Doctor of Philosophy in
Materials

by

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Bilge Imer

This dissertation is dedicated to the most incredible parents

Zahide & Sencer Imer

... and the best brothers

Cagri & Tugrul Imer

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PUBLICATIONS

First-Author Publications

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2. “Stability of non-polar $(1\bar{1}00)$ m-plane GaN films with Metalorganic Chemical Vapor Deposition (MOCVD)”, **Bilge Imer**, Feng Wu, Michael D.

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2. “Temperature-dependent Radiative Lifetime of Excitons in NON-polar GaN/AlGaIn Quantum Wells”, (S. Rudin, G. A. Garrett, H. Shen, W. Wraback) from U.S. Army Research Laboratory, Sensors and Electron Devices Directorate (**B. Imer**, B. Haskell, J. S. Speck, S. Keller, S. Nakamura, and S. P. DenBaars) from UCSB, *IEEE, Semiconductor Device Research Symposium, 2005 International Proceedings, 225, December 2005.*
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6. "Microstructural evolution of *a*-plane GaN grown on *a*-plane SiC by Metalorganic chemical vapor deposition", M. D. Craven, F. Wu, A. Chakraborty, **B. Imer**, U. K. Mishra, S. P. DenBaars, and J. S. Speck, Applied Physics Letters, Vol. 84, No.8, February 2004
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6. 47th Electronic Materials Conference (EMC), Santa Barbara, CA, June 22-24, 2005 “Growth of Planar, Non-polar (1 -1 0 0) m-plane GaN over m-plane SiC by Metalorganic Chemical Vapor Deposition (MOCVD)”, **Bilge Imer**, Feng Wu, James S. Speck, Steven P. DenBaars
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ABSTRACT

Improved Quality Non-polar III-Nitride Heteroepitaxial Films and Devices

by

Bilge Imer

GaN and its compounds suffer from polarization fields when grown along the c - $\langle 0001 \rangle$ direction. These polarization fields result in poor carrier recombination efficiencies in quantum wells and shift in emission wavelength caused by the quantum confined stark effect due to spatial separation of the electron and hole wavefunction separation. It is possible to eliminate these effects by growing along one of the nonpolar directions, namely a - $\langle 11\bar{2}0 \rangle$ or m - $\langle 1\bar{1}00 \rangle$, so that the polarization fields are normal to the growth direction. However films grown along these nonpolar directions suffer from high defect densities. Consequently, devices structures grown with these orientations suffer from poor electrical and optical characteristics.

In this dissertation these two issues were addressed so as to eliminate the polarization fields by nonpolar growth along the m - $\langle 1\bar{1}00 \rangle$ direction with

metalorganic chemical vapor deposition (MOCVD) and to effectively reduce the high defect densities in films grown along the nonpolar directions employing a new growth technique, sidewall lateral epitaxial overgrowth (SLEO).

Previously, device-quality nonpolar a- $(11\bar{2}0)$ plane GaN films were achieved. Growth along this plane however was extremely sensitive to changes in growth conditions and was limited to low pressures and restricted precursor flows. In this dissertation it was shown that nonpolar m- $(\bar{1}100)$ plane GaN films are more stable and less sensitive to changes in growth conditions. The first device-quality m- $(\bar{1}100)$ GaN films were then grown on m-plane 6H-SiC substrates with an AlN nucleation layer by MOCVD.

A new technique, SLEO, was developed to effectively reduce defect densities in nonpolar films. Using this method it is possible to reduce threading dislocation (TD) densities by 3-4 orders of magnitude to $\sim 10^6$ - 10^7 cm⁻² and stacking fault (SF) densities by 1-2 orders to $\sim 10^3$ - 10^4 cm⁻¹. This would improve the performance of devices subsequently grown on these high quality templates. For direct comparison, GaN/Al_{0.15}GaN 360 nm UV LEDs were grown on co-loaded planar a-GaN, planar c-GaN and SLEO a-GaN templates. Devices grown on nonpolar SLEO films demonstrated ~ 2 - $3\times$ higher electron mobility, $\sim 3\times$ lower series resistance, and ~ 100 - $300\times$ higher EQE when compared with the planar nonpolar films.

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CHAPTER 1

Introduction

1.1. Introduction to III-Nitrides

Still a relatively new compound semiconductor system, III-nitrides, which include GaN and its alloys with In and Al, are used in various optoelectronic and electronic devices in energy, defense, information and medical technology applications. This material system is extremely versatile in that the range of bandgap energies available is very large. The bandgap energy ranges from 0.65 eV for InN, to 3.4 eV for GaN, to 6.2 eV for AlN (Figure 1.1). For active optical devices, the III-nitrides therefore can potentially cover from the deep UV through the visible spectrum and into the infrared (IR). Other favorable attributes of the III-nitrides, particularly for high power microwave electronic devices, are a high breakdown field and high electron velocity [1]. Moreover these wide bandgap materials are chemically inert and high temperature resistant making them excellent candidates for harsh chemical environment and high-temperature operation. Currently, some device applications for III-nitrides include light emitting diodes (LEDs) for solid-state lighting and for displays, blue lasers for DVD players, and high electron mobility transistors (HEMTs) for high power microwave circuits.

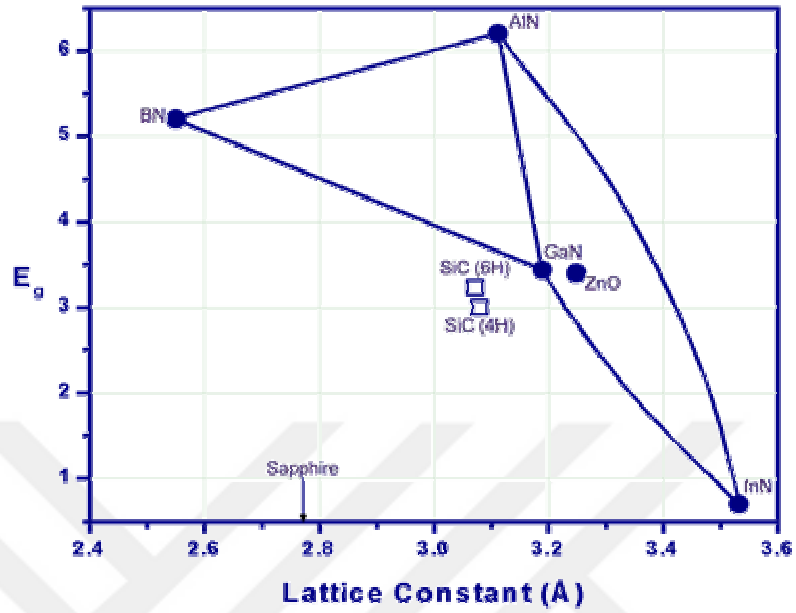


Figure 1.1 III-Nitrides Band-gap energies and lattice constants. (Courtesy of Craig Moe)

1.1.1. Crystallography

GaN is present in two forms as cubic zincblende and hexagonal wurtzite. The wurtzite structure is the most thermodynamically stable for bulk III-nitrides. The space group for the wurtzite structure is $P6_3mc(C_{6v}^4)$. The unit cell is hexagonal and is described by three basal plane vectors ($\mathbf{a}_1$, $\mathbf{a}_2$, $\mathbf{a}_3$) and a vector normal to the basal planes ($\mathbf{c}$). This structure consists of two interpenetrating hexagonal close packed (hcp) sublattices displaced by $5/8c$ (where c is the cell height). Each sublattice contains either Group III atoms or nitrogen atoms only. The bilayers stacking sequence of the basal c - $\{0001\}$ plane is therefore AaBbAaBb [2]. Fig. 1.2 shows the crystal planes and directions in hexagonal GaN. The basal plane of the

unit cell is the polar c-(0001) plane. The common nonpolar planes are the $(11\bar{2}0)$ plane and the $(1\bar{1}00)$ plane and are denoted as a-plane and m-plane respectively.

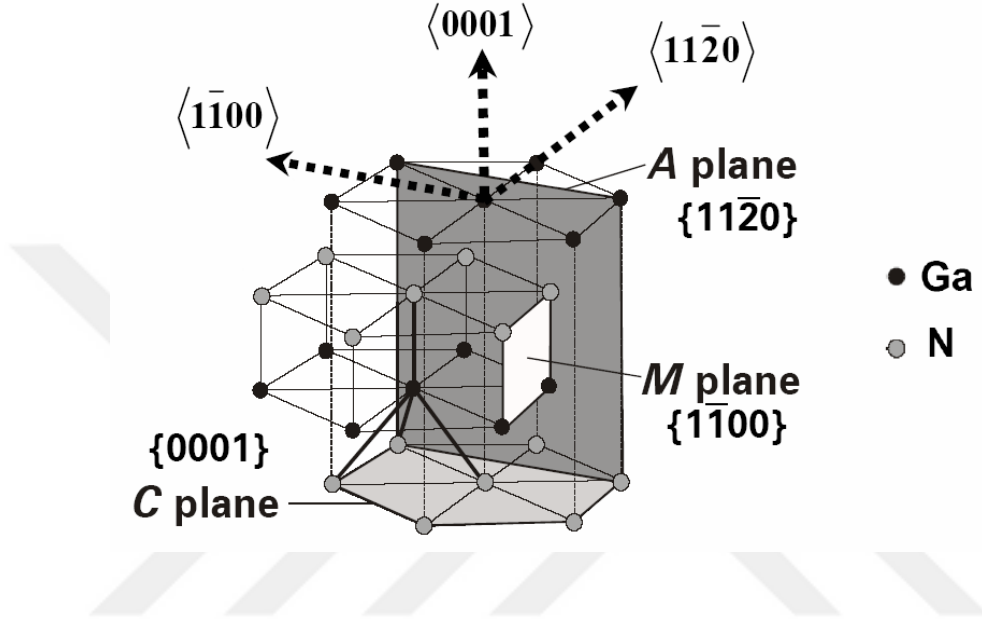


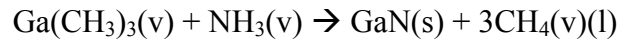
Figure 1.2 Polar and nonpolar crystal planes and directions of interest in hexagonal GaN [3].

1.1.2. MOCVD Growth

Most widely used methods to grow III-nitride films are molecular beam epitaxy (MBE), hydride vapor phase epitaxy (HVPE), and metalorganic chemical vapor deposition (MOCVD). With MBE, a plasma is often used for supplying an atomic source of nitrogen. This may be advantageous because in contrast to other growth techniques employing ammonia, lower growth temperatures can be used and the growth environment is free of hydrogen. Growth rates in MBE are typically low and controllable allowing for abrupt growth interfaces necessary for electronic

device structures. With HVPE, chlorine is used to transport the Group III species and very high growth rates can be achieved. Although thick GaN films can be deposited for templates using this technique, it is unsuitable for the growth of device structures. The MOCVD growth technique uses vapor transport of metalorganic precursors and subsequent reactions of Group III and Group V alkyls in a heated zone for deposition of epitaxial films. The growth rate and composition are well controlled with MOCVD and typical growth rates are higher than that for MBE and lower than that of HVPE. MOCVD has been widely used in the growth of epitaxial films for both electronic and optoelectronic device structures. MOCVD is used in this work for the growth of nonpolar III-nitride films and for optoelectronic device structures.

By precisely controlling the mass flow rate and dilution of the various components of the injected gas stream, the composition and growth rate can be well controlled with MOCVD. For the group III sources, trimethylgallium (TMGa), trimethylaluminum (TMAI), and trimethylindium (TMIn) can be used. Ammonia (NH₃), which is a gaseous hydride, is used as the group V source in III nitride growth. Cyclopentadienylmagnesium (Cp₂Mg) is used as a p-dopant and disilane (Si₂H₆) is used as an n-dopant. The basic reaction for the deposition of GaN films by MOCVD using the aforementioned source materials is as follows:



Because of the high bond strength of the N-H atoms in ammonia, high growth temperatures are required to crack these precursors.

Using MOCVD we always grow on the Ga face. It is only possible by doping heavily with Mg to grow on the N face. However, we do not want N face due to its unstable and rough surface characteristics.

1.1.3. Challenges

Since the wurtzite structure has polar point (6mm) symmetry, the material has a spontaneous polarization in the polar axis parallel to c -<0001>. For example by using the given spontaneous polarization value of AlN, -0.081 C/m^2 , we can obtain a huge electric field that is about 9.2 MV/cm . Furthermore, if we calculate the voltage change for a 3 nm AlN layer (without any other extra field) it gives the value of 2.7 V . The addition of strain causes piezoelectric polarization, which is much larger in value and might be either on the same or the opposite direction of the spontaneous polarization. Following Fig. 1.3 exhibits the effect of strain and the growth face on the direction of the spontaneous and piezoelectric polarization. Piezoelectric field is induced since group III- nitrides have large piezoelectric constants along c -<0001> direction. As can be seen from Fig. 1.3, if we grow on different faces of the hexagon, either Ga-(0001) face or N-(000-1) face, we see opposite effects.

As a result of the piezoelectric and spontaneous polarizations the optical properties of III-nitrides are strongly affected by the quantum confined Stark effect (QCSE) as demonstrated in Fig. 1.4, leading to shifts in emission wavelength and decrease in intensity which is discussed in the following section.

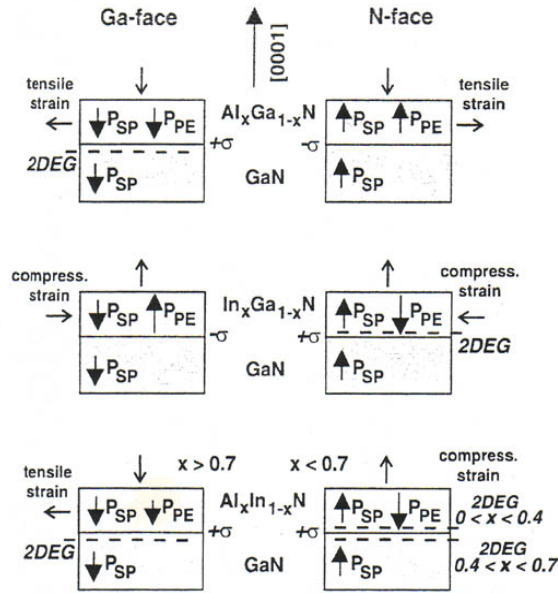


Figure 1.3 Spontaneous and piezoelectric polarization effects on different faces of GaN under compressive and tensile strain.

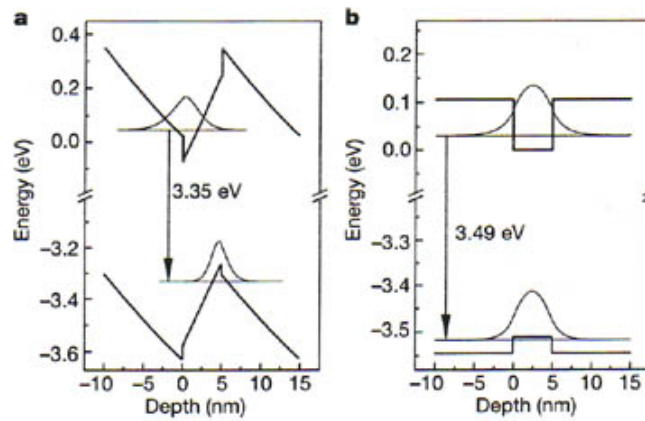


Figure 1.4 A comparison between a) polar c-plane GaN/AlGaIn SQW band diagram in the presence of PE and b) m- or a-plane GaN/AlGaIn SQW band diagram in the absence of PE [4].

Polarization fields cause the device to perform inefficiently due to poor electron-hole recombination resulting from electron-hole separation. In Figure 1.4 a), it is shown that the electron and hole wavefunctions are separated and pushed toward the opposite sides of the well. The reduced overlap results in a corresponding reduction in radiative recombination rates. The probability of carriers tunneling out of the wells also increases, resulting in a decrease in carrier lifetimes and broadening of the absorption spectra [5]. As a result, optical transitions are less intense, and red-shifted.

Another challenge in III-nitrides is the presence of high extended defect densities. The most common types of these defects are threading dislocations (TD) and planar stacking faults (SF). TDs form at the growth interface due to lattice mismatch and strain between the heteroepitaxial film and the foreign substrate. For polar planar c-plane films TD densities are $\sim 10^8$ - 10^9 cm⁻², while this value goes up to $\sim 10^{10}$ cm⁻² for nonpolar planar films. TDs are mostly composed of screw components and aligned along the growth direction and extend to the surface. SFs are the errors in usual layer stacking sequence. These defects extend to the surface, parallel to the growth direction, initiating on the N-face for nonpolar nitrides and usually their densities are $\sim 10^5$ cm⁻¹ in planar nonpolar films. Despite having rather large defect densities compared with other conventional III-V semiconductors, III-nitrides still can tolerate the presence of these defects. However it is essential to reduce these defect densities to improve upon the device performances.

In summary, extended defects, such as threading dislocations (TDs) and stacking faults (SFs), and polarization related internal electric fields are two

limitations in the performance of III-nitride devices. In c-axis oriented wurtzite group-III nitride heterostructures, internal electrical fields are present due to discontinuities in electric polarization. The internal electric fields, in quantum wells for instance, cause spatial separation of electron and hole wave functions and reduce recombination rates and possibly internal quantum efficiency. TDs hinder the device performances by acting as non-radiative recombination centers, charge scattering centers for carriers, and leakage paths in p-n junctions [6]. Therefore eliminating polarization related electric fields and reducing extended defect densities is essential for improving III-nitride device performance.

1.1.4. History of GaN

Polar History

In 1932 the first GaN was produced in the powder form [7]. About 35 years later the first single crystalline GaN was deposited using HVPE [8]. Following this Pankove et al. demonstrated the first GaN based LED [9]. However the crystal quality was poor and the surface was rough and growing smooth epitaxial films was a substantial challenge because of the large lattice mismatch between GaN and sapphire. This was overcome in 1985 when Amano et al. [10] reported a “two-step” growth process by which a low temperature AlN nucleation layer was deposited prior to GaN growth. Another early challenge was obtaining good p-type doping in GaN, which tends to be n-type. It was found that magnesium-doped p-type films required electrical activation. While Amano et al. used low-energy electron beam

for activation [11], Nakamura et al. discovered that as-grown magnesium-doped films were passivated by the presence of hydrogen during growth and this activation could be achieved by thermal annealing [12,13]. These films became p-type after thermal annealing in a N_2 ambient. These achievements accelerated GaN based device research and made currently available UV, blue, and green LEDs, and high power transistors possible.

Nonpolar History

It is relatively easy to grow planar c-plane GaN due to its large growth stability. Nearly all wurtzite III-nitride-based devices have been grown predominantly in the (0001) or c-plane orientation. However, owing to their 6mm point symmetry, wurtzite structures are polar. The polarization effect and its consequences were discussed in section 1.1.3. One possible approach for eliminating piezoelectric polarization effects in III-nitride devices is to grow the devices on the nonpolar planes of crystals such as a- $(11\bar{2}0)$ and the m- $(1\bar{1}00)$ planes of GaN. On the basis of crystal symmetry, a- or m-plane– oriented films have their polar c-axis parallel to their surface, and thus, heterostructures grown in either of these orientations are free of polarization-related electric fields. The recent studies of a-plane [14,15] and m-plane [16,17] AlGaIn/GaN quantum wells and a-plane [18,19] and m-plane [20] GaN/InGaIn quantum wells demonstrate that it is possible to eliminate such polarization fields by growing device structures along these non-polar orientations.

The history of nonpolar III-nitride growth is fairly limited. In the 1990s several groups attempted to grow nonpolar planar m - $(\bar{1}\bar{1}00)$ plane GaN [21-25] and nonpolar a - $(11\bar{2}0)$ plane GaN films [26-28] on various substrates by different growth methods. However, in these first attempts the film quality was poor and not suitable for device growth. Therefore, much of the early work in GaN growth involved polar c -plane films until early-2000s. In 2000, Waltereit et al. reported the first nonpolar m -plane GaN/AlGaIn quantum wells grown on a lithium aluminate (LiAlO_2) substrate by MBE [16]. After this the research in nonpolar growth accelerated and since then it has come a long way with the help of pioneering researchers at UCSB. In 2002 Craven et al. reported the first device-quality MOCVD growth of a -plane GaN on r -plane sapphire [29] and a -plane SiC [30]. Also Haskell et al. reported high-quality a -plane GaN growth with HVPE [31]. However the range of MOCVD growth parameters to sustain planar a -plane films (growth ‘window’) is narrow and highly sensitive to changes in growth variables such as reactor pressure, temperature and precursor flow rates [32], and also the extended defect densities in a -plane $(11\bar{2}0)$ GaN were fairly high ($\sim 10^{10} \text{ cm}^{-2}$ TDs and $\sim 10^{-5} \text{ cm}^{-1}$). Various methods including single-step lateral epitaxial overgrowth (LEO) [33-35] and two-step LEO [36] were exhausted to reduce these defect densities. Due to limited growth window of a -GaIn films, it was extremely difficult to coalesce the overgrowing LEO islands and to get reproducible results. To date the research efforts to grow m - $(\bar{1}\bar{1}00)$ plane GaN have been mostly focused on using HVPE [37] and MBE [4,16] growth methods due to unavailability of the proper substrate to withstand the high growth

temperatures required by MOCVD. Although there was one report by Chen et al. on MOCVD growth of GaN homoepitaxy on HVPE grown freestanding m-plane GaN [38], the realization of the first device-quality heteroepitaxial m-plane GaN films in this dissertation was made possible with the emergence of commercially available m-plane SiC.

In summary, there are two main issues which need to be addressed in nonpolar III-nitrides: high defect densities compared with the polar counterpart and also the limited growth window of nonpolar a-plane films restricted at low growth pressures. Both of these issues are addressed and resolved in this dissertation.

1.2. Thesis Overview

The main objective of this work is to improve the material quality and the growth stability of nonpolar GaN epitaxial films. The effect of material quality improvement upon the device performances and electrical characteristics is also incorporated.

Chapter 2 covers heteroepitaxial growth and structural characterization of nonpolar m-plane GaN films (an alternative to nonpolar a-plane GaN films) using AlN nucleation layer (NL) on m-plane 6H-SiC substrates with MOCVD. This chapter starts with the discussion of stability of nonpolar m-plane GaN films over nonpolar a-plane GaN films with experimental evidence. Further in the chapter, MOCVD growth optimization studies of AlN NL and GaN heteroepitaxial films are presented in detail along with structural characterization. The chapter concludes

with a side-by-side material quality comparison between the two nonpolar orientations, as a-plane and m-plane GaN.

The presence of defects in III-nitrides is associated with compromised device performance and lifetime. Therefore it is vital to reduce, and ideally completely eliminate these defects, in particular threading dislocations (TDs). Fundamentals of sidewall lateral epitaxial overgrowth (SLEO), a new method to reduce these defect densities in nonpolar GaN are discussed in Chapter 3. The processing and growth steps are individually considered. Finally the improvement in film quality by utilizing the SLEO method is compared to conventional single-step LEO and planar films.

The electrical properties of reduced defect density high quality nonpolar a-plane GaN SLEO material are presented in Chapter 4. For electrical characterization Hall and TLM measurements were utilized for n- and p-type doping of SLEO a-GaN. 365 nm GaN/AlGaN UV LEDs were grown over the high quality SLEO material. The performance of the devices grown over SLEO a-plane GaN was compared with the ones grown over planar a- and c-plane GaN films.

The final chapter is the summary and discussion of this thesis along with suggested future directions in nonpolar research.

Also an Appendix is included which discusses the temperature dependent time resolved photoluminescence (TRPL) studies of a-plane GaN/AlGaN multiple quantum wells (MQWs).

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CHAPTER 2:

Heteroepitaxial growth of nonpolar m-plane GaN on m-plane 6H-SiC with Metalorganic Chemical Vapor Deposition (MOCVD)

2.1. Introduction

On the basis of crystal symmetry, a- $(11\bar{2}0)$ or m- $(\bar{1}\bar{1}00)$ plane-oriented films have their polar c-axis parallel to their surface, and thus, heterostructures grown in either of these orientations are free of polarization-related electric fields. Several groups attempted to grow planar m- $(\bar{1}\bar{1}00)$ plane GaN films on various substrates by different growth methods; first time in 1996 m-GaN growth on m-plane SiC with metalorganic chemical vapor deposition (MOCVD) by Horino et al. at Fujitsu [1], in 1997 m-GaN growth on (100) γ -LiAlO₂ with molecular beam epitaxy (MBE) by Hellman et al. [2] and m-AlN growth on m-plane SiC with MBE by Stemmer et al. [3], and in 2002 m-GaN growth on (100) LiAlO₂ with hydride vapor phase epitaxy (HVPE) by Maruska et al. [4,5]. However in these first attempts the film quality was poor and not suitable for device growth. The first device-quality m-plane GaN films on LiAlO₂ were achieved by Waltereit et al. [6,7] in 2000 with MBE. After 2000 the research in nonpolar growth field accelerated, especially for films and devices in the a- $(11\bar{2}0)$ plane orientation. The high quality nonpolar $(11\bar{2}0)$ a-plane growths of GaN on r-plane sapphire [8] and a-plane SiC [9] have been demonstrated by MOCVD and HVPE [10]. However, it was found that the range of MOCVD growth

parameters to sustain planar a-plane films (growth ‘window’) is narrow and highly sensitive to changes in growth variables such as reactor pressure and precursor flow rates [11]. This drove us to explore a new non-polar orientation in GaN growth with MOCVD. The following section demonstrates why m-plane GaN can be a good counterpart for a-plane GaN in nonpolar nitride growth with MOCVD. In the subsequent segments of this chapter, we will be discussing the optimization of heteroepitaxial growth of m-plane GaN on m-plane 6H-SiC.

2.2. Why m-plane?

2.2.1. *m-plane Facet Stability: $[0001]_{\text{GaN}}$ Oriented a-plane GaN LEO*

We observed that m-plane GaN is more stable than a-plane GaN over a wide range of growth conditions. The stability of m-GaN facets was first observed when we investigated the pressure and temperature dependence of overgrown stripes with single-step lateral epitaxial overgrowth (LEO) over a-plane GaN, as demonstrated in Fig. 2.1. $\sim 1\ \mu\text{m}$ thick a-plane GaN films were grown on r-plane sapphire at 1160°C and 60 Torr with 27 sccm TMGa and 1 slpm NH_3 precursor flow rate. An approximately $\sim 2000\ \text{\AA}$ thick SiO_2 layer was deposited by plasma-enhanced chemical vapor deposition (PECVD) as a LEO mask. Then LEO stripes with $5\ \mu\text{m}$ openings (window) and $15\ \mu\text{m}$ mask (wing) size were patterned parallel to $[0001]_{\text{GaN}}$ with photolithography such that the growing sides would be either $\{\bar{1}100\}$ m-plane or $\{1\bar{1}20\}$ a-plane facets or a combination of a- and m-plane facets. The SiO_2 mask was

etched down to the GaN template through these windows using inductively coupled plasma (ICP) etching. These processing and growth flows are shown in Fig. 2.1. LEO processed templates were grown in a close-spaced, rotating disk vertical MOCVD reactor by varying the temperature between 1115 and 1190°C and with a total pressure between 45 and 150 Torr. Trimethylgallium (TMGa) and NH_3 precursors were used as the group III and V sources with a molecular ratio of 740 in H_2 carrier gas. The growth was stopped at ~1800 s, before adjacent GaN stripes coalesced.

As demonstrated in Fig. 2.1, the $\{\bar{1}100\}$ m-plane sidewall facets in the uncoalesced a-plane LEO structures were remarkably stable with increasing growth pressure and temperature. The a-plane, which is the primary growth plane, was dominated by m-plane facets as the pressure and/or temperature was raised. Also M. D. Craven, observed a very small growth stability window limited at low pressures below 76 Torr for planar a-GaN films [11]. Over 76 Torr severe surface faceting and degradation in crystal quality was unavoidable and over 300 Torr there was no dominant a-plane GaN surface. Therefore observation of m-facet stability was the main motivation in studying the planar m-plane GaN growth by MOCVD.

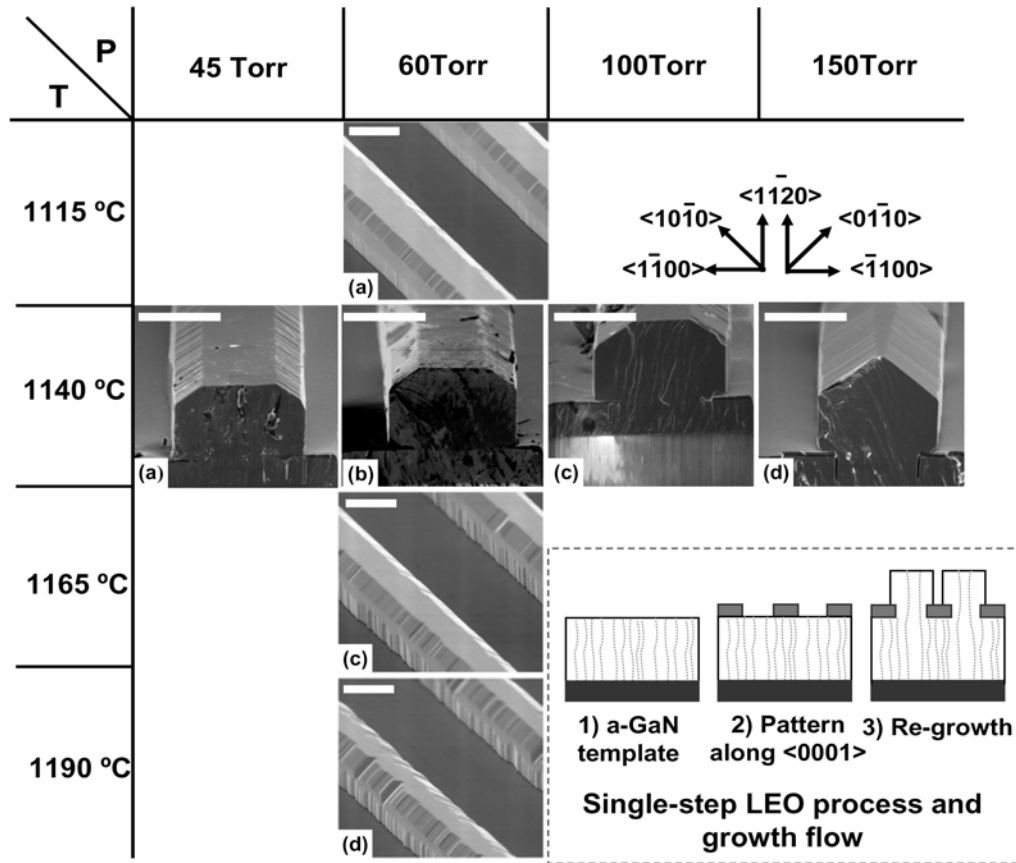


Figure 2.1 m-Facets stability experiment with changing pressure and temperature on exposed m-faces from LEO stripe patterns. The same figure shows the schematic of the single-step LEO experiment and the planes. (The scale bar is 5 μm .)

2.2.2. Carbon incorporation

In GaN films grown by MOCVD, carbon can come from incomplete dissociation of TMGa precursor gas since metalorganics intrinsically contain carbon. Film growth conditions such as temperature, pressure, and precursor flows or

combination of all, play an important role in controlling the amount of carbon incorporation. Carbon concentration level can be varied over a wide range by altering the deposition pressure. Carbon incorporation increased with lower growth temperatures [12-14], lower growth pressures [13-17], and lower V/III ratios [12, 18]. Among all growth parameters, pressure has the greatest impact on carbon concentrations.

In this study the effect of growth pressure on carbon incorporation in GaN was studied. A SIMS stack with six periods of different pressures varying between 40 – 300 Torr were grown over a c-plane GaN template. Each period was made up of 500 nm varying pressure UID GaN and 200 nm GaN:Si spacer. The SIMS stack structure is given in Fig. 2.2 accompanied by the SIMS profile. Throughout the stack, the rest of the growth variables were kept constant at a growth temperature of 1130 °C, 27 sccm TMGa flow, NH₃ flow of 1 slpm, and 10 slpm total flow. H₂ was used as carrier gas.

The SIMS profile revealed that the decrease in growth pressure was accompanied by an increase in carbon incorporation in GaN. Especially below 76 Torr carbon incorporation rate increased prominently. At 40 Torr more than 4 times carbon incorporated in the material than at 76 Torr. This increasing trend with decreasing reactor pressure is represented in Fig 2.3. In the same figure, it is also possible to see that over 76 Torr, carbon incorporation gets in the area of little variation while still dropping.

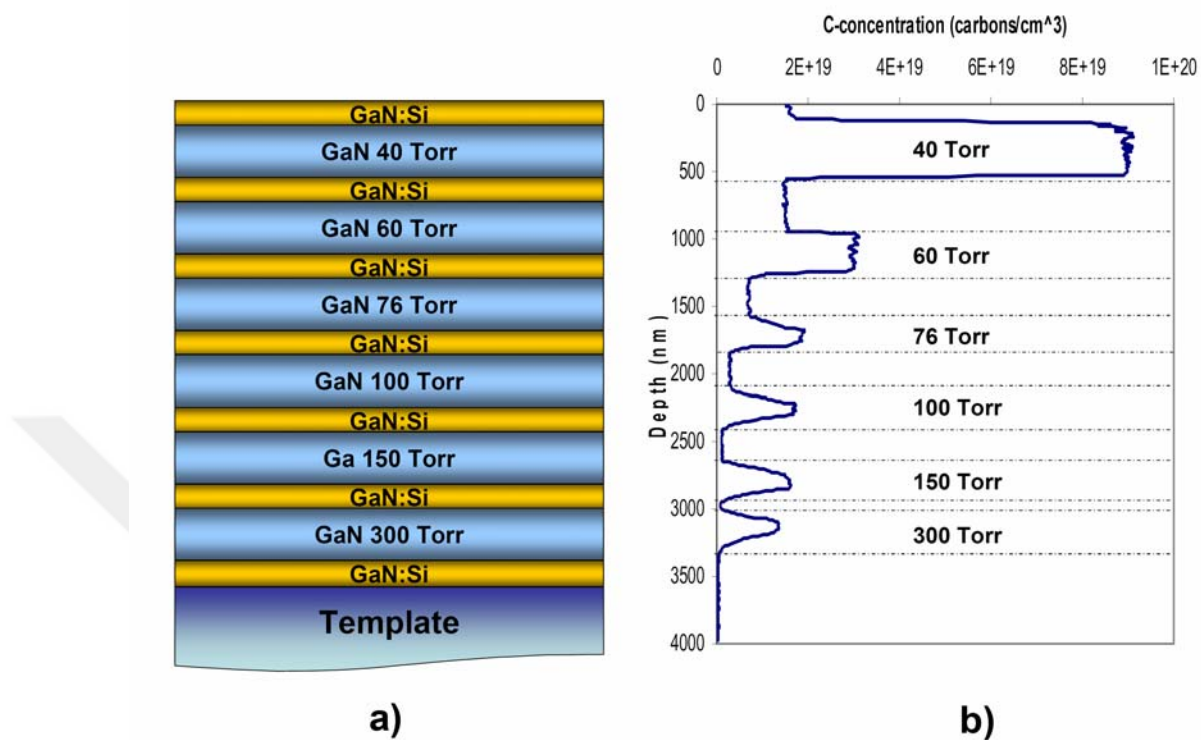


Figure 2.2 Carbon incorporation with change in growth pressure: a) SIMS stack structure, b) SIMS profile of carbon concentration

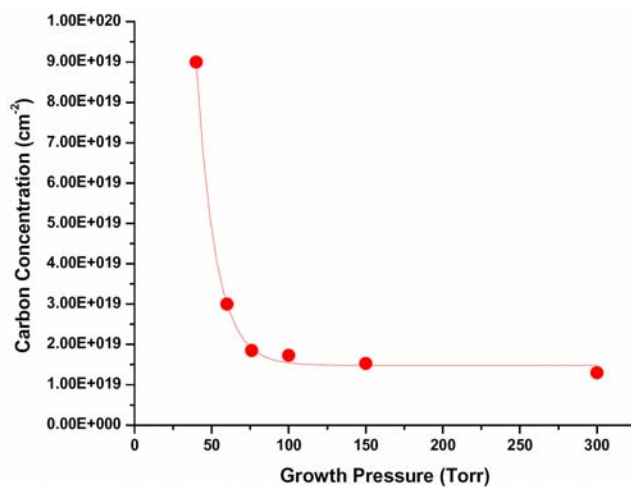


Figure 2.3 Carbon incorporation with change in growth pressure.

Carbon has the potential in achieving semi-insulating GaN in HEMTs when the carbon concentration exceeds the silicon concentration carbon occupies nitrogen (C_N) and gallium sites (C_{Ga}) [15], and also it is the low activation energy alternative to Mg p-type doping when substituted as C_N at a sufficient level [17, 19]. However the carbon related states in III-nitrides is not well understood. Carbon is always present in GaN, however controlled doping has been a problem.

Aside from the purposeful introduction of carbon, unintentional incorporation of such an impurity in GaN can cause carrier compensation when it is a shallow acceptor or yellow luminescence problems when it is a deep acceptor. Armstrong et al. [15] found that GaN samples grown at 76 Torr had higher carbon and GaN samples with high carbon content were found to be highly resistive, and samples co-doped with C and Si were heavily compensated. It has been shown that at carbon concentrations less than $2 \times 10^{18} \text{ cm}^{-3}$, carbon was found to incorporate predominantly in the form of C_N shallow acceptors [20]. Hwang et al. [14] studied the effect of carbon incorporation on Hall mobilities and proved that carbon is the main reason for carrier compensation since it acts as acceptor in nitrogen vacancy sites C_N . Normally as the carrier concentration decreases mobilities should increase. However under a certain level of carrier concentration, ($2 \times 10^{17} \text{ cm}^{-3}$ for c-GaN and $2 \times 10^{18} \text{ cm}^{-3}$ for a-GaN because of higher carbon incorporation at lower pressures) the mobility values drop with compensation.

As mentioned previously, carbon forms not only a shallow acceptor level but also a deep acceptor level. Yellow luminescence on the other hand is from shallow donor to deep acceptor [21]. There has been controversy in determining the source

of the yellow emission. Some groups argue that YL is related to carbon [17, 20] while others dispute that it is related with gallium vacancy [13, 22].

At high carbon concentrations ($>2 \times 10^{18} \text{ cm}^{-3}$) the resistivity and conduction activation energy decreases although for low density of Ga vacancies the material still exhibits strong YL emission at 2.2 eV. YL involves both V_{Ga} and interstitial carbon emission mechanism [20]. Also Seager et al. [17] suggested that carbon can be responsible for YL if $[\text{C}] > [\text{Si}]$ leading to transition between Si (donor) to C_{N} (deep acceptor) state.

As mentioned earlier, the growth pressures for nonpolar device-quality a-plane GaN films have a growth window limited with low growth pressures (60-76 Torr). Being restricted in the low pressure regime for a-plane GaN creates the risk of incorporating unintentional carbon impurities at a level at which it is highly compensating and a probable source of yellow emission, degrading electrical and optical characteristics of the devices. In comparison, m-plane stability increases with increasing growth pressure, as was proved in the previous section. Therefore m-plane growth is a promising nonpolar candidate to prevent high carbon incorporation in GaN by giving the flexibility of wider growth stability window.

2.2.3. Substrate

For m-plane growth, substrate availability has been a problem due to high growth temperatures required in MOCVD. Commercially available substrates such as $\gamma\text{-LiAlO}_2$ have melting points lower than the temperature required for MOCVD growth. With the emergence of commercially available m-SiC substrate, which is

stable during MOCVD growth, current invention was made possible. However, the current maturity level for substrate-to-substrate quality of m-plane SiC is not yet at the same level as other commercial substrates.

The wafers were obtained from INTRINSIC Semiconductors. ($\bar{1}\bar{1}00$) 6H-SiC $\pm 1^\circ$ surface orientation tolerance according to specs. The wafers were prepared by cutting from cylindrical boules grown along c-plane on-axis. The size of the substrates varied from 16x45 mm smallest to 21x54 mm largest depending on the boule size.

SiC and GaN are both from the same space group of $P6_3mc$ therefore both share the same crystal structure, however their stacking sequence differs. While for GaN the stacking sequence is defined as AaBbAaBb, the stacking sequence for 6H-SiC is AaBbAaCcAaBbAaCc. Table 2.1 shows the lattice mismatch between these 6H-SiC substrates and m-plane GaN and AlN nucleation layer.

Table 2.1 Lattice mismatch between m-plane GaN, AlN, and m-plane 6H-SiC.

Epitaxial Layer Relation	Mismatch along a-direction (%)	Mismatch along c-direction (%)
GaN/AlN	2.477	3.95
AlN/6H-SiC	0.945	-1.1867
GaN/6H-SiC	3.39	2.8

In summary, there are many challenges in producing nonpolar GaN films with a-plane orientation including very small growth window, limited precursor

flows and high carbon incorporation due to being restricted at low growth pressures (<76 Torr), and high dislocation densities ($\sim 10^{10} \text{ cm}^{-2}$). By having the flexibility in wide range of growth pressures most of these challenges can be overcome, and m-plane has the pressure and temperature stability as was proved in section 2.2.1. Therefore, with the commercial availability of proper growth substrate for m-plane by MOCVD, this study made it possible to address these challenges.

2.3. Growth and Characterization

We investigated the stability of m-plane GaN films on m-plane 6H-SiC substrates under various growth conditions. 6H-SiC substrates were treated in dilute buffered hydrofluoric acid (1 portion BHF:10 portion DI) to remove the native oxide layer from the surface. The wafer was then loaded into a vertical close-spaced rotating disk 1x2'' Thomas Swan MOCVD reactor. The growth was carried out in two steps: 1) Deposition of a high temperature (HT) AlN nucleation layer (NL), 2) Heteroepitaxial growth of m-plane GaN layer. The NL was utilized to compensate the large difference in lattice mismatch and thermal expansion coefficients between the substrate and the film to prevent cracking and to also create a smooth surface morphology. TMGa, and trimethylaluminum (TMA) precursors were used as group III sources, and NH_3 was utilized as a group V source. First, an m-plane 6H-SiC substrate was annealed in situ to remove residual surface species for 300 s in H_2 before the deposition of AlN NL. The m-plane GaN film was then grown over the AlN NL. Figure 2.4 shows the processing and growth flow and the surface morphology evolution in each stage.

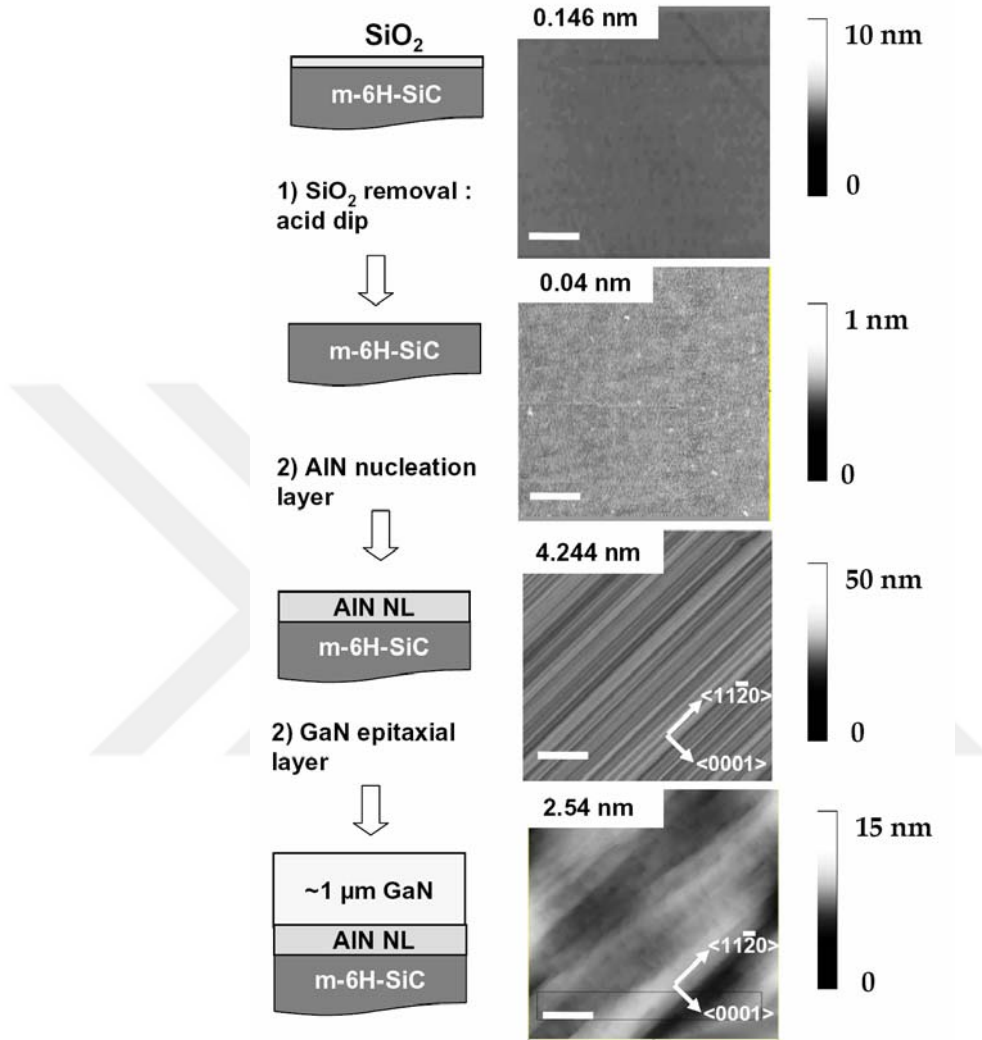


Figure 2.4 Process and growth schematic of m-plane GaN. All AFM images are $5 \times 5 \mu\text{m}^2$.

The standard reference growth conditions for m-SiC anneal, and AIN NL and GaN growth were adapted from optimized conditions for a-plane GaN growth on a-plane SiC (23). These conditions are given in Table 2.2.

Table 2.2 Standard Reference Conditions Used in m-GaN Growth Optimization

Parameter	
m-SiC anneal	
Anneal Temperature	1250 °C
Anneal Duration	300''
Total Flow (in H ₂)	4.5 slpm
Reactor Pressure	76 Torr
AlN Nucleation Layer	
Growth Temperature	1250 °C
Growth Duration	2000''
Total Flow	6.0 slpm
Reactor Pressure	76 Torr
TMA Flow	40.0 sccm
NH ₃ Flow	4.0 slpm
V/III Ratio	3536
Growth Rate	~1 Å/s
Thickness	~200 nm
High Temperature GaN	
Growth Temperature	1130 °C
Growth Duration	1800''
Total Flow	10.0 slpm
Reactor Pressure	76 Torr
TMGa Flow	27.0 sccm
NH ₃ Flow	1.0 slpm
V/III ratio	~734
Growth Rate	~5.6 Å/s
Thickness	~1 µm

Any modification to the standard growth conditions was realized by systematically changing one growth parameter at a time and keeping the remaining growth parameters constant. The optimization was carried out on over 70 samples in two main stages. For this purpose, first, we maintained the GaN epitaxial layer under standard growth conditions nominally constant while modifying the growth variables of the AlN nucleation layer such as pressure, temperature, thickness, and V/III ratio. We also characterized the surface morphology of a bare AlN NL without

GaN on m-plane 6H-SiC with the same growth variables. After tuning the growth parameters for the nucleation layer, the GaN epitaxial crystal and surface qualities were optimized for varying pressure, temperature and V/III ratios. The growth conditions that have been tested for each individual layer are provided in Table 2.3.

Table 2.3 The range of growth conditions explored in m-plane GaN and AlN nucleation layer optimization series.

LAYER	CONDITIONS VARIED
AlN Nucleation Layer	Temperature = 1100 - 1275 °C
	Pressure = 50-760 Torr
	V/III Ratio = 400 – 5500
	Thickness = 50 – 300 nm
HT GaN	Temperature = 1000 °C- 1160 °C
	Pressure = 50-760 Torr
	V/III Ratio = 200 – 3000

The growth studies can be summarized in three major sets of experiments as following:

1) Study the effect of growth conditions with respect to standard reference conditions on AlN nucleation layer grown over m-SiC.

2) Study the effect of changing AlN NL growth conditions with respect to standard reference conditions on GaN epitaxial layer. (with constant GaN growth conditions)

3) Study the effect of change in growth conditions with respect to standard reference conditions on GaN epitaxial layer. (with constant AlN NL growth conditions)

Experiment sets 1 and 2 will be discussed further in section 2.3.1 and 3 will be investigated in detail in section 2.3.2.

To optimize m-plane GaN films, the effects of growth parameters on layer morphology and extended defect density were explored. To characterize the m-plane GaN crystal quality, the full width at half maximum (FWHM) values of ω -scan X-ray rocking curves were measured for the $(1\bar{1}00)$ on-axis with the in-plane a- and c-axis coaxial with incident and scattered wavevectors, and for $(10\bar{1}2)$ off-axis. Only the in-plane a-axis scattering geometry of m-plane films avoids any contribution to the rocking curve width from stacking faults; the issue of peak broadening from stacking faults is still under investigation. Therefore the scattering geometry for on-axis $(1\bar{1}00)$ with the in-plane a- and c-axis, also $(10\bar{1}2)$ off-axis scattering geometries are included in this chapter as relative measure until the issue of peak broadening from stacking faults resolved.

Structural characterization of the m-plane GaN optimization series involved surface morphology and crystallography analysis. Scanning electron microscopy (SEM) images were recorded using FEI Sirion and JEOL 6300 FE-SEM instruments. Atomic force microscopy (AFM) images were recorded using a Digital Instruments D3000 tool operating in tapping mode. The mosaic of the films was determined using a Philips MRD PRO machine in the receiving slit (rocking curve) mode. Transmission electron microscopy (TEM) characterization was carried out using an FEI Tecnica G2 Sphera microscope at an operating voltage of 200 kV.

Although, the substrate quality varied for different batches, there was a self-consistent trend in each experiment. Also, along a given sample, the crystal and surface quality varied from center to edge, hence characterization steps were repeated from the same area of each sample with extra care. In addition, the combined effect of run-to-run variability in MOCVD process should be taken into account. To eliminate the latter effect and to maintain consistency, the reactor conditions were monitored while obeying the same conditioning and bake routine before each growth period.

2.3.1. High temperature AlN nucleation layer

The nature of the AlN NL directly affects the heteroepitaxial GaN microstructure. Therefore AlN NL optimization experiments were performed in two

stages: 1) by growing only an AlN NL on m-SiC substrates with varying growth conditions to demonstrate the effect of change in growth conditions on AlN NL morphology, 2) by growing a 1 μm GaN film with standard growth conditions while changing AlN NL growth conditions to determine the effect of change in AlN NL growth conditions on GaN crystal quality and surface morphology. The effect of growth variables such as reactor pressure, growth temperature, film thickness, and precursor flows are analyzed in the following sections. For all analysis, the change in surface morphology of AlN NL was directly transferred to GaN surface morphology.

2.3.1.1. Effect of reactor pressure

AlN nucleation layers were grown on m-SiC substrates with varying growth pressures. As the reactor pressure lowered, growth rates increased. For the sake of keeping the film thickness the same, the growth times were adjusted accordingly. As displayed in Figure 2.5., with increasing growth pressures and decreasing growth rates, the surface roughness rms values increased up to 100 Torr. However, at 300 Torr the surface roughness dropped down to half. During growth, there was no traceable oscillation with laser monitoring. The growth time was extrapolated according to growth rates from other pressure experiments to maintain the same thickness. After the growth, FILMETRICS measurements were carried out, the AlN NL thickness was determined to be ~ 100 nm instead of 200 nm. Thus the actual growth and/or nucleation rates were much lower than expected for 300 Torr. As observed in thickness variations experiments (see section 2.3.1.4), it is expected to

have lower rms values for thinner AlN films. So the smoother surface morphology at relatively higher pressures can be attributed to thickness.

The film quality had a high tolerance to changes in both NL and GaN layer growth pressures up to 150 Torr as surface roughness increased only from 5 to 8 nm with increasing reactor pressure.

To see the effect of change in AlN NL growth pressure on GaN quality, the AlN NL was grown over m-SiC with growth pressures of 50, 76, 150, and 300 Torr keeping everything else constant, and it was followed by 1 μm GaN growth with the reference conditions disclosed in Table 2.2. As demonstrated in Fig 2.6, an increase in the AlN NL growth pressure, systematically increased the on-axis c mosaic FWHM values for GaN, while on-axis a-mosaic and off-axis twist mosaic remained relatively same up to 150 Torr. However, there was a sudden increase in FWHM values at 300 Torr for on-axis a-mosaic and off-axis twist mosaic.

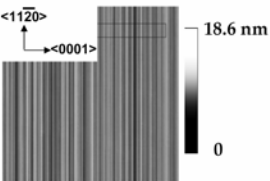
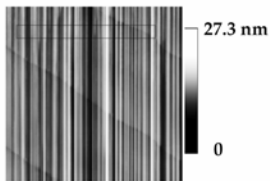
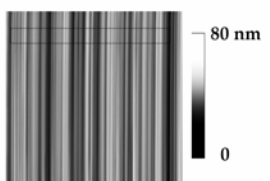
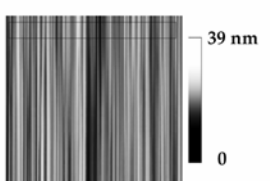
AlN Growth Pressure	AlN NL	Rms
50 Torr		3.173 nm
76 Torr		4.051 nm
100 Torr		12.831 nm
300 Torr		6.639 nm

Figure 2.5 20x20 μm^2 AFM images and surface roughness (rms) values of the AlN NL for varying AlN growth pressures between 50 – 300 Torr.

Integrating surface morphology and crystal mosaic characterization analysis, we conclude that although the AlN NL has a large growth pressure window, the optimum growth pressures for AlN NL were below 100 Torr.

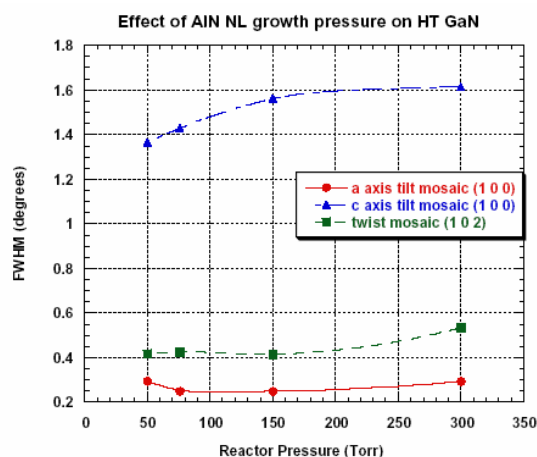


Figure 2.6 The effect of the change in the AlN NL growth pressure on GaN epitaxial film crystal quality. X-ray rocking curve FWHM values for on- and off-axis.

2.3.1.2. Effect of growth temperature

The AlN growth temperature was varied from 1100 to 1275 °C. The growth temperature for AlN NL or GaN had negligible effects on the surface roughness and crystal mosaic of GaN films. Increasing the reactor temperature caused an increase in the surface desorption rate causing the growth rate to decrease. To keep the nucleation layer thickness at 200 nm, the growth time was adjusted depending on the growth rate while simultaneously monitoring the laser trace. As can be seen from Fig. 2.7, although the surface morphology did not change between 1100 -1200 °C, the roughness values increased slightly for temperatures over 1200 °C.

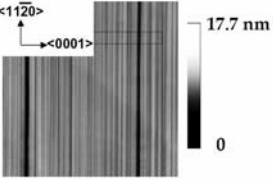
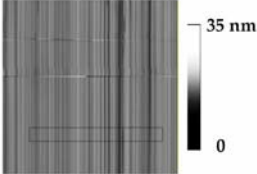
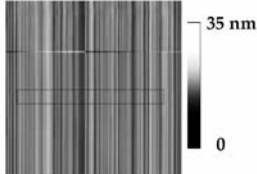
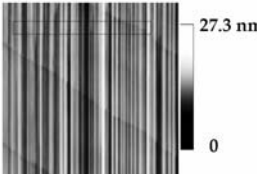
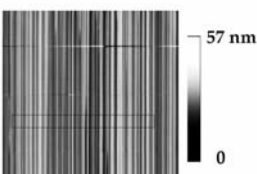
AlN Growth Temperature	AlN NL	Rms
1100 °C		2.44 nm
1150 °C		2.607 nm
1200 °C		2.842 nm
1260 °C		4.051 nm
1275 °C		4.09 nm

Figure 2.7 20x20 μm^2 AFM images and surface roughness (rms) values of AlN NL for varying AlN growth temperatures between 1100 °C- 1275 °C.

To see the effect of change in AlN NL growth temperature on GaN quality, AlN NL was grown over m-SiC with growth temperatures of 1100, 1175, 1225, 1250 and 1275 °C, keeping everything else constant. It was followed by a 1 μm

GaN growth with the reference conditions disclosed in Table 2.2. As is shown in Figure 2.8, all a-axis and c-mosaic tilt mosaic and off-axis twist mosaic FWHM values of GaN followed similar trends such that as the AlN NL growth temperature increased up to 1225-1250 °C, all values decreased, after which they started to rise up again.

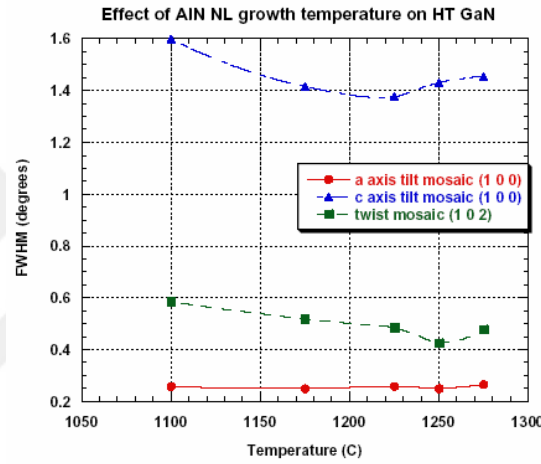


Figure 2.8 The effect of the change in the AlN NL growth temperature on GaN epitaxial film crystal quality. X-ray rocking curve FWHM values for on- and off-axis.

Relatively lower on-axis FWHM values of GaN films were realized at growth temperatures higher than 1175 °C while lower surface roughness values were obtained at lower than 1200 °C. To compromise the competing effects of change in growth temperature on surface morphology and crystal quality, the most appropriate growth temperatures for AlN NL were determined to be in the range of 1175- 1225 °C.

2.3.1.3. Effect of precursor flows

Among all the variables tested for AlN NL optimization, the effect of change in NH_3 and TMA was found to be least effective.

Four different NH_3 flows, 0.5, 1, 4, and 5 slpm, were tested by keeping TMA at 40 sccm and total flow at 6 slpm. TMA flows of 20, 40, and 80 sccm were examined with same NH_3 flow of 4 slpm and total flow of 6 slpm. Typical rms values were in the range of 2.5 – 4 nm. In general there was no distinctive trend of increase or decrease in surface roughness and x-ray rocking curve FWHM values. However, an NH_3 flow of 4 slpm and TMA flow of 40 sccm, which are the flows for reference condition, produced the most desirable results in terms of film quality.

Changes in the AlN V/III ratio between 400 and 5500 had no marked effect on GaN crystal quality or surface roughness. The typical GaN FWHM values with changes in NL V/III ratio were in the range of 0.29-0.32°, 1.25-1.45°, and 0.42-0.49° for a- and c- on-axis tilt mosaic and off-axis twist mosaic, respectively.

2.3.1.4. Effect of nucleation layer thickness

The AlN NL thickness was varied between 50 – 300 nm while keeping all the growth variables constant. Starting with 50 nm and going up to 300 nm thick nucleation layer, AlN islands start off finer and form bigger islands as the films grow thicker. This results in almost 15 times higher roughness values and larger surface undulations in the nucleation layer. These surface undulations and trend in surface roughness continued in the subsequently grown GaN film. As shown in Fig.

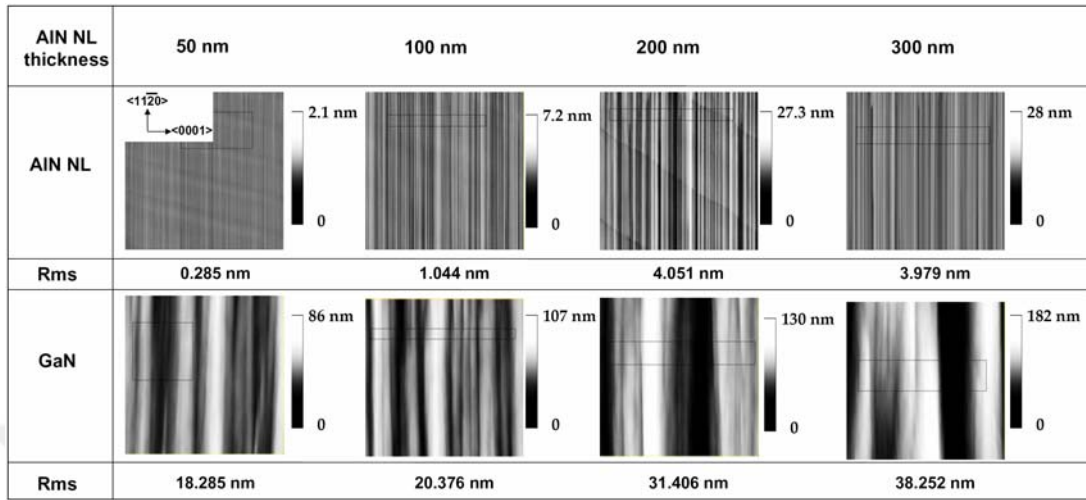


Figure 2.9 $20 \times 20 \mu\text{m}^2$ AFM images and surface roughness (rms) values of the AlN NL and the GaN for varying AlN NL thickness varying between 50 – 300 nm.

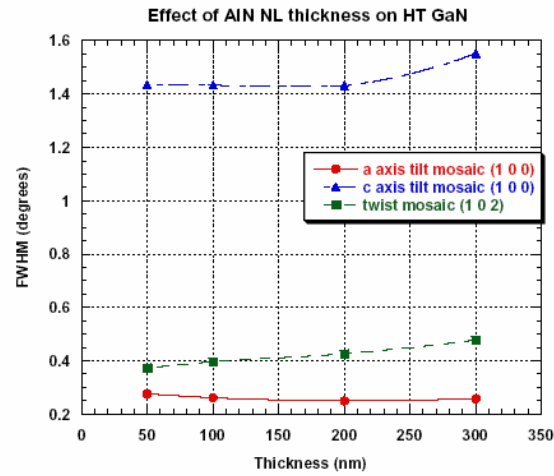


Figure 2.10 The effect of the change in the AlN NL thickness on GaN epitaxial film crystal quality. X-ray rocking curve FWHM values for on- and off-axis.

2.9 and 2.10, the thickness of the nucleation layer islands directly affects the surface and crystal quality of the subsequent GaN layer. The same growth conditions and same number of nucleation sites in the nucleation layer can create different defect structures by only varying the growth time and thickness, i.e. size of the islands.

In summary, optimum AlN NL growth conditions were realized when the AlN growth pressure was below 100 Torr, growth temperature was in the range of 1175 – 1225 °C, the thickness was 50 nm and below, and the V/III ratio was 3500 with 40 sccm of TMA and 4 slpm NH₃ flows.

2.3.2. GaN epitaxial layer

Similar to the AlN NL optimization process, the effect of various reactor pressures, temperatures, and precursor flows were varied to find the optimum growth conditions needed for the best quality m-GaN.

2.3.2.1. Effect of reactor pressure

As mentioned earlier, one of the biggest problems with nonpolar a-plane GaN films was the small growth window in terms of pressure stability. The growth regime for a-plane had to be 76 Torr and lower. Also no dominant growth surface was evident at 300 Torr. As was proved previously in the pressure stability experiment of single step LEO a-GaN, m-plane was more stable at higher pressures over a-plane. The pressure stability window should be expected to be similar for planar m-plane GaN films. So GaN growth pressure was varied between 50 – 300

Torr. The film quality had a high tolerance to changes in growth pressures up to 150 Torr as surface roughness increased only from 5 to 8 nm with increasing reactor pressure (Fig. 2.11). The highest reactor pressure realized without faceting was 300

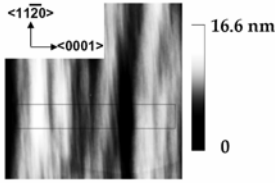
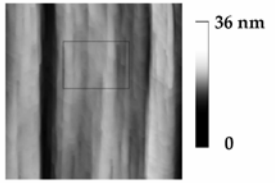
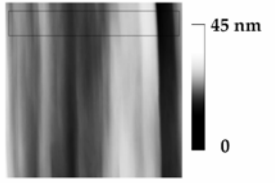
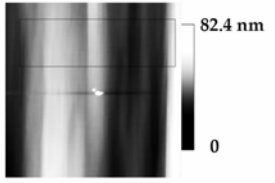
GaN Growth Pressure	GaN	Rms
50 Torr		2.752 nm
76 Torr		6.482 nm
150 Torr		7.915 nm
300 Torr		12.202 nm

Figure 2.11 5x5 μm^2 AFM images and surface roughness (rms) values of GaN epitaxial layer for varying GaN growth pressures between 50 – 300 Torr.

Torr, in contrast to the reactor pressure in excess of 76 Torr that resulted in severe surface faceting in a-plane GaN. With increasing growth pressures, larger surface undulations aligned along the c-axis became prominent. Therefore, surface morphology turned out to be rougher at higher pressures.

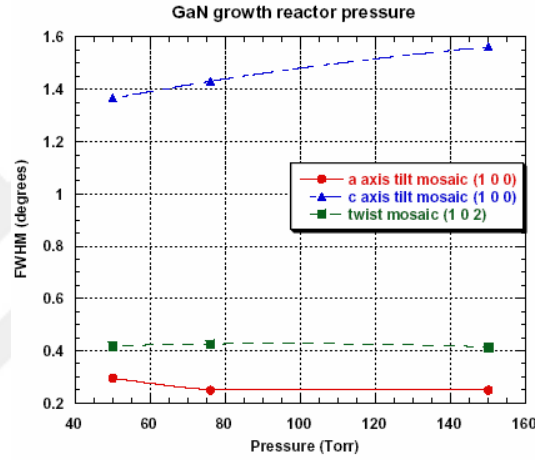


Figure 2.12 The effect of the change in the GaN growth pressure on crystal quality. X-ray rocking curve FWHM values for on- and off-axis.

Pressure does not seem to affect the off-axis twist mosaic or on-axis a-mosaic. On the other hand, c-mosaic FWHM values increased rapidly with increase in pressure. Taking surface morphology evolution and FWHM values into consideration, the optimum growth pressure for GaN films turned out to be 150 Torr and below.

2.3.2.2. Effect of growth temperature

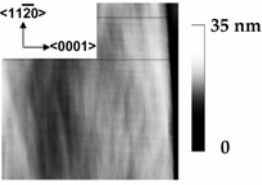
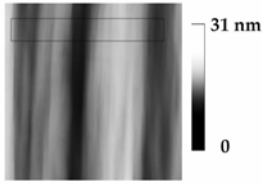
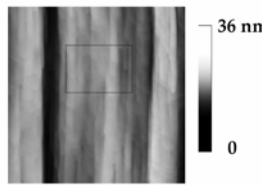
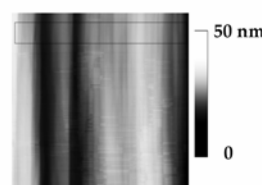
GaN Growth Temperature	GaN	Rms
1000 °C		4.712 nm
1080 °C		6.129 nm
1130 °C		6.482 nm
1160 °C		9.604 nm

Figure 2.13 $5 \times 5 \mu\text{m}^2$ AFM images and surface roughness (rms) values of GaN epitaxial layer for varying GaN growth temperatures between 1000 – 1160 °C.

Change in GaN growth temperature exhibited a proportional increase in surface roughness values. With increasing the growth temperature, the growth rate decreased, larger surface undulations dominated the surface morphology and the roughness increased (Fig 2.13). Surface roughness increased from 4 to 9 nm as the temperature was increased from 1000 to 1160 °C. Figure 2.14 shows an opposite

trend of surface roughness in crystal quality of m-plane GaN for increasing growth temperature. The best FWHM values were obtained at 1130 °C.

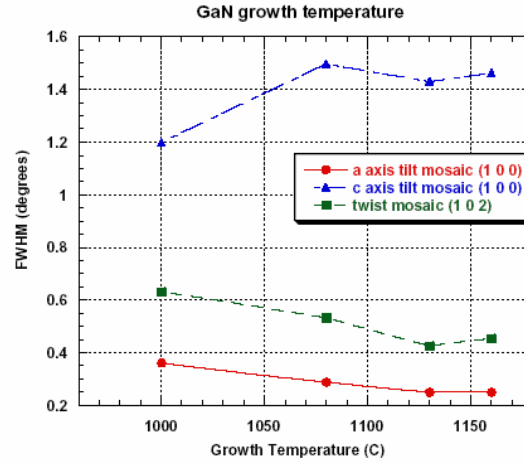


Figure 2.14 The effect of the change in the GaN growth temperature on crystal quality. X-ray rocking curve FWHM values for on- and off-axis.

In selecting the best growth temperature for m-GaN growth, the compromise should be made between differing trends of surface roughness and FWHM values with change in growth temperatures. Which leads to the conclusion as the growth temperature for GaN films should be in the range of 1080-1130 °C.

2.3.2.3. Effect of precursor flows

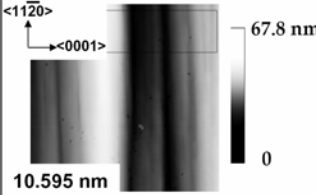
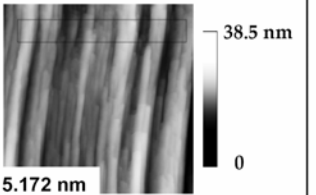
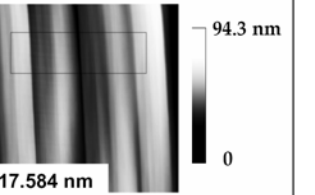
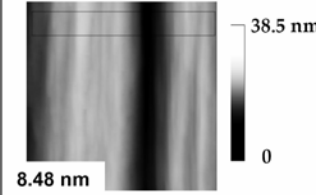
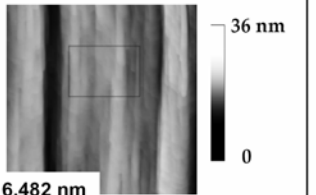
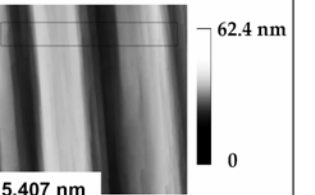
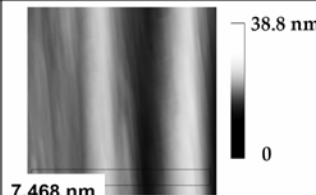
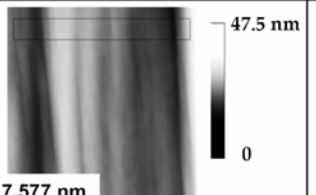
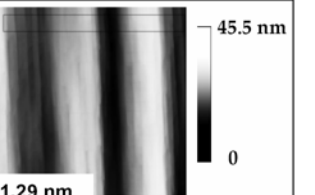
NH_3 / TMGa	0.5 slpm	1 slpm	2 slpm
13.5 sccm			
FWHM	0.2483° / 1.5601° / 0.4393°	0.2226° / 1.5674° / 0.547°	0.2517° / 1.5598° / 0.4473°
27 sccm			
FWHM	0.2688° / 1.7116° / 0.3543°	0.2499° / 1.4301° / 0.3819°	0.2653° / 1.604° / 0.4643°
54 sccm			
FWHM	0.2911° / 1.7077° / 0.5433°	0.2755° / 1.6595° / 0.5127°	0.2673° / 1.6949° / 0.4948°

Figure 2.15 $5 \times 5 \mu\text{m}^2$ AFM images, surface roughness (rms) values, and X-ray rocking curve FWHM values (on-axis a-tilt mosaic / on-axis c-tilt mosaic / off-axis twist mosaic) of GaN epitaxial layer for varying group V (NH_3) and group III (TMGa) flows between 0.5 – 2 slpm and 13.5 – 54 sccm, respectively.

As demonstrated in Fig 2.15, the effect of a wide range of group III and V flows in the range of 13.5 – 54 sccm and 0.5 – 2 slpm, respectively, was investigated. Along the same columns for various group III flows surface morphology exhibited similarities, while in the same rows from left to right with

increasing group V flows, the surface morphologies exhibited an apparent trend. As a general trend, at 0.5 slpm larger GaN islands were formed as an indication of enhanced lateral growth and coalescence of neighboring islands and as the NH_3 flow increased these undulations became finer with smaller island formation as an evidence of suppressed lateral growth. The lowest surface roughness values were accomplished at 1 slpm NH_3 . Therefore the increase in surface roughness at low NH_3 flows, 0.5 slpm, can be associated with long range order undulations while at 2 slpm it can be attributed to short range undulations. For these two extreme conditions, the effect of reduced V/III ratio by increasing only the group III flow reduced the surface roughness values by increased growth rates; however, the crystal mosaic FWHM values degraded. For 1 slpm NH_3 flow changing TMGa flow did not affect the surface roughness significantly.

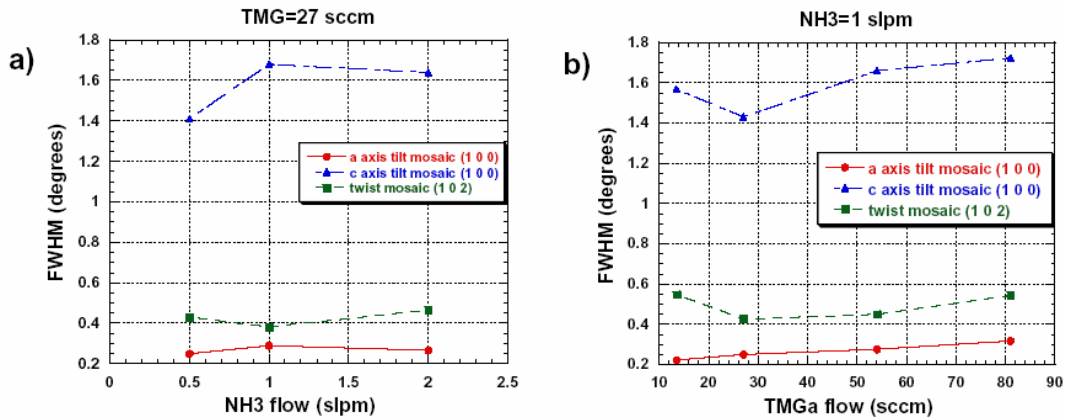


Figure 2.16 The effect of change in a) NH_3 flow for constant TMGa flow of 27 sccm, b) TMGa flow for constant NH_3 flow of 1 slpm on GaN crystal quality. X-Ray Rocking Curve FWHM values for on- and off-axis.

Since the optimum group III and V flow rates were determined to be 27 sccm and 0.5 – 1 slpm from Fig. 2.15, it would be useful to analyze various V/III ratios around these values. For this purpose three different NH_3 flows, 0.5, 1, and 2 slpm, were tested by keeping the TMGa flow at 27 sccm and total flow at 10 slpm. While off-axis x-ray rocking curve FWHM values ramped up with increasing NH_3 , the a-axis tilt mosaic remained unchanged and the c-axis FWHM values decreased. The lowest surface roughness rms value was obtained at an NH_3 flow of 1 slpm. So considering both surface and crystal quality, 0.5 - 1 slpm of NH_3 is the most proper choice to optimize m-GaN.

TMGa flows of 13.5, 27, 54, and 81 sccm were examined with the same NH_3 flow of 1 slpm and total flow of 10 slpm. With increasing TMGa flow, the x-ray rocking curve FWHM values were augmented for on- and off-axis reflections. No significant change was observed in surface roughness values. Therefore taking only improving the crystal quality constraint the optimum choice of TMG flow was determined to be 27 sccm at 0.5 or 1 slpm NH_3 .

As a result, low, controlled, growth rates are favored while making the optimum selection for precursor flows to provide the best quality material. Which is the condition determined as low NH_3 , 0.5 -1 slpm, and TMG, 27 sccm, yielding V/III ratio of ~360 - 720. For GaN V/III ratio, below 1000, the films exhibited a smoother surface morphology and better crystal quality with lower on- and off-axis FWHM values.

In summary, optimum GaN growth conditions were realized when the GaN growth pressure was below 150 Torr, growth temperature was in the range of 1080 – 1130 °C, and the 27 sccm of TMGa and 0.5 -1 slpm NH₃ flows.

2.4. Microstructure and Surface Morphology

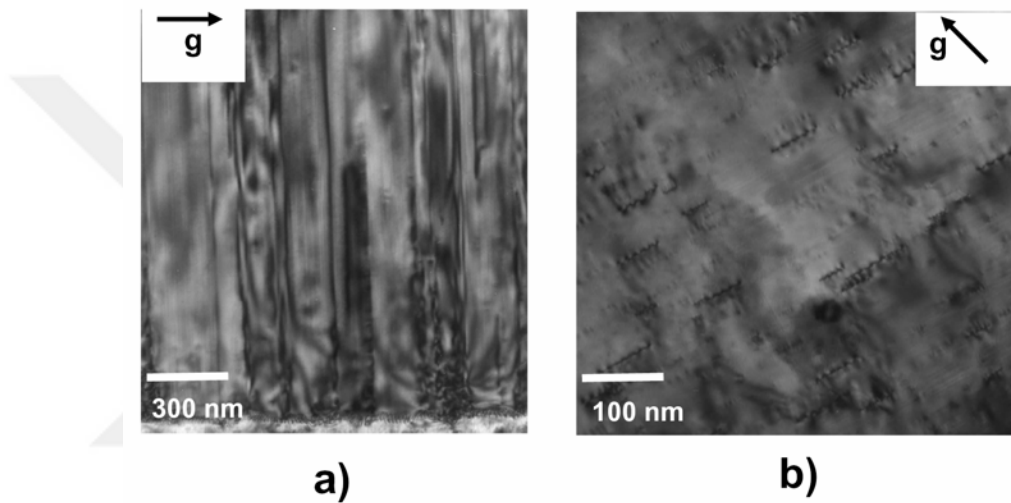


Figure 2.17 a) Cross-sectional and b) Plan-view TEM images of m-plane GaN on m-plane 6H-SiC ($g=0002$).

From plan-view TEM analysis, the threading dislocation (TD) densities and stacking fault (SF) densities were found to be $5 \times 10^9 \text{ cm}^{-2}$ and $8 \times 10^5 \text{ cm}^{-1}$, respectively (Fig. 2.17). These defect densities were slightly larger than HVPE grown m-plane GaN films which were reported as $4 \times 10^9 \text{ cm}^{-2}$ and $1 \times 10^5 \text{ cm}^{-1}$ for TDs and SFs, respectively [24].

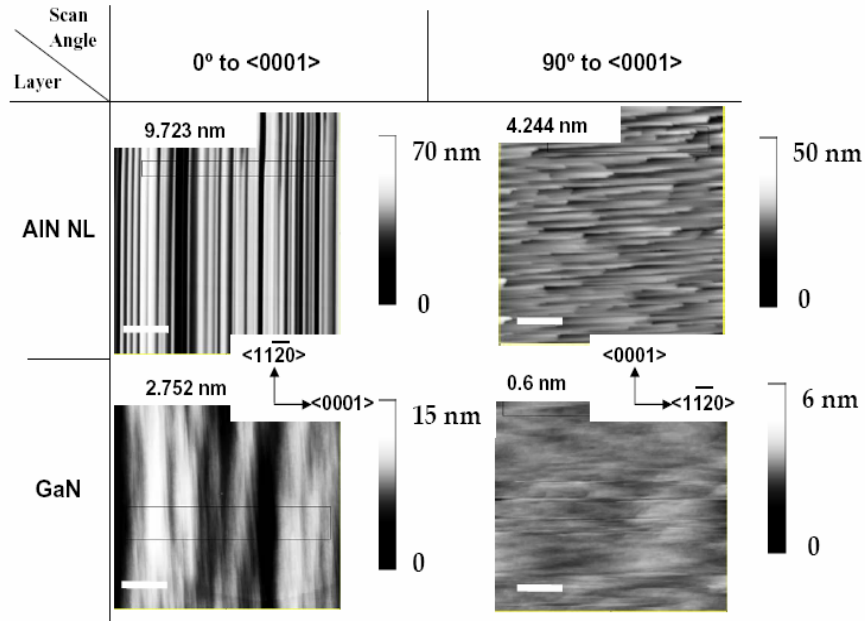


Figure 2.18 The effect of scanning angle in m-GaN and AlN films surface morphology analysis and surface roughness (rms) values. All images are $5 \times 5 \mu\text{m}^2$.

The m-plane GaN films showed undulated surface morphologies. These undulations were aligned along c - $\langle 0001 \rangle$, i.e. perpendicular to $[0001]$, for the m-plane GaN. These undulations were revealed when the surface was scanned 0° to the c - $\langle 0001 \rangle$ direction. If the surface was scanned parallel to these undulations, i.e., 90° to c - $\langle 0001 \rangle$ direction, it was possible to observe terrace –“slate” morphology-formations with smaller rms values avoiding rough undulation features. The “slate” morphology was more defined in AlN films. Therefore, to obtain the absolute surface roughness values, we used the 45° scanning direction with respect to the c -

$\langle 0001 \rangle$ direction, which revealed both undulations and terrace features on the m-plane surface. On the other hand, when making comparative surface analysis for optimization series throughout the chapter, we used the 0° scanning direction with respect to the c- $\langle 0001 \rangle$ direction.

2.5. Structural comparison with nonpolar a-plane GaN on a-plane 6H-SiC

One striking similarity between a-plane and m-plane GaN was the presence of surface undulations. These undulations were detected to be parallel to the c-axis. On the other hand, for a-plane GaN the formation axis of these surface features parallel to the m-axis and perpendicular to c-axis. AFM images in Fig. 2.19 show that the a-plane GaN layer surface was pitted on the sub-micron scale, whereas there was no such observation for the m-plane GaN layer surface. The submicron-scale pits in the a-plane GaN layer have previously been associated with threading dislocations (TDs) [25]. Compared with the a-plane GaN layer grown on a-plane 6H-SiC, the m-plane GaN layer on m-plane 6H-SiC has lower TD density. According to plan-view TEM analysis, for the m-plane GaN layer, the TD and stacking fault densities were $5 \times 10^9 \text{ cm}^{-2}$ and $8 \times 10^5 \text{ cm}^{-1}$, respectively. The m-plane GaN layer has a TD density of one order of magnitude lower than that of the a-plane GaN layer and similar stacking fault density. The on-axis values for the a- and m-plane GaN layers were comparable.

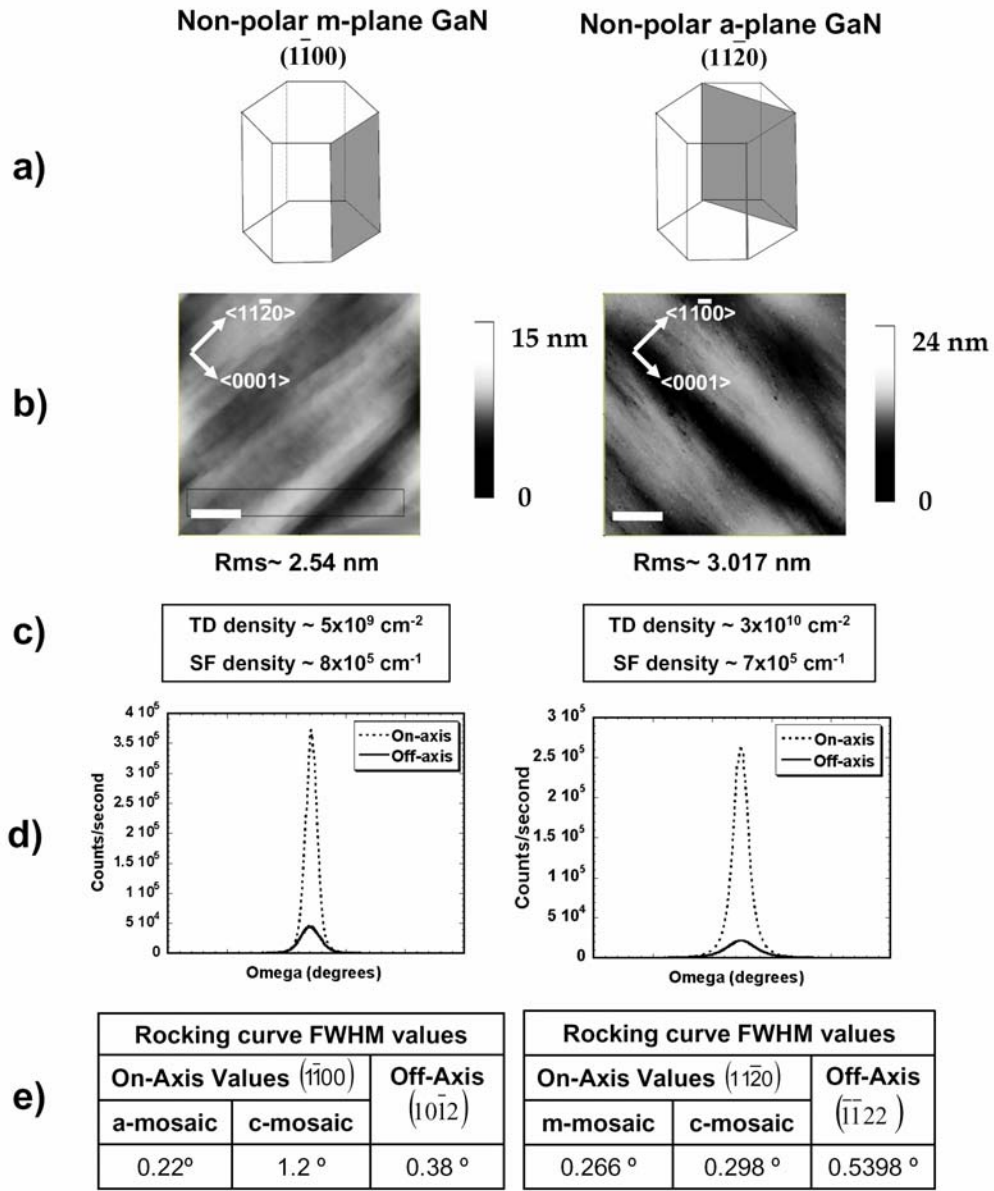


Figure 2.19 Comparison between optimized planar m-plane GaN on m-plane SiC and a-plane GaN on a-plane SiC, from top to bottom: a) crystal planes of interest in unit cell, b) atomic force microscopy (AFM) surface images with surface roughness (rms) values, c) defect densities, d) X-ray rocking curves, and e) FWHM values for on- and off-axis.

Because of anisotropy in the mosaic, the on-axis x-ray rocking curves were found to be orientation dependent around Φ within the same scattering plane as a- and c-mosaic for m-GaN films and as m- and c- mosaic for a-GaN films. Figure 2.19 demonstrates these anisotropic crystal plane geometries as well as the differences and similarities between m-plane and a-plane GaN grown on m- and a-plane 6H-SiC. To characterize the m-plane GaN crystal quality, the full width at half maximum (FWHM) of the ω -scan x-ray rocking curves were measured for the $(\bar{1}\bar{1}00)$ on-axis and $(10\bar{1}2)$ off-axis reflections (the latter was measured in a skew symmetric geometry). Within the same $(\bar{1}\bar{1}00)$ on-axis scattering plane, when $\Phi = 0^\circ$ it represented narrower a-mosaic, and when $\Phi = 90^\circ$ it represented broader c-mosaic, long side of the rectangle. This asymmetry leads to different epitaxial relationship and strains along these two orientations resulting in a c-mosaic to a-mosaic FWHM ratio of ~ 5 . The typical a-mosaic FWHM values were as low as 0.22° while c-mosaic values were 1.2° . In the case of off-axis measurements, FWHM values exhibited a broader peak with 0.38° . While on-axis values for a- and m-plane GaN were comparable the off-axis measurements for m-plane GaN was lower. As Heying et al. demonstrated for c-plane GaN films on c-plane sapphire [25], the off-axis FWHM broadening was explained by the presence of edge dislocations. This also explains the reason as to why lower threading dislocation (TD) densities were observed for m-plane GaN.

2.5. Conclusion

We showed the stability of planar nonpolar $(1\bar{1}00)$ m-plane GaN thin films grown on m-plane 6H-SiC substrates using an AlN NL and device-quality m-plane GaN films were achieved by MOCVD. To optimize the m-plane planar GaN quality with MOCVD, a wide range of growth parameters both for AlN nucleation layer and GaN were tested. Reactor pressure in the range of 40 – 300 Torr, growth temperature in the range of 1000 – 1160 °C GaN and 1100 – 1275 °C for AlN, V/III ratios in the range of 185-1500 for GaN and ~450-5700 for AlN were studied. The surface morphology and crystal quality of the m-plane GaN films were less sensitive to changes in these growth variables than those of nonpolar $(11\bar{2}0)$ a-plane heteroepitaxial planar GaN films. The m-plane, for both AlN and GaN layers, was stable over wide ranges of temperatures, reactor pressures, and precursor flow rates. The most commonly observed off-axis twist mosaic, on-axis a- and c-mosaic values were around 0.45, 0.25, and 1.6°, respectively. According to x-ray rocking curve FWHM results, the best and worst off-axis $(10\bar{1}2)$ twist mosaic values were 0.32688 and 0.6332°, respectively. The lowest on-axis $(10\bar{1}0)$ a-mosaic and c-mosaic values were 0.1937 and 1.1813°, respectively, and the highest on-axis a-mosaic and c-mosaic values were 0.3606 and 1.7241°, respectively. For a huge range in growth variables, the resulting m-GaN quality was relatively stable. So the pressure stability experiments, done with [0001] oriented a-GaN LEO stripes, are justified once again and that m-GaN is more stable in a wider growth range compared to a-

plane GaN. The optimum AlN NL epitaxy conditions, resulting in the best quality GaN films, were realized at temperatures over 1175°C, at relatively low pressures, and V/III ratios of ~3500 with NL thicknesses of 50 nm. For GaN layer epitaxy, the most favorable conditions were realized at low pressures below 150 Torr even though films were stable up to 300 Torr, at temperatures in the range of 1100 - 1160°C, and at V/III ratios below 700 at low NH₃ vapor pressures. A roughness as low as 2.54 nm was achieved under optimized conditions.

Table 2.3 summarizes all the variables carefully examined to optimize the growth condition required to obtain best quality m-plane planar GaN using AlN nucleation layer on m-6H-SiC with MOCVD.

Table 2.4 Optimum growth conditions for best quality m-GaN with MOCVD.

LAYER	OPTIMUM CONDITIONS
AlN Nucleation Layer	Temperature = 1175 °C- 1225 °C
	Pressure = 50-76 Torr
	NH ₃ and TMA= 4 slpm and 40 sccm
	Thickness = 50 nm
HT GaN	Temperature = 1080 °C- 1130 °C
	Pressure = 60-76 Torr
	NH ₃ and TMG= 0.5 - 1 slpm and 13.5-27 sccm

The m-plane GaN is very promising for future non-polar device research. In addition to its stability, m-plane GaN has less defect densities, and it is easier to establish the growth conditions required for LEO and SLEO for defect reduction. The only obstacle is the substrate supply and quality.

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CHAPTER 3:

Sidewall Lateral Epitaxial Overgrowth (SLEO)

3.1. Introduction

Extended defects and polarization related internal electric fields are two major limitations in the performance of III-nitride devices. By growing along the nonpolar directions such as a- $(11\bar{2}0)$ and m- $(\bar{1}100)$ plane the latter can be avoided, however eliminating the extended defect densities is as important to address and solve. Especially in nonpolar III-nitride epitaxial films the extended defect densities are fairly high. The main extended defects are threading dislocations and stacking faults, in current state-of-the-art non-polar GaN films. MOCVD grown a-plane GaN on r-plane Al_2O_3 has a TD density of $\sim 3 \times 10^{10} \text{ cm}^{-2}$ and a basal stacking fault density of $\sim 3.5 \times 10^5 \text{ cm}^{-1}$ [1]. These defects give rise to internal stresses in the material. Threading dislocations (TDs) act as non-radiative recombination centers, i.e. behave as acceptors in n-type material and as donors in p-type material with acceptor- and donor-like localized traps [2,3], and perform as scattering centers for carriers, causing low mobilities [4,5]. They also act as carrier charge sinks, causing ineffective doping [6]. The reduction of dislocation densities causes dramatic drop in the leakage current in p-n junctions [7,8]. Therefore reducing and ideally completely eliminating these defect densities is indispensable to improve upon device performance. Single-step LEO [9-11] and two-step LEO [12] are two of the major methods employed to reduce such defects in non-polar GaN. These two LEO

methods and others will be discussed in section 3.1.2 in detail. Sidewall Lateral Epitaxial Overgrowth (SLEO) combines the advantages of these two LEO methods as being simple and effective at the same time. Essentially SLEO is a technique to reduce the extended defect densities in non-polar III-nitrides in a single re-growth by employing lateral overgrowth from sidewalls of an etched nitride through the mask openings.

3.1.1. Nature of defects in a-plane GaN

The main interest of defects in a-plane GaN films are dislocations and stacking faults.

Dislocations:

The threading dislocation density in planar a-GaN is $\sim 2 \times 10^{10} \text{ cm}^{-2}$. Burgers vectors of the dislocations in planar a-plane GaN were reported as following [1]

- 1) Pure screw dislocations $b = \pm [1\bar{1}20]$
- 2) Pure edge dislocations $b = \pm [0001]$
- 3) Due to reduced symmetry of a-GaN 3 types of mixed dislocations:
 - i) $b = \pm \frac{1}{3} [\bar{1}210]$ or $b = \pm \frac{1}{3} [\bar{2}110]$ or $b = \pm \frac{1}{3} [1\bar{1}20]$
 - ii) $b = \pm \frac{1}{3} [1\bar{1}20] \pm [0001]$
 - iii) $b = \pm [0001] \pm \frac{1}{3} [\bar{1}210]$ or $b = \pm [0001] \pm \frac{1}{3} [\bar{2}110]$

Stacking Faults:

The stacking faults are the errors in usual stacking sequence that preserve original tetrahedral bonding which is AaBbAaBbAaBb along c-direction for hexagonal GaN. Stacking faults are planar defects and lie along the $\{0001\}$ planes of the nitrogen face for a-plane GaN. These defects start at the growth interface and extend to the surface. There are three main types of stacking faults: I_1 , I_2 , and E (extrinsic). When I_1 type stacking faults are present the stacking sequence becomes AaBbAaBbCcBbCc where one atomic plane is misplaced by a displacement of $1/3[\bar{1}100]$ and also $[0002]$ planes are added or subtracted [13]. These type of faults represent the presence of cubic phases embedded in the hexagonal phase. The type of SF explained above is I_1 and it is bounded by Shockley partial dislocation of opposite sign [14]. I_2 type stacking faults involve only the displacement of $1/3[\bar{1}100]$ which results in stacking sequence of AaBbCcAaCc. The “extrinsic” type E stacking faults are formed with the addition of Cc bilayers in normal 2H AaBb stacking sequence which structure the stacking sequence of AaBbCcAaBb. The stacking faults form on the nitrogen $(000\bar{1})$ face for a-plane GaN growth. These defects can only be introduced by either strain in the basal plane or mistakes during nucleation, due to steps on the substrate surface. Also oxygen rich samples were found to be containing I_1 stacking faults [7]. For planar a- $(11\bar{2}0)$ GaN the density of SF was reported as $3.8 \times 10^5 \text{ cm}^{-1}$ [1].

3.1.2. Other defect reduction methods

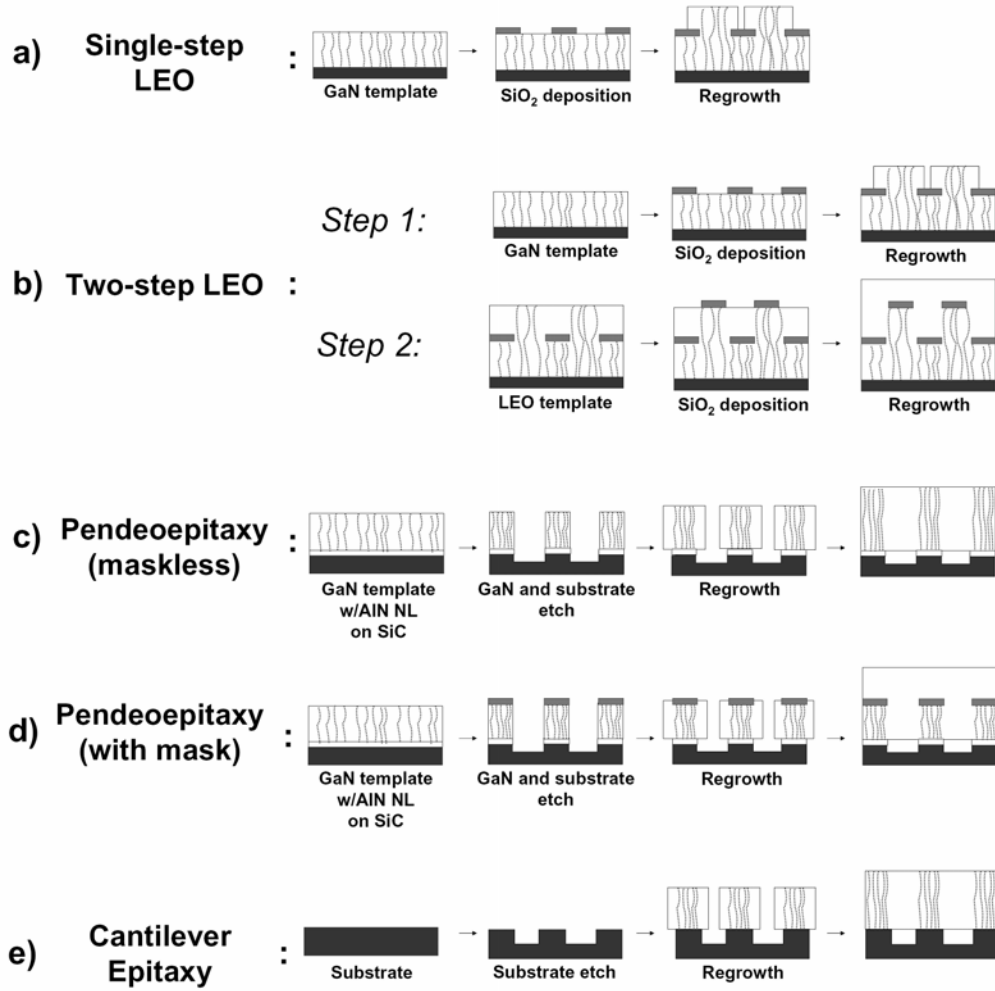


Figure 3.1 Processing and growth schematic of various defect reduction methods for III-Nitrides.

To reduce the defect densities, mainly the dislocations, in polar and non-polar GaN, many methods have been studied over the years. For polar c-plane GaN some of these methods include: single step LEO [26]; air-bridged LEO [27]; facet

controlled LEO (FACELEO) [28]; cantilever epitaxy [29]; LEO from grooved stripe structure [30]; Lateral overgrowth from trenches [31]; pendeoepitaxy [32-34]. The essence of these methods is to block or discourage dislocations from extending to the film surface by favoring lateral growth over vertical growth with assistance of a mask medium. Figure 3.1 shows the processing and growth schematics of the most commonly used methods among those mentioned above.

Single-step lateral epitaxial overgrowth (LEO) (Fig 3.1 a)) and two-step LEO (Fig 3.1 b)) are the most commonly used methods for defect reduction in III-nitrides including the non-polar orientations. Single-step LEO is simple to process and grow and two-step LEO is effective in improving film quality however it is labor and time intensive due to the fact that it utilizes single-step LEO processing and growth steps two times as the name implies.

Figure 3.1 d) and c) shows the Pendeoepitaxy methods with and without mask, respectively. Original pendeoepitaxy was developed for its maskless technique. The fundamental of pendeoepitaxy is to use a template with a substrate which is utilized as a *pseudomask* so that the growth is accomplished only on the exposed pillar surfaces. Pendeoepitaxy with mask yields similar results to the two-step LEO. Although it is not as much elaborative as two-step LEO, it requires SiC substrate which is relatively expensive and involves an extra step of template and substrate etch.

Maskless Pendeoepitaxy (Fig. 3.1 c)) and cantilever epitaxy (Fig. 3.1. e)) also yielded similar results with only one difference in the processing and growth steps; while cantilever epitaxy the substrate is patterned and etched before initiating

the growth in Pendeoepitaxy the template is grown first then it is patterned and etched along with the substrate before regrowth.

The methods used to reduce defects for non-polar GaN include: single-step LEO [9]; and selective area LEO (SALEO) [12] which is similar to two-step LEO. M. D. Craven et al. [24] conducted a detailed study on a-plane single-step LEO of GaN films which was used as the building blocks of SLEO growth studies. With the single step LEO technique for non-polar a-plane GaN the effect of stripe orientation was explored with a wagon-wheel pattern. Accordingly only $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ and $\langle 0001 \rangle_{\text{GaN}}$ oriented stripes yielded homogeneously grown pillars from the seed region. However as shown in Fig. 3.2, while $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ stripes creating vertical sidewalls, $\langle 0001 \rangle_{\text{GaN}}$ oriented stripes formed faces composed of a- and m-facets. As $\langle 0001 \rangle_{\text{GaN}}$ had stubborn facets that would not coalesce independent of growth time, $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ with vertical sidewalls was better suited in order to achieve coalescence. Moreover as Fig. 3.3 reveals c- direction oriented stripes created m-plane facets causing the dislocations to bend and spread over the wing regions, the straight sidewalls created by m-direction oriented stripes making the dislocations confined in the window region. Therefore, by using $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ direction in SLEO the dislocations will be confined under the mask and only in the seed region.

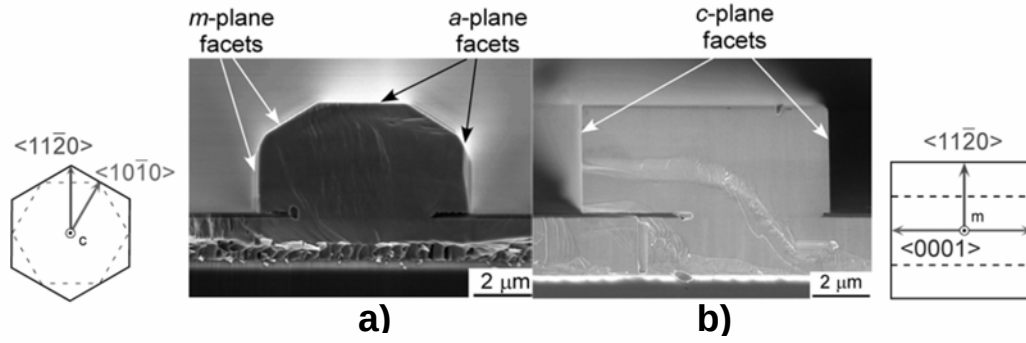


Figure 3.2 Cross-sectional SEM image of overgrowing a-plane single-step LEO GaN for a) $\langle 0001 \rangle_{\text{GaN}}$ oriented, and b) $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ oriented stripes. [24]

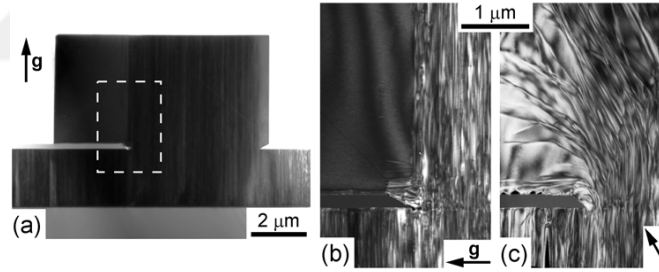


Figure 3.3 The effect of stripe orientation in a-plane single step LEO GaN. Cross-sectional TEM images of a) overgrowing complete LEO island for $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ oriented stripes ($g=11\bar{2}0$), and magnified views of mask edge for b) $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ oriented stripes ($g=0006$) and c) $\langle 0001 \rangle_{\text{GaN}}$ oriented stripes ($g=01\bar{1}0$). [9]

The stacking faults on the basal plane were associated with growth on exposed $(000\bar{1})$ N-faces. The Ga-face growth rate was ~10 times faster than the N-face growth rate and thus confined the high stacking fault density material to a region near the N-face of the window. However, single step LEO resulted in defect reduction only in the overgrown wing regions. Therefore, mask alignment for device processing was an issue due to presence of defects in the window (seed) regions in the single-step LEO material. With two-step LEO it was possible to reduce the defect densities effectively throughout the film. However, two-step LEO involves three growth steps and two processing steps. The technique described herein, sidewall lateral epitaxial overgrowth (SLEO), utilizes the advantages of these two LEO techniques as being simple and effective at the same time.

Advantages of sidewall lateral epitaxial overgrowth (SLEO) over other methods can be summarized as:

- Can be executed on GaN epitaxial films grown over Al_2O_3 which is comparably cost effective.
- Time and labor efficient.
- Provides wing region quality throughout the material so the whole surface area can be used for device processing.

3.2. Evolution of SLEO

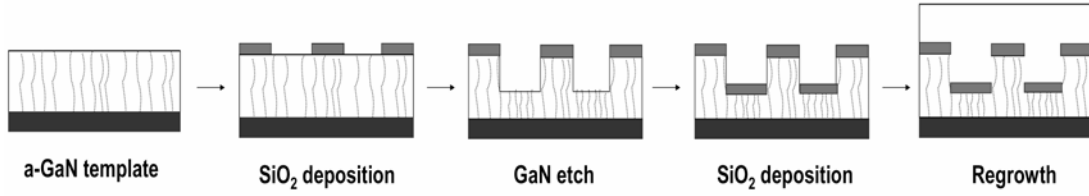


Figure 3.4 Processing and growth schematic for early version of SLEO.

The development of SLEO went through two phases. In the early stages the processing flow was formed as shown in Fig. 3.4. Instead of etching deep trenches the material was etched $\sim 1\text{-}2\text{ }\mu\text{m}$ deep, then the bottom of the trenches was covered with a mask (SiO_2). Hence the etching step in processing scheme became extremely important. At that point planar and any LEO growth of c-plane GaN was well understood and more mature than any nonpolar crystallographic direction growth. Therefore the first trial was executed on c-plane GaN templates. Figure 3.5 exhibits this first attempt of SLEO efforts in its primitive stage. Despite all the meticulous efforts and careful planning, the growth initiation from sidewalls was either extremely inhomogeneous or none at all due to partial or complete coverage of sidewalls with mask material.

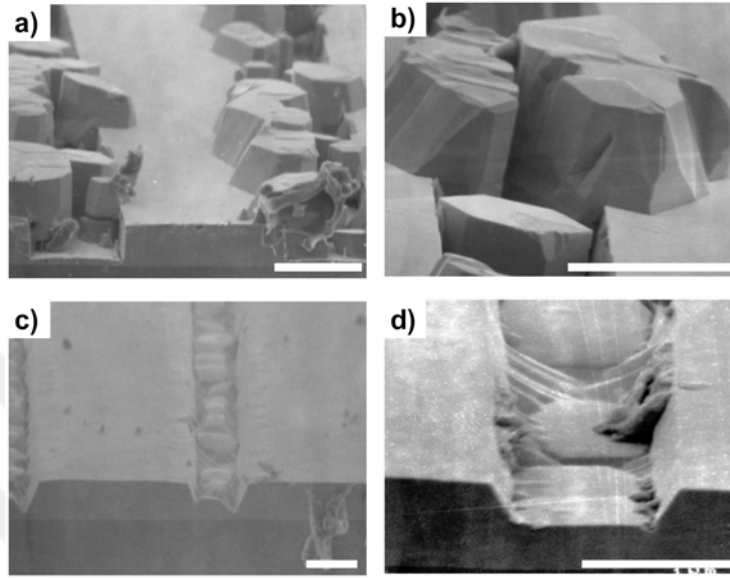


Figure 3.5 Cross-sectional SEM image of c-GaN growth from sidewalls in early version SLEO (Scale bar = 5 μm): a) 3000x, b) 6000x, c) 1500x, and d) 5000x.

Since deposition of SiO_2 was desired only at the trench bottoms. Any mask deposition on the sidewalls was intolerable. The sidewalls had to be perfectly perpendicular if not undercut and in any of these cases reproducibility would be a big issue. Therefore it was decided to remove the mask deposition at the trench bottom, instead the pillars would be created with deep etching and the material would be attempted to coalesce with increased lateral growth rates sustaining proper growth conditions. The final decision formed the basis for principles of SLEO as described in the following sections.

3.3. Experimental Procedure

3.3.1. Template Growth

The templates were grown with MOCVD on double side polished Kyocera r-plane Al_2O_3 substrates using ~25 nm thick low temperature GaN nucleation layer followed by high temperature GaN heteroepitaxial film. The thickness for the heteroepitaxial film a-GaN was varied between 3 μm -6 μm for different mask patterns. The detailed growth and substrate anneal conditions for the template is shown in Table 3.1.

3.3.2. Processing

The a-plane GaN templates were grown in a close-spaced vertical rotating disk MOCVD reactor on double side polished r-plane Al_2O_3 substrates using conditions described in Table 3.1. After growth, the template was solvent cleaned and let sit in the dehydration oven at 120°C for 15 minutes prior to SiO_2 deposition to prepare a suitable base for mask deposition by removing any left over moisture and solvents from the surface. The main processing steps involved mask deposition and patterning, mask etching, and GaN etching, as shown in Fig. 3.6.a) The process flow was similar to single-step LEO except for the addition of the etching of exposed GaN through the mask openings. The SiO_2 mask was deposited using plasma enhanced chemical vapor deposition (PECVD). A 3 μm and 6 μm thick a-plane GaN template was used for 2 μm / 8 μm (window / wing) and 5 μm / 15 μm LEO

Table 3.1 a-plane GaN template growth conditions on r-Al₂O₃

Parameter	
r-Al₂O₃ anneal	
Anneal Temperature	1160 °C
Anneal Duration	1200''
Total Flow (in H ₂)	5.4 slpm
Reactor Pressure	~50 Torr
Low Temperature GaN Nucleation Layer	
Growth Temperature	650 °C
Growth Duration	41''
Total Flow (N ₂ balance)	5.0 slpm (5.4)
Reactor Pressure	760 Torr
TMGa Flow	26.9 sccm
NH ₃ Flow	3.6 slpm
V/III Ratio	~2653
High Temperature GaN	
Growth Temperature	1160 °C
Total Flow (N ₂ balance)	10.0 slpm (10.7)
Reactor Pressure	60 Torr
TMGa Flow	27.0 sccm
NH ₃ Flow	1.0 slpm
V/III ratio	~734
Growth Rate	~5.7 Å/s

stripe patterns, respectively. The template thickness was chosen to be at least as large as the window width of the LEO stripe pattern. The stripe orientation was parallel to the $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ direction (due to the vertical c-plane sidewalls in the lateral overgrowth [9]) and was transferred using conventional photolithography

techniques. The mask patterning was followed by etching of SiO₂ using inductively coupled plasma (ICP) etching through the windows to the GaN epitaxial film. Finally, reactive ion etching (RIE) was used for GaN etching through the mask openings, not necessarily down to the r-plane sapphire substrate, thus exposing Ga-face (0001) and N-face (000 $\bar{1}$) planes on the sidewalls and (11 $\bar{2}$ 0) plane at the bottom of the trenches. The etch depth was chosen such that it was greater than the window width so that there was sufficient space for the material growing laterally from the sidewalls to coalesce before the heavily defected material growing from the bottom reached the surface. After the nitride etch, the processed samples were loaded into the reactor for regrowth.

3.3.3. Regrowth

As demonstrated in Fig. 3.6 b), the SLEO growth involved three main stages: the first stage - “SIDE”; the second stage - “UP”; and the third stage - “OVER” growth. For all three stages, high lateral to vertical growth rate ratios were preferred.

The first, “SIDE”, stage involved growth predominantly from the exposed Ga-face sidewall until coalescence with the opposite N-face sidewall – this stage suppressed growth from the bottom of the trench by starving the bottom of precursor species. The N-face formed straight sidewalls with hexagonal hillock features. The Ga-face sidewall formed an arrowhead shape with inclined $\{11\bar{2}n\}$ facets. While analyzing the growth from sidewalls with SEM and TEM images, it is useful to

know how to identify the faces by looking at them. A useful study done by Mike Craven et al. [25]: a-GaN LEO from the circular mask openings reveals necessary

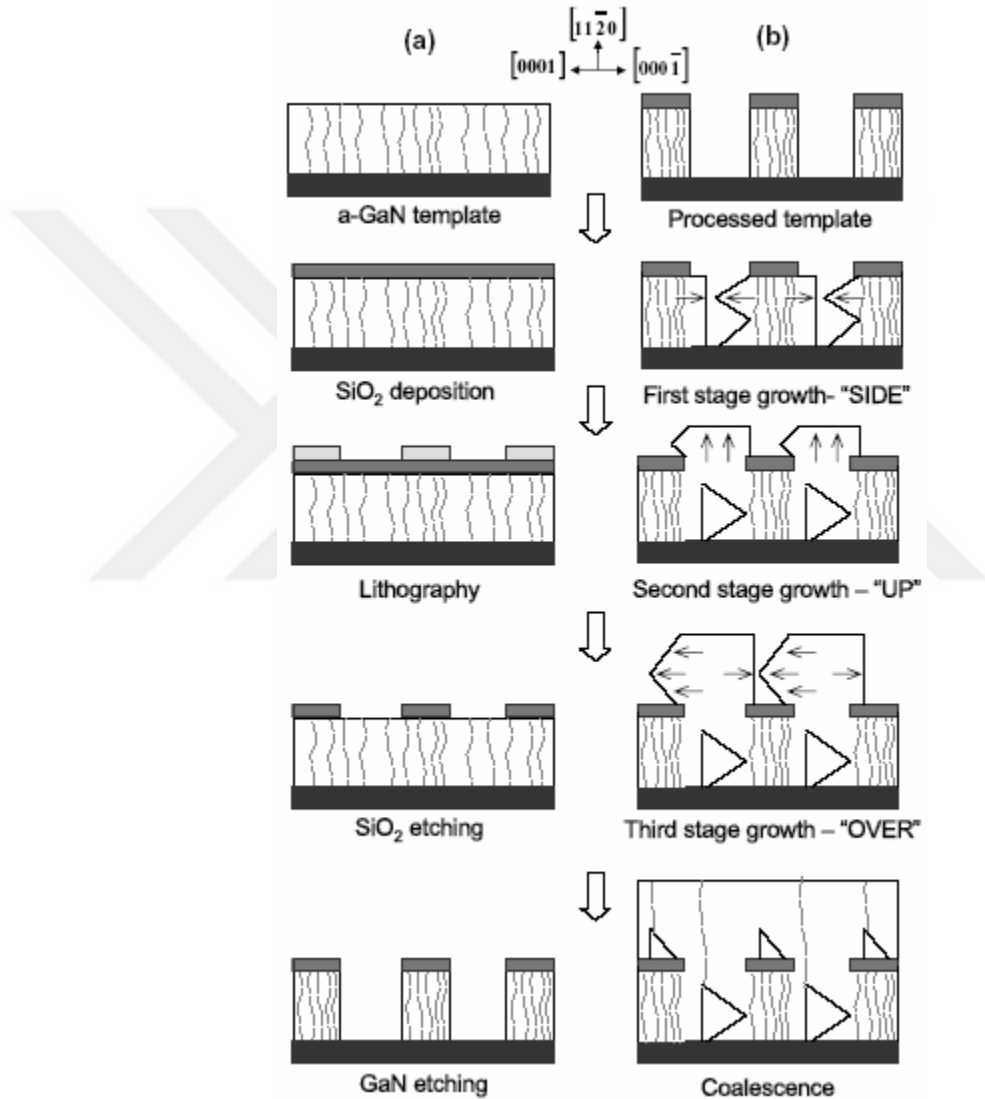


Figure 3.6 a) Processing sequence of the a-GaN template for subsequent sidewall lateral overgrowth. b) Schematic of the growth stages of sidewall lateral epitaxial overgrowth (SLEO).

information for the formation of the facets in different directions. According to the SEM image shown in Figure 3.7, N face forms straight sidewalls with hexagonal hillock features, and the Ga face forms an arrowhead shape with inclined $\{\bar{1}101\}$ facets.

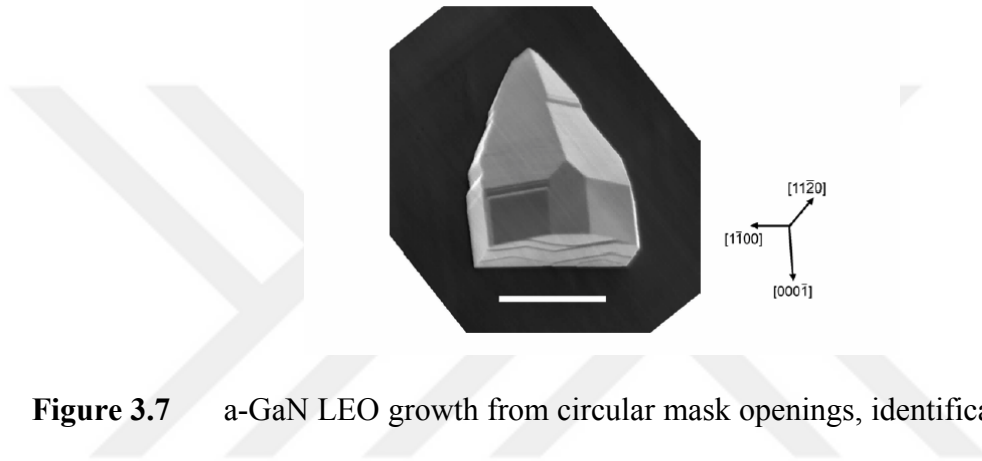


Figure 3.7 a-GaN LEO growth from circular mask openings, identification of the faces (scale bar = 5 μm) (Courtesy of Mike Craven [25])

SLEO growth modes involve three main stages as shown in Fig. 3.6 b). In this study all stages were examined separately by changing the growth variables such as precursor flows; NH_3 and TMGa, reactor pressure, growth temperature, and rotation rate as well as the stripe width. The standard growth parameter used as a reference condition is given in Table 3.2. Any other growth variables changed by keeping the rest of the variables constant as in reference conditions.

The main concerns while analyzing the results for different stages of growth were competing lateral and vertical growth rates, relative lateral growth rates of Ga

and N face, sidewall morphology, homogeneity and smoothness, and coalesced material defect structure and surface morphology for all growth stages.

Table 3.2 Standard reference growth conditions

Parameter	
Growth Temperature (TC)	1160 °C
TMGa flow	27 sccm
NH3 flow	1.0 slpm
V/III ratio	~734
Carrier Gas	H2
Total Flow/ Nitrogen balanced	10.0/ 10.7 slpm
Total Pressure	60 Torr
Rotation Per Minute (rpm)	200

After upon completion of each stage and after coalescence, the samples were characterized by atomic force microscopy (AFM) for surface morphology; x-ray rocking curves for crystal mosaic; transmission electron microscopy (TEM) and cathodoluminescence (CL) for extended defect densities and distribution, and scanning electron microscopy (SEM) to monitor physical evolution. AFM images were recorded with a Digital Instruments D3000 in tapping mode. Crystallographies of as-grown films were determined by using Phillips MRD PRO in receiving slit (rocking curve) mode. 1 mm slit on the detector arm was used. In order to characterize the crystal quality omega x-ray rocking curves were measured for on- $(11\bar{2}0)$ and off- $(10\bar{1}1)$ axis reflections. The a-plane crystal geometry is anisotropic in plane along $c-\langle 000\bar{1} \rangle$ and $m-\langle 1\bar{1}00 \rangle$ directions, therefore two distinct on-axis

reflections were taken into consideration along these orientations to measure the c- and m-mosaic, respectively. For cross-section and plan-view TEM imaging, samples were polished manually by wedge polishing and followed by ion milling for electrical transparency in Gatan precision ion polishing system. TEM characterization was carried out with FEI Tecnia G2 Sphera Microscope at operating voltage of 200 kV. The digital TEM images were taken with FEI Tecnai G2 F30 FEG Microscope operating at 300 kV. Both for scanning electron microscopy (SEM) and cathodoluminescence (CL) analysis the samples were coated with Au. SEM images were recorded with an FEI Sirion and FEOL 6300 FE-SEM instruments. CL images were obtained at room temperature with an operating voltage of 5 kV using a Gatan MonoCL system.

3.4. 1st stage growth

The quality of the sidewall coalescence in the first growth stage was the determining factor in the quality of the final coalesced material. The defects present after first stage coalescence were present in the window regions of the final coalesced material. In the first stage, Ga-face (0001) and N-face ($000\bar{1}$) planes on the sidewalls and ($11\bar{2}0$) plane at the bottom of the trenches were exposed to growth. To achieve optimum defect reduction, it was essential to control the growth rates of these exposed planes. The N-face formed straight sidewalls with hexagonal hillock features and the Ga-face sidewall formed an arrowhead shape with inclined

$\{11\bar{2}n\}$ facets. The polarity of the Ga- and N-faces was determined previously by using convergent beam electron diffraction (CBED) analysis [25]. Stacking faults were localized in N-face material, and the growth at the bottom of the trenches was typically defective a-plane material. It was essential to coalesce the sidewalls sufficiently fast to prevent the defected material growing from the bottom of the trenches. In addition, the Ga-face growth rate had to be fast enough to achieve the coalescence at the same time being slow enough to preserve the crystal quality and to sustain uniform growth. To achieve these goals, various pressure, temperature, and V/III ratios were optimized. The standard growth condition was 27 sccm TMGa and 1.0 slpm NH_3 flow with V/III ratio~734 at 60 Torr and 1160°C in total 10.0 slpm of H_2 carrier gas as given in Table 3.2. While changing one variable the rest of these variables were kept nominally constant at standard growth condition. The growth was interrupted after 900 s for characterization.

First stage growth was liable for the quality of the coalesced material. No matter how properly the consequent growth stages are executed, if the initial growth is not created appropriately the defect propagation will be inevitable. Therefore analysis should be done with extreme care.

3.4.1. Pressure:

Due to high defect densities in planar a-plane GaN, growth stability of these films was achieved at low pressures [24]. The pressure dependency experiments for

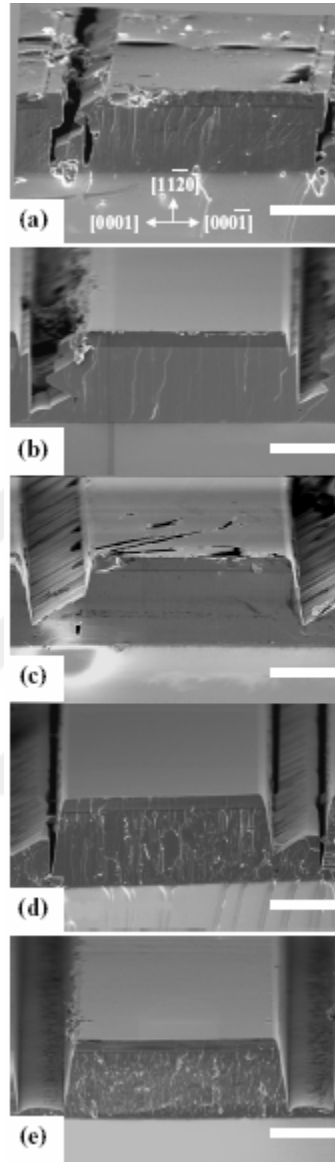


Figure 3.8 Scanning electron microscopy images for the effect of growth pressure in the first stage coalescence of SLEO (scale bar = 5 μm), (a) 40 Torr, (b) 60 Torr, (c) 100 Torr, (d) 150 Torr, (e) 760 Torr

the first stage SLEO yielded similar results. In this experiment series, the growth pressure was varied in between 40 – 760 Torr (Fig. 3.8 a-e) while keeping all other

growth parameters constant. At low reactor pressure reacting species are more directional and forced into the trenches, the growth rate from the bottom of the trench was comparable with the lateral growth rate from the sidewalls. Additionally the N-face and the Ga-face growth rates competed with each other from the sidewalls.

As the pressure was raised to 60 Torr both lateral and vertical growth rates decreased and Ga-face growth rate dominated N-face growth rate. It can be seen in Fig. 3.8 c) that when the reactor pressure was increased to 100 Torr the growth was only observed from the trench bottoms with inclined $(1\bar{1}01)$ facets. Similarly at 150 Torr the lateral growth disappeared completely and growth from trench bottom was isolated with more defined $(1\bar{1}01)$ facets and exposed $(000\bar{1})$ face. At 760 Torr there was no lateral growth or growth from the trench bottom. These results demonstrate that the optimum growth pressure favoring controlled sidewall coalescence was 60 Torr.

3.4.2. Temperature:

Various growth temperatures in the range of 1100-1190°C (Fig. 3.9 a-d) were tested for the first stage SLEO. The N- and Ga- face growth rates increased with increasing temperature. At 1100°C the growth from the sidewalls was initiated randomly and was not homogeneous. As the temperature was raised to 1130°C (Fig. 3.9 b) while the Ga-face growth rate increased N-face growth stayed

inhomogeneous. It is shown in Fig. 3.9 c) and 3.9 d) that the most favorable condition to achieve fast

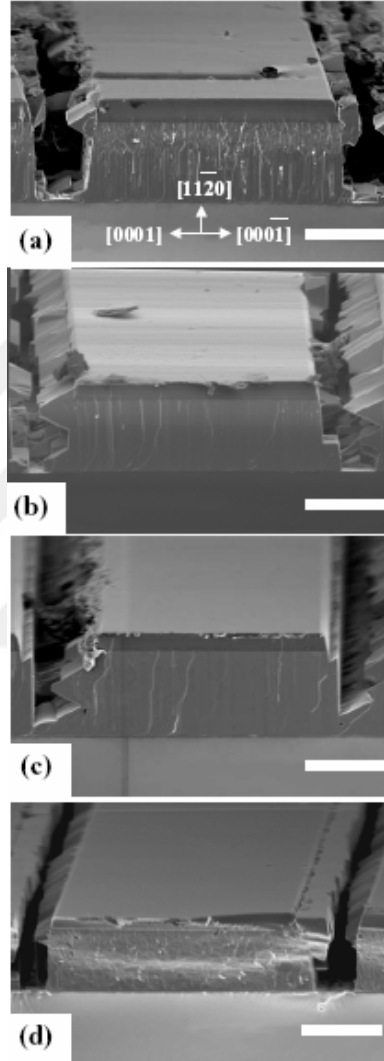


Figure 3.9 Scanning electron microscopy images for the effect of growth temperature in the first stage coalescence of SLEO (scale bar = 5 μ m), (a) 1100 °C, (b) 1130 °C, (c) 1160 °C, (d) 1190 °C

and controlled coalescence were realized at relatively high temperatures in between 1160 – 1190 °C. At 1160 °C growth from all exposed surfaces was accomplished homogeneously. As the temperature was raised to 1190 °C while the N-face growth rate increased significantly, the vertical growth rate at the trench base diminished due to higher mobility of species at high temperatures.

3.4.3. NH_3 and TMGa:

The V/III ratio is a significant variable to control the relative growth rates from the sidewalls and trench bottom. Figure 3.10 shows SEM images for cleaved cross-sections in the first stage growth - each column is for constant group III flow with increasing the group V flow from 0.5 slpm at the top to 3 slpm at the bottom image, and each row represents constant group V flow with increasing group III flow from 13.5 sccm at the left to 81 sccm at the right image. Each image also is labeled with the V/III ratio. Along the diagonal the V/III ratio is constant at ~730 as the group III and V flow increase from top-left corner to the bottom-right corner the growth rate from the sidewalls and the trench bottoms increased proportionally. When the V/III ratio was reduced, the lateral growth rates increased at constant NH_3 flow along the same row. Similarly when the V/III ratio increased at constant TMGa flow, the vertical growth rate from the trench bottoms increased. At 0.5 slpm NH_3 flow, the growth from the sidewalls was inhomogeneous and Ga-face growth rate was significantly higher than N-face growth rate. Also as the TMGa flow increased to 81 sccm the growth rate increased sufficiently high to coalesce the sidewalls. At

81 sccm TMGa flow at all different group V flows the growth rates were high enough to achieve coalescence from the sidewalls. As the NH_3 flow increased along

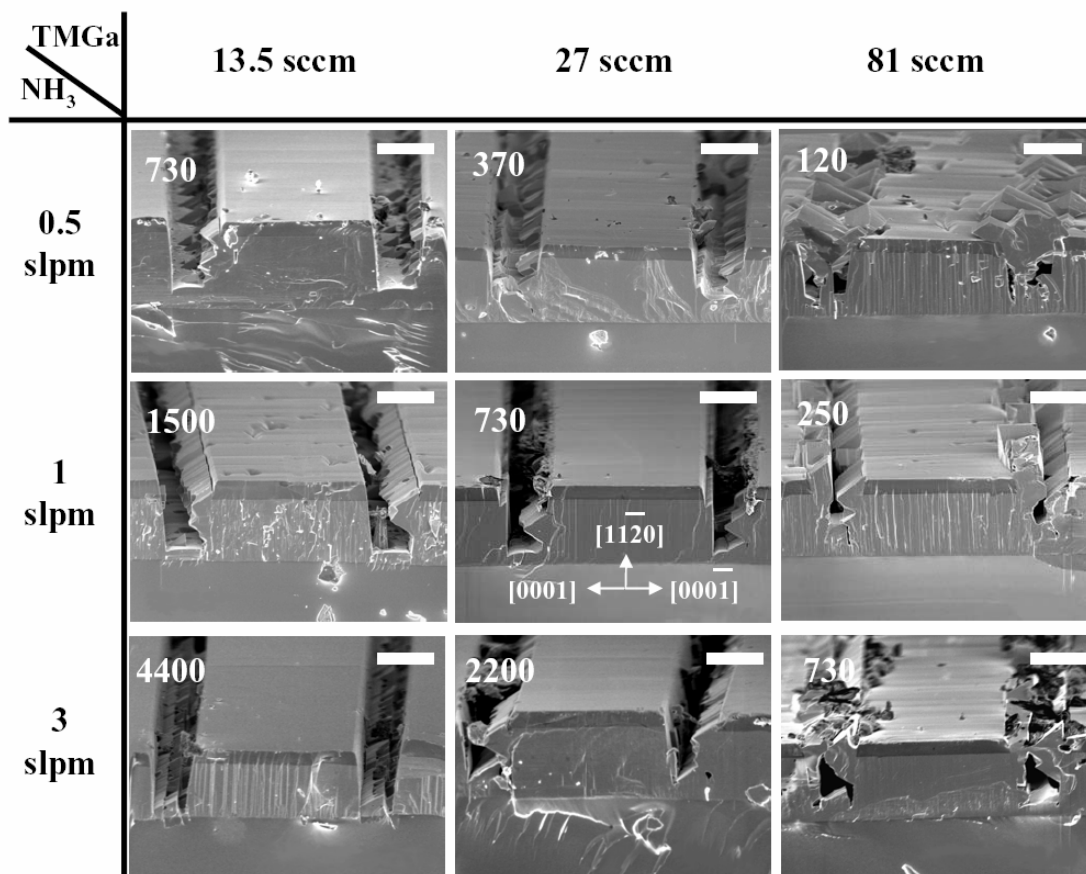


Figure 3.10 Scanning electron microscopy images for the effect of V/III ratios in the first stage coalescence of SLEO. The V/III ratios are indicated on the each corresponding SEM image. (Scale bar = 5 μm)

the same 81 sccm column, it enhanced the N-face growth. However, if the lateral growth rates were too high, the coalescence was inhomogeneous leading to poor film quality. At 3 slpm NH_3 the characteristics of the lateral sidewall growth was similar

to what was observed at 0.5 slpm flow except at 3 slpm NH_3 flow the vertical growth from the bottom of the trenches become more noticeable. At 1 slpm NH_3 flow as the group III flow was raised the growth rate from all exposed surfaces increased. The most viable first stage growth conditions were achieved at 1 slpm NH_3 flow. So a V/III ratio of ~300-700 with 1 slpm NH_3 balance was sufficient to realize high and uniform lateral growth rates from the sidewalls.

While too high growth rates led to irregular sidewall morphologies, too slow growth rates were not sufficient to accomplish coalescence. For the same V/III ratio sufficient growth rates are required to provide controlled coalescence. V/III ratio appears to be the key to accomplish better crystal morphology and improved defect structure for following stage growths and final coalesced material. Quality of the final coalesced material depends on how the growth initiated in the first stage. Therefore the lateral growth rates in the first stage should be sufficiently high to provide coalescence, and slow enough to control the defect propagation. As evidence, Table 3.3 compares two coalesced samples defect structure. Both samples were coalesced with the same conditions except for the V/III ratio used in the first stage. The second sample, N041227CD, was grown at relatively high lateral growth rates with NH_3 flow of 1 slpm and TMGa flows of 81 and 54 sccm, while the first sample, N050123D was grown with the same NH_3 flow of 1 slpm but lower TMGa flows of 27 and 40.5 sccm. As a consequence the first sample with controlled growth rate had one order of magnitude lower TD and SF densities compared with the second sample which had higher growth rates.

Table 3.3 The effects of V/III ratio in the first stage growth conditions on defect densities.

Sample Number	Growth Conditions	TD density	SF density
N050123D	Stage I: V/III~ 734-490 T= 1200 °C P=60 Torr	$3\text{-}4 \times 10^7 \text{ cm}^{-2}$	$6 \times 10^4 \text{ cm}^{-1}$
N041227CD	Stage I: V/III~360-245 T=1200 °C P= 60 Torr	$5 \times 10^8 \text{ cm}^{-2}$	$1 \times 10^5 \text{ cm}^{-1}$

3.4.4. Rotation:

Figure 3.11 demonstrates the effect of rotation rates (per minute – rpm) for 100, 200 and 300 rpm in the first stage sidewall growth. Higher rotation rates lead to lower V/III ratio due to giving not enough time for NH_3 to crack. Low rotation rates give more time for the species to reach down to the bottom of the trenches. As the rotation rate was increased the growth was dominated at the top of the sidewalls. If the rotation is raised above 200 rpm, the sidewall morphology homogeneity was affected negatively. Therefore median rotation rate of 200 rpm was preferred for the first stage. The importance of rotation comes into play mostly in the third growth stage which we will discuss further in section 3.5.5.

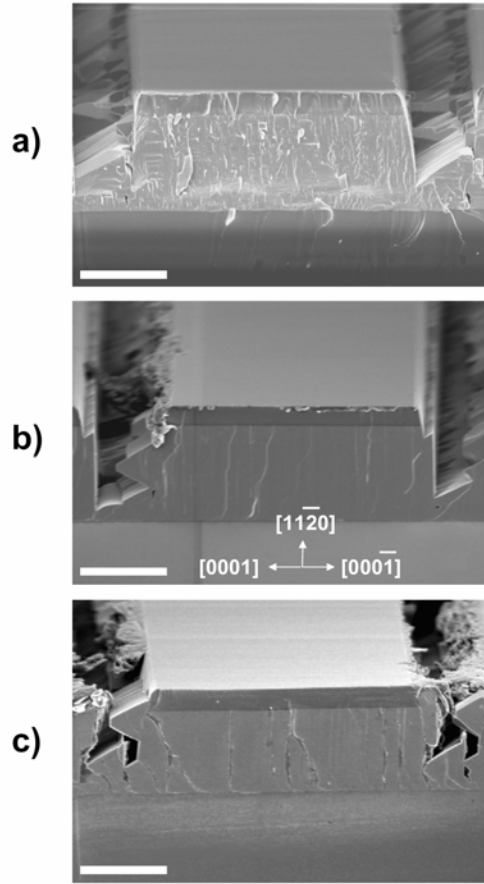


Figure 3.11 The effect of Rotation (per minute) (Scale bar = 5 μm): a) 100 rpm, b) 200 rpm, and c) 300 rpm

3.4.5. Defect Formation in the first stage:

Figure 3.12 b) displays the bright field cross-sectional TEM image of first stage SLEO sample inserted in the schematic for easy visualization along with the cross-sectional SEM image of the same sample in Fig. 3.12 a). The TEM images insured that the material growing from the sidewalls was nearly defect free. The defects on the $(000\bar{1})$ N-face were the dislocations extending along $\langle 000\bar{1} \rangle$ direction; therefore the material growing up was clear of defects. On the Ga-

face(0001) the most prominent feature was the emergence of the stacking faults where the growing material coincides with the mask corners and they do not extend with laterally growing N-face over the mask. In planar a-plane GaN films stacking faults were found on the N-face but these images are the evidence to show that unless there is physical disturbance, such as mask corners, the material lacked these defects. The N-face may be more susceptible to such defects in the presence of physical perturbations, however once they are eliminated the quality of the N-face was comparable to the Ga-face.

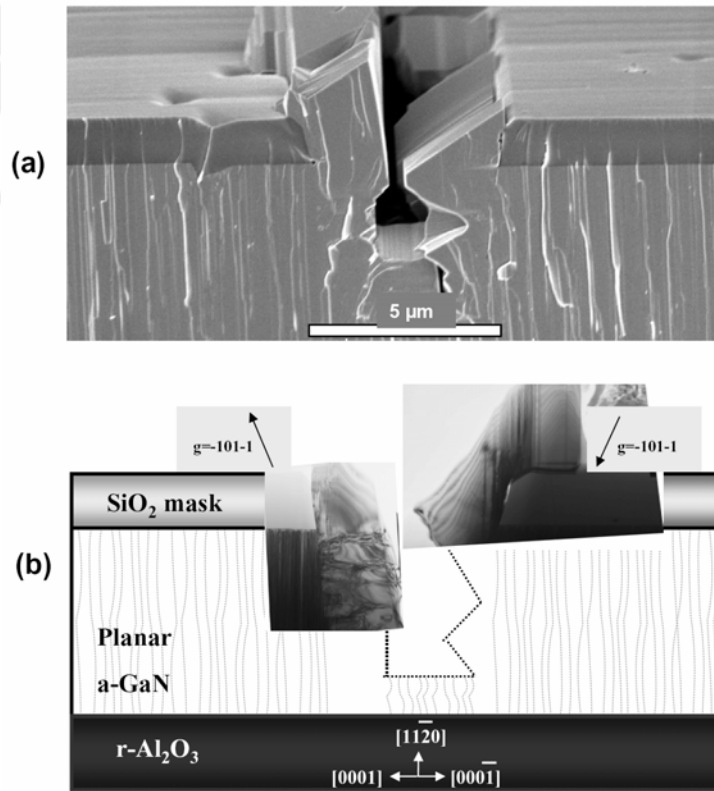


Figure 3.12 (a) First stage growth cross-sectional SEM image (scale bar = 5 μm), (b) Cross-sectional transmission electron microscopy (TEM) image combined with the schematic for first stage growth

3.5. 2nd and 3rd stage growth

After achieving the first stage growth coalescence from the sidewalls, the growth evolution in subsequent stages resembled single-step LEO. However, after the first stage growth the coalesced material in the window region had reduced extended defect densities; therefore the “UP” and “OVER” growth stages were accomplished rather with ease but different mechanism. For second and third stage growths the effect of V/III ratios, growth pressure and temperatures, and different stripe sizes with constant fill factor were investigated. During second and third stage growth, features such as the sidewall morphology, i.e. whether it was jagged or smooth, which was required for homogeneous coalescence, Ga-face to N-face growth rates, and lateral to vertical growth rates were monitored. Same as in first stage growth the standard growth condition given in Table 3.2 were used as 27 sccm TMGa and 1.0 slpm NH₃ flow with V/III ratio~734 at 60 Torr and 1160°C in total 10.0 slpm of H₂ carrier gas. While changing one variable the rest of these variables were kept nominally constant at standard growth condition. The growth was interrupted after 18,000 s for characterization.

3.5.1. *Temperature vs. Pressure:*

In this set of experiments the effect of change in pressure 60 to 40 Torr and change in temperature from 1160 to 1190 °C was investigated. These values were picked based upon the most promising conditions tested in the first stage. Figure 3.13 summarizes the competing effect of change in pressure and temperature in

second and third stage growth mode. Similar to first stage growth experiments increase in temperature lead to higher lateral growth rates. After the first stage coalescence was accomplished, Ga face dominated the N face growth and the lateral growth rate dominated the vertical growth rate with increasing temperature. The enhanced lateral growth is attributed to increased surface migration rates at high temperatures.

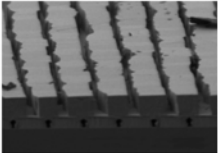
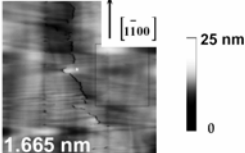
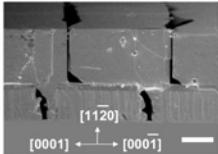
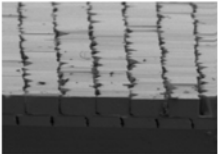
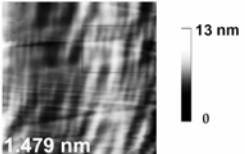
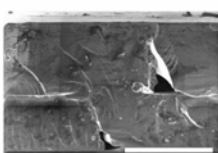
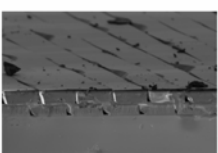
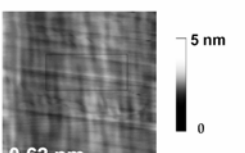
	X-section SEM	Top view SEM	20x20 μm AFM (w/ Rms value)	FWHM (On- and Off-axis)
P= 40 Torr T= 1160 °C				<u>On-axis:</u> 0.146° / 0.372° <u>Off-axis:</u> 0.234° / 0.341°
P= 60 Torr T= 1160 °C				<u>On-axis:</u> 0.082° / 0.276° <u>Off-axis:</u> 0.114° / 0.120°
P= 40 Torr T= 1190 °C				<u>On-axis:</u> 0.146° / 0.392° <u>Off-axis:</u> 0.218° / 0.246°

Figure 3.13 The effect of change in pressure and temperature on second and third stage SLEO. Cross-sectional SEM images are 5000x, and top-view SEM images are 1500x. The AFM images are 20x20 μm^2 (The scale bar = 5 μm .)

Reduced reactor pressure resulted in higher N-face growth rate, the vertical growth rate dominated the lateral growth rate while both the lateral and vertical

growth rates decreased. With decreasing reactor pressure, jagged sidewall features dominated on both Ga and N faces of SLEO. Both increase in temperature and decrease in pressure created degradation in crystal morphologies as can be seen from on- and off-axis FWHM values in Figure 3.13.

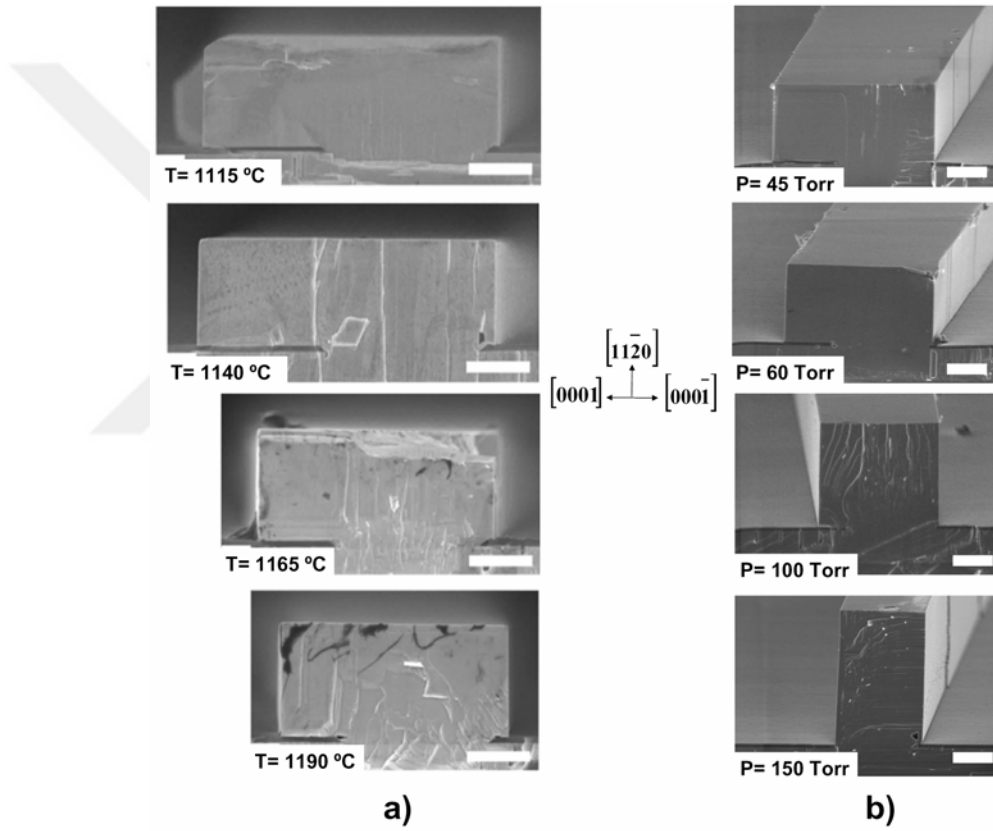


Figure 3.14 Cross section SEM images of the $\langle 1\bar{1}00 \rangle_{\text{GaN}}$ stripe oriented single-step LEO (The scale bar = 5 μm): a) temperature series of 1115 – 1190 °C, and b) pressure series of 45 – 150 Torr. (Courtesy of M. D. Craven [24])

Figure 3.14 shows the effect of change in temperature and pressure on $[\bar{1}\bar{1}00]$ oriented a-GaN single-step LEO stripes. Interestingly change in both temperature and pressure results in the opposite of what was observed for SLEO. In other words, while in SLEO lowering reactor pressure or temperature slowed down the lateral growth rates (Fig. 3.13), in the case of single-step LEO with lower growth pressures or temperatures lateral growth was encouraged (Fig. 3.14 b)). It was also shown that the results can be completely different than what was observed for the first stage growth for the same changes in variables. Although, the second and the third stage growth results should resemble what was observed for single step LEO for a-plane GaN, the reason for conflicting results can be attributed to the presence of defects in window region for single step LEO and for SLEO lacking them.

3.5.2. Group III: TMGa

A range of V/III ratios were tested in the range of ~700-250 by keeping the rest of the growth variables constant. While keeping NH_3 at 1 slpm TMGa was varied as 27, 54, and 81 sccm. Reducing the V/III ratio, while increasing TMGa flow by keeping NH_3 flow constant, created distinct outcomes for first and second-third stage growth modes. In the first stage, reduced V/III ratios (at constant NH_3 flows and increasing TMGa flow from 13.5, 27, and 81) resulted in faster coalescence laterally from the sidewalls. For the subsequent second and third growth stages, however, the vertical growth rate dominated the lateral growth rates due to larger exposed surface area for growth initiation, and so the stripe height

increased. Also, at high growth rates the sidewalls had a rough and inhomogeneous morphology.

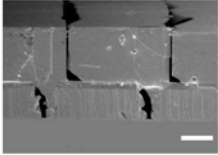
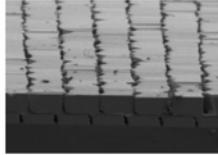
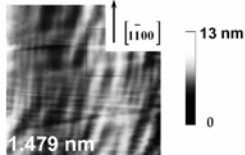
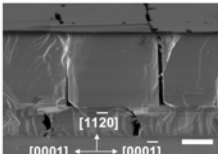
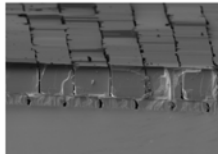
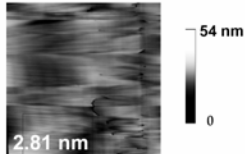
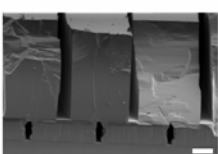
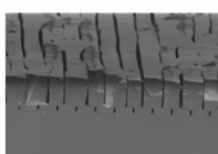
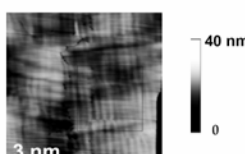
	X-section SEM	Top view SEM	20x20 μm AFM (w/ Rms value)	FWHM (On- and Off-axis)
$\text{NH}_3 = 1 \text{ slpm}$ $\text{TMGa} = 27 \text{ sccm}$ $\text{V/III} = 734$				<u>On-axis:</u> $0.082^\circ / 0.276^\circ$ <u>Off-axis:</u> $0.114^\circ / 0.120^\circ$
$\text{NH}_3 = 1 \text{ slpm}$ $\text{TMGa} = 54 \text{ sccm}$ $\text{V/III} = 367$				<u>On-axis:</u> $0.143^\circ / 0.357^\circ$ <u>Off-axis:</u> $0.310^\circ / 0.275^\circ$
$\text{NH}_3 = 1 \text{ slpm}$ $\text{TMGa} = 81 \text{ sccm}$ $\text{V/III} = 245$				<u>On-axis:</u> $0.299^\circ / 0.462^\circ$ <u>Off-axis:</u> $0.444^\circ / 0.279^\circ$

Figure 3.15 The effect of change in group III (TMGa) flow between 27 – 81 sccm at constant group V (NH_3) flow 1 slpm on second and third stage SLEO. Cross-sectional SEM images are 5000x, and top-view SEM images are 1500x. The AFM images are $20 \times 20 \mu\text{m}^2$ (The scale bar = $5 \mu\text{m}$.)

For all stages increasing TMGa flow enhances N face growth rate. However at 54 sccm TMGa flow, etching effect of NH_3 is balanced with group III precursor flows improving the sidewall morphology, leaving jagged sidewall to smoother sidewall morphology.

3.5.3. NH_3 vs. TMGa :

	X-section SEM	Top view SEM	20x20 μm AFM (w/ Rms value)	FWHM (On- and Off-axis)
$\text{NH}_3 = 1 \text{ slpm}$ $\text{TMGa} = 27 \text{ sccm}$ $\text{V/III} = 734$				<u>On-axis:</u> $0.082^\circ / 0.276^\circ$ <u>Off-axis:</u> $0.114^\circ / 0.120^\circ$
$\text{NH}_3 = 1 \text{ slpm}$ $\text{TMGa} = 54 \text{ sccm}$ $\text{V/III} = 367$				<u>On-axis:</u> $0.143^\circ / 0.357^\circ$ <u>Off-axis:</u> $0.310^\circ / 0.275^\circ$
$\text{NH}_3 = 2 \text{ slpm}$ $\text{TMGa} = 54 \text{ sccm}$ $\text{V/III} = 734$				<u>On-axis:</u> $0.189^\circ / 0.517^\circ$ <u>Off-axis:</u> $0.357^\circ / 1.364^\circ$

Figure 3.16 The effect of change in group III (TMGa) flow between 27 – 54 sccm vs. change in group V (NH_3) flow between 1 -2 slpm on second and third stage SLEO. Cross-sectional SEM images are 5000x, and top-view SEM images are 1500x. The AFM images are $20 \times 20 \mu\text{m}^2$ (The scale bar = $5 \mu\text{m}$.)

NH_3 flow was changed between 1 and 2 slpm at 27 and 54 sccm TMGa flows to observe the effect of group V flow and increased growth rates for the same V/III ratio. For the same TMGa flow, 54 sccm, increasing NH_3 enhances the N face growth. Also, irregular sidewall and surface morphology dominates the stripes as

growing in higher NH_3 flows with increased etching effect. For the same V/III ratio, increased growth rate by doubling both precursor flows, degrades the crystal quality, the surface and the sidewall morphology.

In summary, in terms of controlling the lateral to vertical growth rate ratio and Ga-face to N-face growth rate ratio and to realize smooth sidewall and surface morphologies, the total precursor flows (V+III) were important for all stages. In the second and third stages increasing only the group III flow enhanced the N-face growth rate and lowering the lateral to vertical growth rate yielded taller uncoalesced stripes. Increasing only the group V flows also yielded increased N-face growth rate however creating jagged sidewall morphology due to NH_3 etching effect. In addition, for the same V/III ratio increased V+III degraded the crystal quality and the surface and sidewall morphology.

As a result, the relative amount of group V and group III precursor flows is also important for second and third stages of growth in terms of controlling lateral to vertical growth rate ratio and Ga face to N face growth rate ratio, also achieving smooth sidewall and surface morphologies.

3.5.4. Stripe Width:

In addition to growth variables we also explored the effect of stripe width with a constant fill factor of 0.25. Fill factor can be defined as the ratio of window to period (window and wing) width. The size of the stripes explored were 3/9, 4/12,

and 5/15 μm (window/wing). As the stripe size decreased, as expected faster coalescence was achieved.

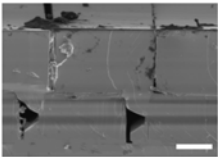
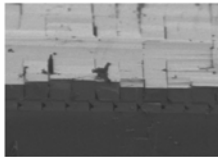
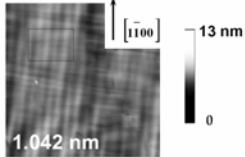
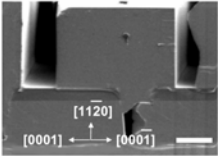
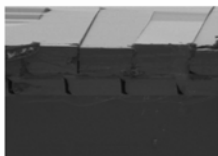
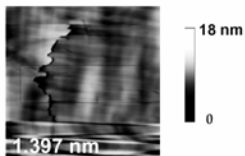
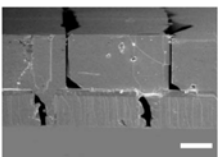
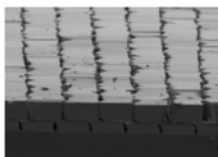
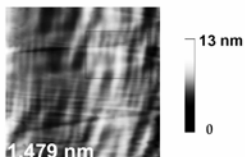
	X-section SEM	Top view SEM	20x20 μm AFM (w/ Rms value)	FWHM (On- and Off-axis)
3 μm / 9 μm				<u>On-axis:</u> 0.193° / 0.325° <u>Off-axis:</u> 0.251° / 0.245°
4 μm / 12 μm				<u>On-axis:</u> 0.1535° / 0.5035° <u>Off-axis:</u> 0.3487° / 0.3882°
5 μm / 15 μm				<u>On-axis:</u> 0.082° / 0.276° <u>Off-axis:</u> 0.114° / 0.120°

Figure 3.17 The effect of change in stripe width with constant fill factor of 0.25 on second and third stage SLEO. Cross-sectional SEM images are 5000x, and top-view SEM images are 1500x. The AFM images are 20x20 μm^2 (The scale bar = 5 μm .)

Although, faster coalescence can be accomplished with decreasing stripe width, keeping the fill factor constant, the crystal quality gets poorer as can be observed by comparing the on- and off-axis FWHM values. Since, each coalescence front was associated with planar array single edge dislocation propagating to the surface, there were more coalescence fronts. However, surface morphology was

improved with smaller stripe widths due to the fact that the material coalesces faster preventing surface undulations as the stripe volume decreasing. In addition as the stripe period is decreased, the material gets more sensitive to variations that might be introduced while processing such as while patterning, and etching for straight sidewalls. Wider the stripe period, higher the crystal quality of the coalesced material but higher the surface undulations as a result of longer growth time needed for coalescence.

3.5.5. Other variables

Rotation

Last stage growth studies showed that toward the end of 3rd stage growth, ramping up the rotation rate from 200 rpm to 300 rpm encourages the coalescence. The reason for this observation was investigated by analyzing the effect of increase in rotation rate to boundary layer thickness for Thomas Swan closed-space vertical MOCVD-3 reactor at various growth temperature and reactor pressures. It is plotted out in Figure 3.18.

Boundary layer is defined as a stagnant layer hanging over the growth surface across which mass transport occurs by simple diffusion [26].

Mathematically, the thickness of this layer can be expressed as: $\delta = 4 \times \sqrt{\frac{\nu}{\omega}}$, where ν stands for kinematic viscosity, which is a temperature and pressure dependant constant, and ω for the rotation rate. The thickness of this layer has the deterministic role in surface kinetics, growth rate, and film uniformity. NH_3 decomposes

heterogeneously on the GaN surface or reactor walls [27]. NH_3 has high bond strength of N-H, with increasing rotation rate the decomposition rate of NH_3 on the GaN surface decreases.

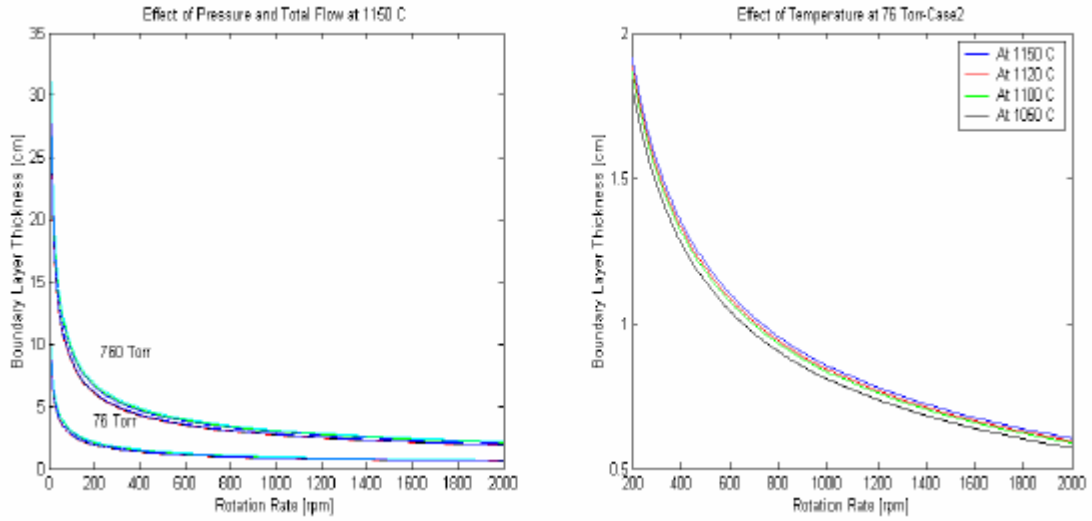


Figure 3.18 The effect of rotation on momentum boundary layer thickness at varying temperature and pressure series for Thomas Swan closed-space vertical MOCVD-3 reactor.

As the rotation rate is increased, the boundary layer is pushed down, enhancing diffusion of group III source to the GaN interface. So, combined effect of latter two causes V/III ratio to fall down. At high temperatures and low V/III ratio, the lateral growth rate increases for third stage SLEO.

Uniformity:

Due to temperature gradient along radius of the wafer, center to the edge, the thickness and the growth quality of the material varies. For a process like a-plane GaN SLEO, this leads to extra sensitive coalescence behavior varying along the wafer and so variations in yield. So far the usable area in MOCVD coalesced a-plane GaN SLEO samples were within the range of 40-70 % because of uniformity gradient effect which is shown in Fig. 3.19.

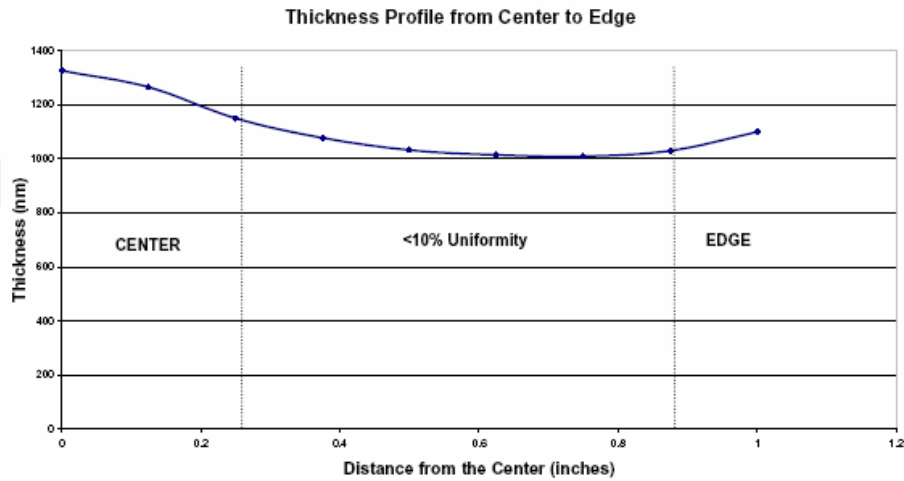


Figure 3.19 Uniformity profile of typical a-plane GaN from center to edge, grown with Thomas Swan MOCVD-3 reactor.

3.6. Coalescence

Figure 3.20 demonstrates the improvement in film quality of 5/15 μm (window/wing) stripes SLEO a-plane GaN compared with 5/15 μm (window/wing) stripes single-step LEO and planar a-plane GaN by comparing the surface roughness values, crystal quality FWHM values for on- $(11\bar{2}0)$ and off- $(10\bar{1}1)$ axis, and defect densities.

According to this figure, the surface roughness for the whole SLEO film was as low as the wing region of the single-step LEO film. It is possible to see the surface quality contrast between the wing and the window regions for single step LEO. While in window regions, where the threading dislocations were still present and were associated with surface depression [24], the rms values were comparable with planar a-plane GaN, in overgrown wing regions the surface quality was comparable with the SLEO film. With the SLEO technique, the surface roughness value improved (from 6 nm to 0.62 nm) one order of magnitude, and the FWHM value decreased (from 0.29° to 0.082° for on-axis and from 0.46° to 0.114° for off-axis) by a factor of four in respect to planar a-GaN yielding a value even comparable with single step LEO c-plane GaN crystal quality. Also from Fig. 3.21 one can see the improvement in the on- and off-axis X-ray rocking curves for all three a-plane samples. TEM results show that the TD densities were in the range of 10^6 - 10^7 cm^{-2} , three to four orders of magnitudes lower than planar films, and the SF densities were in the range of 10^3 - 10^4 cm^{-1} which is at least one to two orders of magnitude that of planar a-GaN.

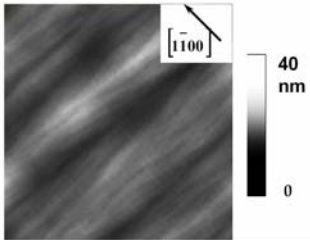
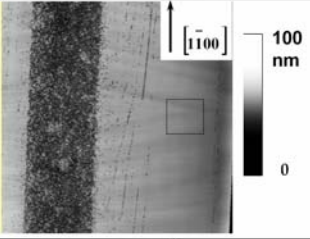
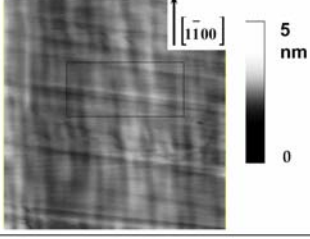
	20x20 μm AFM image	Rms	FWHM		Defect Density
			On-axis (11 $\bar{2}$ 0)	Off-axis (10 $\bar{1}$ 1)	
Planar a-GaN		6 nm	0.29°	0.46°	TD: $\sim 10^{10} \text{ cm}^{-2}$ throughout the material
					SF: $\sim 3.5 \times 10^5 \text{ cm}^{-1}$
Single-step LEO a-GaN		5.82 nm	0.17°	0.27°	TD: $\sim 10^7\text{-}10^8 \text{ cm}^{-2}$ only in the wing region
					SF: $\sim 10^4\text{-}10^5 \text{ cm}^{-1}$
SLEO a-GaN		0.62 nm	0.082°	0.114°	TD: $\sim 10^6\text{-}10^7 \text{ cm}^{-2}$ throughout the material
					SF: $\sim 10^3\text{-}10^4 \text{ cm}^{-1}$

Figure 3.20 Improvement in a-plane GaN film quality by using SLEO. Comparative AFM images, surface roughness and x-ray rocking curve FWHM values, and defect densities for planar a-GaN, 5/15 single-step LEO a-GaN and 5/15 SLEO a-GaN (from top to bottom). The AFM images are $20 \times 20 \mu\text{m}^2$.

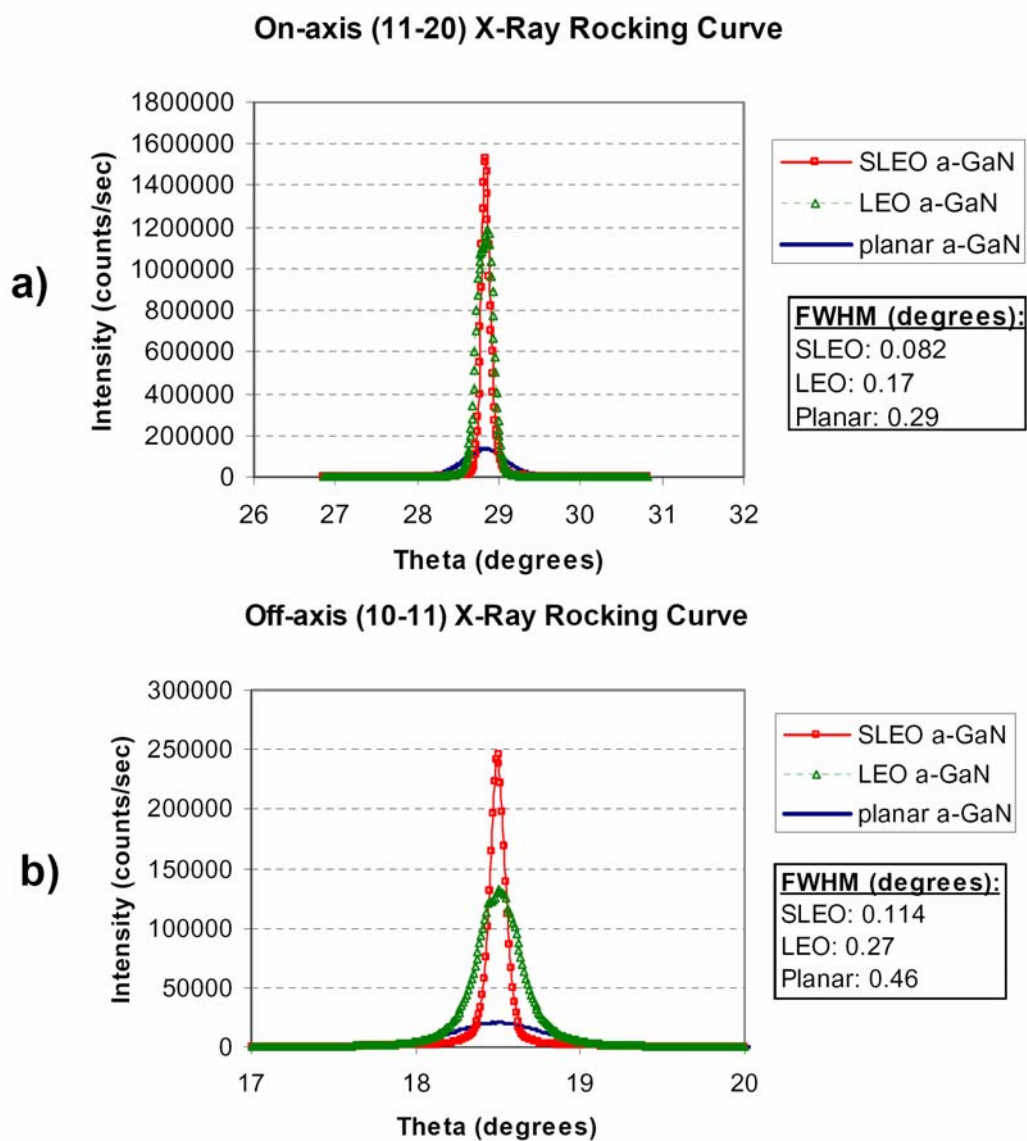


Figure 3.21 Comparison of a) on-axis, and b) off-axis X-ray rocking curves for planar a-GaN, 5/15 single-step LEO a-GaN, and 5/15 SLEO a-GaN.

Figure 3.22 a) is a cross-sectional SEM image of a coalesced SLEO a-plane GaN with 2 μm / 8 μm (window / wing) stripes. The coalescence was achieved in the early stages three hours of growth. This promises, with the same growth conditions, it is possible to coalesce wider stripes. With the presence of defect seed regions, such as the window region in single-step LEO, the film coalescence was almost impossible to achieve, and the yield and reproducibility was an issue with MOCVD. We speculate that the absence of TDs in the seed regions facilitated homogeneous growth leading to coalescence with the proper growth conditions.

When continuous a-plane SLEO material was achieved, there were two coalescence fronts. Each coalescence front was associated with a planar array of TDs, as can be seen from Fig. 3. 22 b) which shows the CL emission across the SLEO film, there was one single dislocation line propagating to the surface at each coalescence front. A recent study done by Haskell et al. [28] showed that stacking faults do not act as non-radiative recombination centers in non-polar III-nitrides. So the dark regions in CL image (where the light is absorbed) , Fig 3.22 b), are associated with TDs at the coalescence fronts and TDs that bound stacking faults rather than being solely stacking faults. The rest of the material is clear of defects and exhibits CL emission.

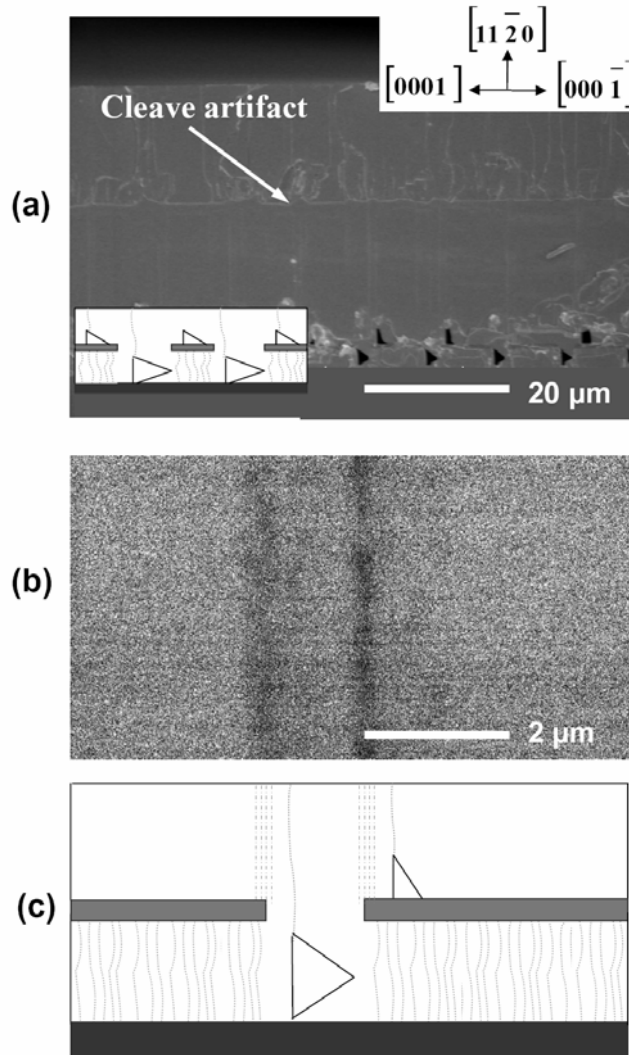


Figure 3.22 (a) Cross-sectional SEM of the coalesced SLEO a-plane GaN film with 2 μm / 8 μm (window/wing) stripes. (b) Panchromatic CL image of the top surface of the coalesced SLEO a-plane GaN film. (c) Schematic cross-section of the coalesced SLEO a-plane GaN film showing the coalescence fronts.

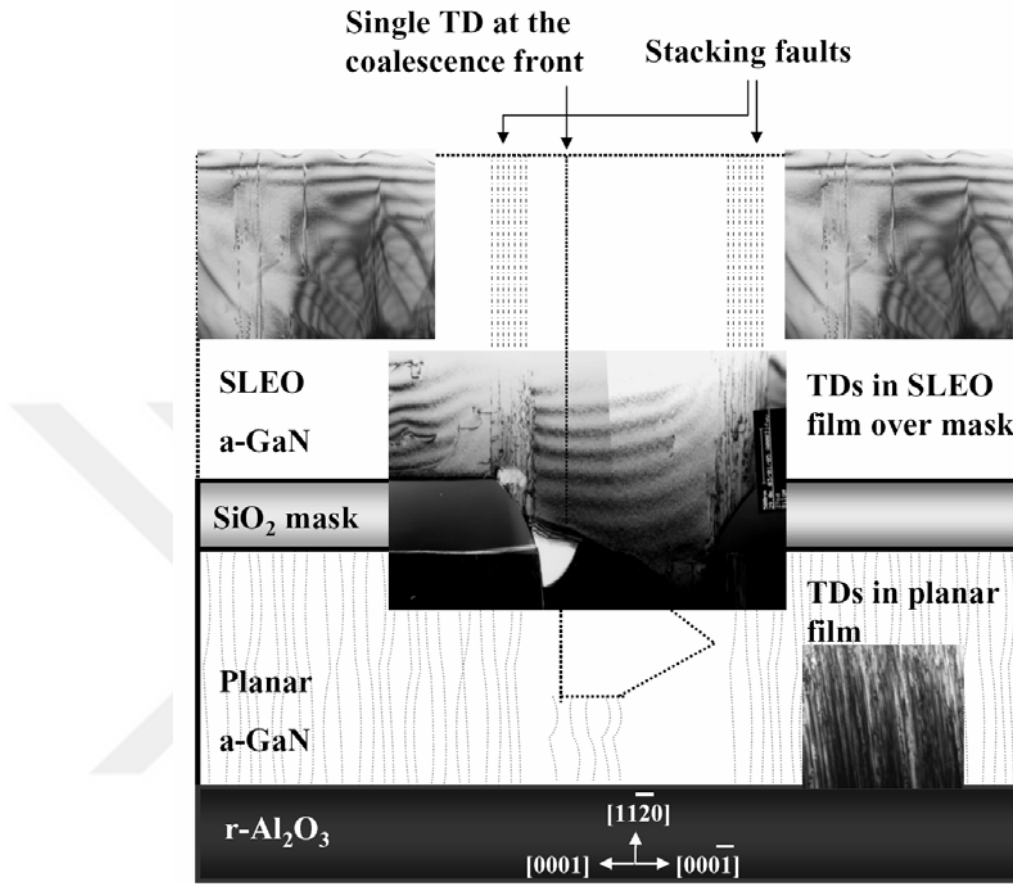


Figure 3.23 Cross-sectional transmission electron microscopy (TEM) image combined with the schematic for the coalesced SLEO material.

Further information on the defect structure in coalesced SLEO a-plane GaN can be obtained by analyzing the cross-section TEM images embedded in the schematic of Fig. 3.23. Similar to the observations made in Figure 3.12, the stacking faults localize only in the mask corners as the material grows over the mask region and N-face more susceptible to these geometric perturbations than the Ga-face. However, as the material passes the corners and grows over the mask, material clears

out of the stacking faults. Almost all the TDs in planar films were blocked under the mask regions. The main source of the dislocations in the fully coalesced SLEO material was the inclined dislocations originated at the sidewalls in the first stage while it is laterally growing (see Fig. 3.12). In addition, if the sidewalls were not etched vertically, i.e. if there was some sidewall angle after etching, then the TDs in the planar material can extend to the surface. Therefore the processing steps should be done with extreme care.

Theory for stacking faults formation in a-GaN revisited

In previous a-GaN LEO studies [9], it was assumed that the origin of the stacking faults is the presence of the N face. However, the cross-section TEM images taken from the mask interface for fully coalesced SLEO sample (Fig 3.23) and for the sample in the early first stage SLEO growth (Figure 3.12) create question marks about the validity of the previous assumption.

From Figure 3.23, it can easily be seen that the stacking faults are localized at the mask corners. It can be said that having SFs localized around the mask is nothing more than a coincidence, and may be related to the theory of Ga face growth rate being considerably higher than the N face growth rate therefore the coalescence front resides at where SFs are ending.

However, if we take a closer look at Figure 3.12, in the small portion of the overgrown N face region, there is no trace of stacking faults except for the region where it coincides with the mask corners. At this point, the speculations can be

made as the formation of stacking faults in a-plane GaN is a physical process, caused by the geometrical stress points at the growth interface, and N face can be more vulnerable to the presence of such geometries on surfaces. Sapphire substrates have surface orientation of $\pm 1^\circ$, and steps and terraces on the surface which are defined even more after *in situ* annealing in H_2 before growth to create better surface morphology for subsequent GaN growth. The inclination of these steps and terraces on the substrate surface, in the case of SLEO the tilt angle of the mask corners due to etching, might be the determining factor for the stacking fault density.

3.7. Pressure stability

Planar a-plane GaN films have at least one to two orders of magnitude higher TD densities compared with planar c-plane GaN films. Planar c-plane GaN films can be grown at atmospheric pressures while planar a-GaN films have to be grown at low pressures (<76 Torr). Over 76 Torr surface faceting and degradation in crystal quality was unavoidable and over 300 Torr there was no dominant a-plane GaN surface present. The limited growth window of a-plane films was associated with presence of high defect densities. SLEO films with reduced defect densities are excellent candidates to confirm the validity of this hypothesis. In this set of experiments, coalesced SLEO pieces from the same template were grown at various growth pressures (60 to 760 Torr) for 1800'' keeping the rest of the growth variables constant at standard reference conditions given in Table 3.2.

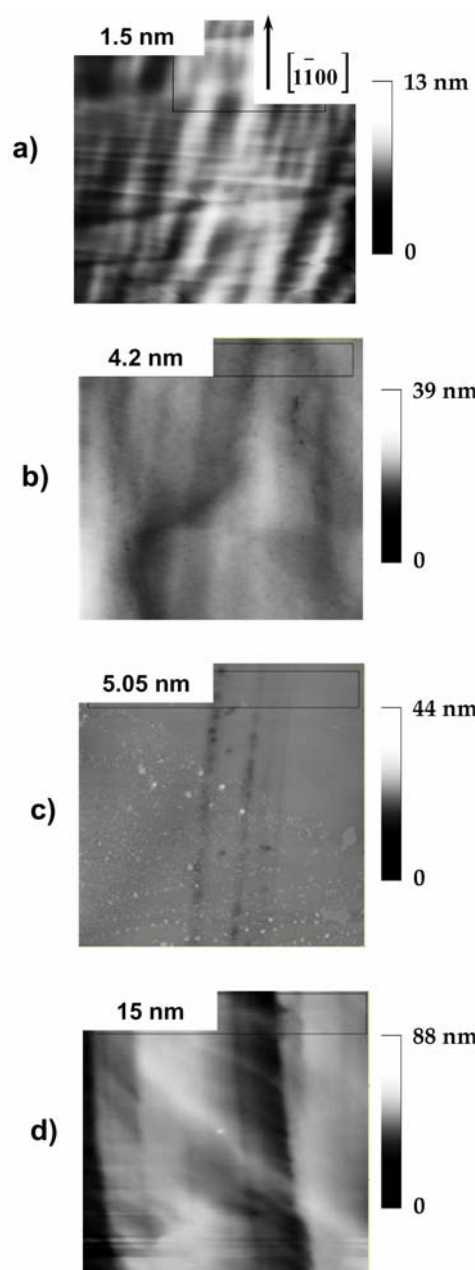


Figure 3.24 $15 \times 15 \mu\text{m}^2$ AFM images and surface roughness values for the effect of change in growth pressure on coalesced $2/8 \mu\text{m}$ SLEO templates: a) 60 Torr, b) 150 Torr, c) 300 Torr, and d) 760 Torr.

Although with increasing growth pressure the surface roughness increased, the growth of a-plane GaN films were achieved even over 300 Torr at which there was no trace of growth for planar films. Figure 3.24 checks with the previous assumption of crystallographic stability of GaN films related with presence of defects. Therefore with utilizing SLEO method it was possible to increase the crystallographic stability of a-plane GaN films.

3.8. Conclusion

SLEO technique yields high-quality continuous non-polar a-plane GaN films. The dislocation densities were three to four orders of magnitude lower and stacking fault densities were one to two orders of magnitude lower compared to planar a-plane GaN. The surface roughness values decreased one order of magnitude with respect to planar a-GaN for the same scale. And the rocking curve on-axis FWHM value was three times lower than planar a-GaN while off-axis FWHM value which is the gauge for the presence of threading dislocations in the material was four times lower with respect to planar a-GaN. These results were achieved by following simple process and growth flow of SLEO and by using one of the most widely used substrates, sapphire.

Normally we should be able to relate the single step LEO growth attributes to the SLEO growth characteristics after the 1st stage growth is completed. However, throughout the analysis we ran into some discrepancies in results for these two methods. It should be kept in mind that in the presence of pits and dislocations the

nucleation and growth mechanisms were altered and they exhibit distinct behaviors. Since single step LEO had the presence of these defects in the window region, as opposed to SLEO, this caused fundamental differences in reaction to the change in growth conditions, such as pressure and temperature, for these two different techniques. This statement can be applied for the differences in 1st stage and 2nd-3rd growth characteristics as well.

Table 3.4 Optimum growth conditions at different stages of SLEO.

GROWTH MODE	GROWTH WINDOW
1	T= 1160 °C-1190 °C P=60 Torr V/III=734-490 Rpm=200 rpm
2	T> 1160 °C P=40-60 Torr V/III=734-367 Rpm=200 rpm
3	T>1160 °C P=40-60 Torr V/III=734-245 Rpm=200-300 rpm

Growth conditions in all three stages before coalescence, etched sidewall angle, stripe widths and stripe alignment had a deterministic role in reducing defect densities and improving the quality of the SLEO a-GaN material. Therefore, extra care should be taken while transferring the LEO mask pattern to achieve proper alignment, and while etching the mask and GaN to obtain straight sidewalls. To minimize the sensitivity to these processing variables and to minimize the defect

densities by reducing number of coalescence fronts largest possible mask widths should be employed.

As a result of analysis of all three stages of growth, the optimum growth conditions were concluded as in Table 3.4. The high lateral growth rates of the Ga-face were favored by high growth temperatures, $>\sim 1160$ °C, low pressure and low V/III ratio, ~ 700 , at low NH_3 flow for the first growth mode. Both the second, “UP”, growth mode and the third, “OVER”, growth mode required high temperature, low pressure growth and concurrently progressively decreasing V/III ratio from ~ 700 -800 to ~ 300 , as the material coalesced and formed a continuous film. For all three stages, high lateral to vertical growth rate ratios were preferred.

3.9. References

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CHAPTER 4:

Electrical Characterization of a-plane SLEO GaN and 360 nm

LEDs

4.1. Introduction

III-nitrides cover the entire visible, UV and infrared spectral region with the bandgap ranging from 0.65 eV to 6.2 eV. Possible use of GaN based UV devices include high density data storage, solid state lighting, and sensor applications. So far i-line (365 nm) Mercury lamps have been used as UV sources, but mercury is toxic and not environmentally friendly. Because of its nontoxicity, thermal and chemical stability, long lifetime and small size, GaN is the major potential substitute to operate at this wavelength range. However the external quantum efficiency (EQE) of these devices is still extremely poor, especially below 370 nm. The highest EQE for UV LEDs to date has been reported by Morita et al. [1] in 2004 as 44% at 365 nm with 1 A pulsed operation and 5% duty cycle. As the operation wavelength range gets shorter, the efficiencies start to suffer from defect densities, absorption, polarization and poor doping more while at 352 nm highest EQE was reported as 1.1% [2], and at 250 nm with Al content over 70% this value dropped down to 0.01% [3]. Threading dislocations (TDs) act as nonradiative recombination centers

leading to reduced internal quantum efficiencies [4], so defect reduction methods should be utilized to eliminate them. Most nitride-based LEDs rely on n-GaN buffer layers and p-GaN capping layers for good current spreading and low contact resistance. These layers absorb at 370 nm or lower wavelength [5]. Therefore reduced defect density wider bandgap templates such as AlGaN or AlN should be produced without cracking to prevent absorption; the templates with semipolar or nonpolar orientations should be employed to avoid polarization in order to improve upon device efficiencies in the UV range.

Although there are some reports on EQE of nonpolar LEDs in the blue and violet range of the spectra[6,7], there is no report on EQE of nonpolar UV LEDs because of poor crystal quality and high defect densities of these nonpolar films (TDs $\sim 10^{10}$ cm $^{-2}$) [8,9]. Morita et al. [1] proved that below TD value of 10^8 cm $^{-2}$ high-efficiency UV LEDs are possible to accomplish. At this point two of the so-called challenges have been surmounted by growing high quality reduced defect density (TDs $\sim 10^6$ - 10^7 cm $^{-2}$ throughout the material) nonpolar a-plane GaN templates with the sidewall lateral epitaxial overgrowth (SLEO) method described in Chapter 3 [10]. In this chapter n- and p-type doping electrical properties of this material are investigated in section 4.2. In the following section device performance of 360 nm GaN/Al $_{0.15}$ GaN UV LEDs grown on SLEO material is discussed in detail and compared with planar a- and planar c-GaN LEDs.

4.2. Doping studies of a-plane SLEO GaN

After n- and p-type samples were grown with a Thomas Swan close-spaced vertical rotating disk MOCVD III reactor, they were processed according to processing details given in Appendix D. Van der Pauw patterns for Hall measurements and transfer-length method (TLM) patterns were defined with photolithography (Fig. 4.1. a)). For n- type the contacts were Al/Au (300/3000 Å) and for p-type contacts Pd/Au (200/2000 Å) was used. Then, square mesa structures, with junction areas of $104 \mu\text{m}^2$ were etched by Cl_2 based reactive-ion etching (RIE) and isolated (Fig 4.1. b)).

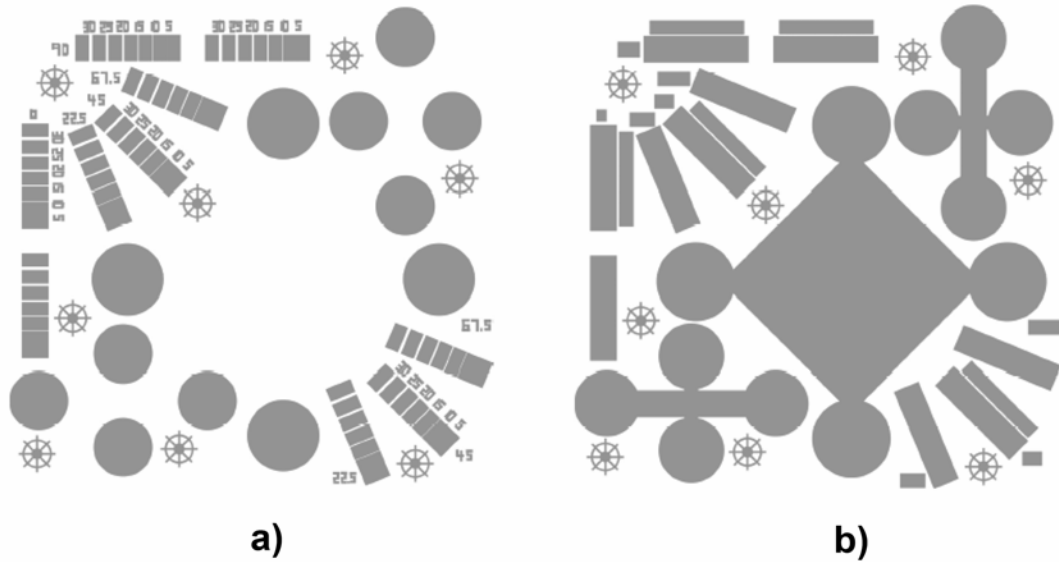


Figure 4.1 Hall and TLM mask pattern for a) metal pads, and b) mesa. (Courtesy of Mel McLaurin)

The mask was specially designed by Mel McLaurin to measure anisotropic conductivity for 0 to 90° from the designated azimuth. For nonpolar a-plane samples

0° was designated for m-azimuth, therefore c-azimuth was aligned with 90° TLM patterns. This mask is useful to understand the dynamics between the defects and the electrical characteristics.

The electrical properties of the *n*- and *p*-type GaN layers were characterized by room-temperature Hall measurements using Van der Pauw patterns, and TLM measurements. The Si concentration in the films was determined using secondary-ion-mass-spectroscopy (SIMS) measurements done using high resolution spectroscopy by Charles Evans and Associates. The Mg concentration in the films was determined with Physical Electronics 6650 dynamic quadrupole SIMS system by Tom Mates.

4.2.1. Si doping

Ideally, as the free carrier concentration decreases, the mobilities should increase. However it was claimed that under certain level of concentrations the carriers get compensated in III- nitrides due to unintentional doping such as carbon [11]. Carbon incorporation increases with decreasing growth pressure, temperature and V/III ratios. A. Chakraborty et al. [12] made an extensive study on Si doping of nonpolar *a*-GaN, and made similar conclusions. What they found was that as the V/III ratio increased by means of increasing NH₃ (from 0.5 to 1 slpm) or decreasing TMGa pure component flow, both electron concentration and mobility values increased. As the pressure increased from 50 to 100 Torr, electron concentration did not change while the mobility values improved.

The low pressure growth conditions required to obtain coalesced a-plane GaN promote carbon incorporation into the films at $\sim 8\text{-}9 \times 10^{17} \text{ cm}^{-3}$. However at doping levels below $1\text{-}2 \times 10^{18} \text{ cm}^{-3}$ the films were highly resistive, which shows that the sole reason for compensation. In addition to carbon, defects in the material can be held accountable for compensation [13]. Also at higher carrier concentrations these impurities and defects act as scattering centers, further degrading the Hall mobilities. Later in this section we will validate this statement by comparing electrical characteristics of two different defect density a-plane GaN films.

To investigate the effect of reduced defect densities on electrical characteristics, $\sim 0.5 \text{ }\mu\text{m}$ GaN:Si layers were grown by varying disilane (Si_2H_6) flows between 0.2 – 1.5 sccm which corresponded to Si/Ga ratio of 2.75×10^{-5} – 2.06×10^{-4} (see Fig. 4.2). To make a relative comparison side-by-side, planar and SLEO a-plane GaN templates were co-loaded for growth. The SLEO templates were highly doped with Si because of the presence of a SiO_2 mask media during regrowth. In order to isolate these doped templates a $\sim 1 \text{ }\mu\text{m}$ thick GaN:Fe insulation layer was grown prior to the Si doped layer as shown in Fig. 4.2. For all samples the growth variables were kept constant at a growth temperature of $1130 \text{ }^\circ\text{C}$, 25 sccm Cp_2Fe and 22 sccm TMGa, 1 slpm NH_3 , and 12 slpm total flows. H_2 was used as carrier gas.

Planar a-plane GaN films had threading dislocation (TD) density of $\sim 2 \times 10^{10} \text{ cm}^{-2}$ and stacking fault (SF) density of $\sim 3.5 \times 10^5 \text{ cm}^{-1}$. SLEO a-plane GaN films had

threading dislocation (TD) density of $\sim 10^6$ - 10^7 cm^{-2} and stacking fault (SF) density of $\sim 10^3$ - 10^4 cm^{-1} .

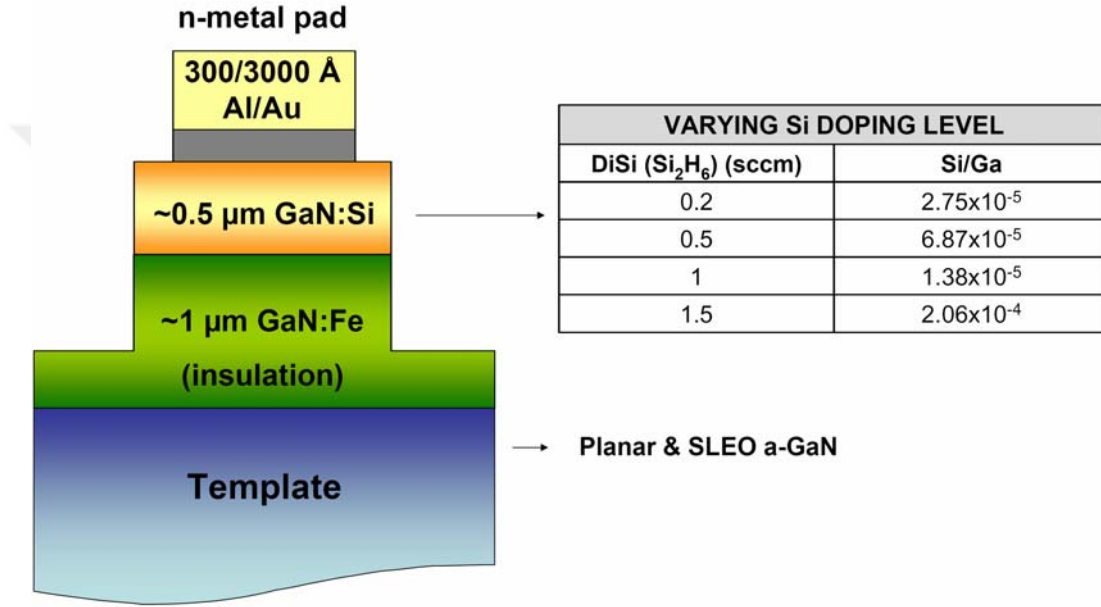


Figure 4.2 Schematic of Si doped Hall and TLM sample structure and varying Si doping levels.

Table 4.1 summarizes the SIMS, Hall, and TLM results for planar and SLEO a-GaN samples. SIMS results revealed that both types of samples incorporated similar amounts of Si in the material and there was a linear trend with increase in DiSi flow. Although the ionization for the SLEO sample was about 10% more than planar samples, the active carrier concentration for both types of materials was also almost identical. The mobility values were two times greater for the reduced defect density SLEO samples. Normally with increase in carrier concentration, mobility

values should decrease. Therefore the opposite trend can only be explained by more efficient screening of defects at higher concentrations.

Table 4.1 Electrical properties of planar a-GaN and SLEO a-GaN at various Si doping levels.

	DiSi flow (sccm)	Si/Ga Ratio	Si Concentration (cm ⁻³)	Hall Concentration (cm ⁻³)	Hall Mobility (cm ² /Vs)	R _{Hall} (Ω/□)	R _{Sheet} (Ω/□)	R _{Contact} (Ω.mm)
Planar a-GaN	0.2	2.75x10 ⁻⁵	4.32x10 ¹⁸	NA	NA	NA	NA	NA
	0.5	6.87x10 ⁻⁵	6.55x10 ¹⁸	5.51x10 ¹⁸	62.84	361	412	1.856
	1	1.38x10 ⁻⁴	1.03x10 ¹⁹	9.02x10 ¹⁸	90.46	153.2	162	1.091
	1.5	2.06x10 ⁻⁴	1.40x10 ¹⁹	1.05x10 ¹⁹	106.77	111.5	116.5	1.03
SLEO a-GaN	0.2	2.75x10 ⁻⁵	4.33x10 ¹⁸	3.4x10 ¹⁸	64.18	572.84	600	2.32
	0.5	6.87x10 ⁻⁵	6.10x10 ¹⁸	5.55x10 ¹⁸	127.86	176	183.4	1.368
	1	1.38x10 ⁻⁴	9.05x10 ¹⁸	8.12x10 ¹⁸	230.45	66.8	68	0.9506
	1.5	2.06x10 ⁻⁴	1.20x10 ¹⁹	1.04x10 ¹⁹	261.12	46.03	49	0.9665

The planar samples with concentrations lower than 5.5x10¹⁸ cm⁻³ were compensated and highly resistive, the SLEO samples on the other hand, were less compensated while still measuring high resistivity values at lower carrier densities 3.4x10¹⁸ cm⁻³. If the impurities (C, O) were held accountable for n-type compensation in GaN, then this is the evidence for reduced impurity incorporation in the material due to reduced defect densities. Figure 4.3 shows the change in mobility and resistivity values with respect to change in carrier concentrations for planar and SLEO films.

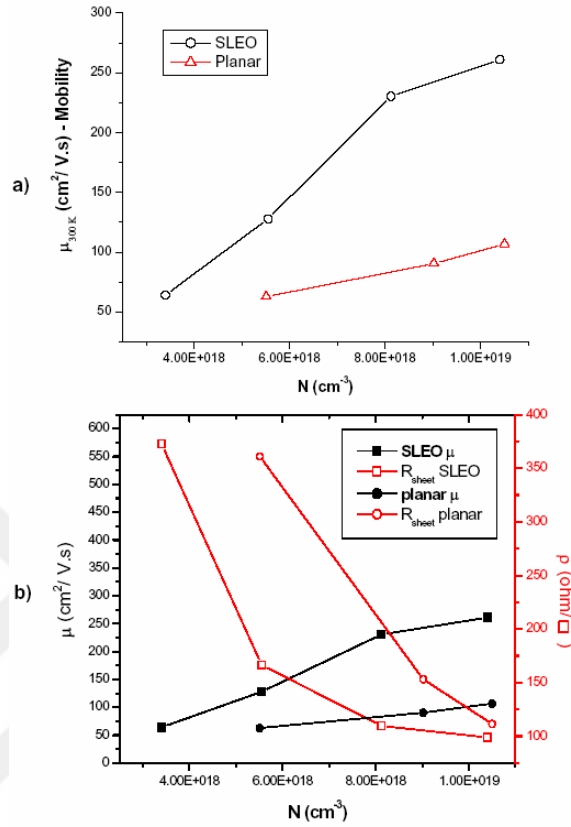


Figure 4.3 a) Change in mobility, and b) Change in mobility vs. sheet resistance for planar a-GaN and SLEO a-GaN with increasing electron concentration levels.

The highest mobility observed for SLEO a-GaN was $261.12 \text{ cm}^2/\text{Vs}$. For the same Si/Ga ratio, planar a-GaN samples yielded mobility values of $106.77 \text{ cm}^2/\text{Vs}$. However for the same Si/Ga ratio SLEO, carrier compensation was $\sim 10\%$ less than planar a-GaN. Previously Mike Craven et al. [13] obtained the highest mobility at $109 \text{ cm}^2/\text{Vs}$ for a carrier concentration of $1.8 \times 10^{19} \text{ cm}^{-3}$ in planar a-GaN samples grown on a-plane SiC, which was comparable to what we found for planar a-GaN

films. Therefore relative to planar films, SLEO a-GaN films had 2.5 times higher mobilities due to reduced compensations and electron scattering sites.

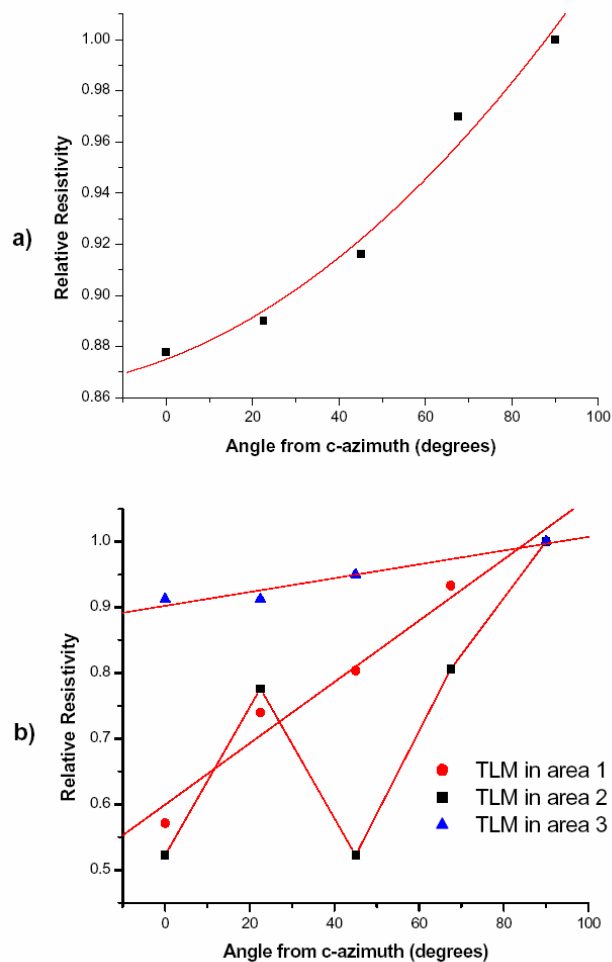


Figure 4.4 Angular dependent resistivity of a) planar a-GaN, and b) SLEO a-GaN sample for three different areas.

Figure 4.4 demonstrates the angular dependence of conductivity in planar and SLEO a-GaN. In planar samples the lowest resistivity was obtained along the c-azimuth and at 90° apart along the m-azimuth the resistivity was highest. When the resistivity values were normalized with respect to highest value we see an increasing trend from 0.88 (0° from c-azimuth) to 1 (90° from c-azimuth). So the percentage drop in resistivity going from m-azimuth to c-azimuth was 12%. These findings are consistent with Arpan Chakraborty et al. results. They observed a similar trend with higher (28%) drop in resistivity [12]. This anisotropic conductivity phenomenon was related to anisotropy in the hole effective mass. However as Fig. 4.4 b) shows, in the case of the SLEO sample, the anisotropic trend differs at different parts of the same sample with the same Si doping. Unlike planar films, the defect distribution in the SLEO films was inhomogeneous. The reported defect densities for SLEO films are averaged across the film, so for different areas this value can be either lower or higher than the average. Similarly we observe a difference in anisotropic conductivity values at different areas of the sample. This varying trend throughout the sample suggests that the anisotropic conductivity is strongly correlated with defects.

4.2.2. Mg doping

Conventional GaN is unintentionally doped and readily n-type, therefore doping with Si was not an issue. However, due to deep acceptor energies of wide bandgap materials and hydrogen impurities related with MOCVD growth processes, an effective p-type doping could not be achieved until the late 1980s until Amano et

al. utilized low energy electron beam irradiation (LEEBI) to activate p-doping [14]. A few years later, in 1992, Nakamura et al. achieved p-type activation by rapid thermal anneal in N₂ ambient [15]. Activation of p-doped layers was the major milestone and start of a new era of possibilities of III-nitride-based electronic and optical devices. Another major milestone in III-nitrides was reached in the early 2000s with realization of device-quality nonpolar films [8, 16]. For development of high performance nonpolar devices conductivity control through n- and p-type doping was needed. It was followed by electrical characterization of n-doped nonpolar GaN by Craven et al in 2003 [13]. An in-depth study of electrical characterization for p-type doping of nonpolar GaN was achieved by Chakraborty et al. [17]. They found that with increasing growth rates (at constant V/III and TMGa/Cp₂Mg rates), and reducing growth temperatures Mg concentration was increased. The highest quality electrical properties were achieved at 66 sccm TMGa, 6 slpm NH₃, and 175 sccm Cp₂Mg. The highest mobility was achieved as 3.32 cm²/Vs at 8.6x10¹⁹ cm⁻³ Mg and 6.1x10¹⁷ cm⁻³ hole concentrations at the optimized growth condition. The acceptor activation energy for Mg in a-plane GaN (75 – 95 meV) was found to be lower than c-plane GaN (~160 meV) with a very low ionization energy ~0.9% at RT.

In order to reproduce and calibrate the same growth conditions, a SIMS stack sample was grown over planar a-plane GaN template. This SIMS structure and profile are given in Fig. 4.5. The stack was made up of 4 periods. Each period was composed of ~0.4 μm thick GaN:Mg, (~0.2 μm thick to prevent cracking and if AlGaIn:Mg) doped film and AlGaIn/GaN/AlGaIn spacer. As the Mg flow was changed to maintain the constant TMGa to Cp₂Mg molar ratio TMGa and NH₃ flows

were adjusted accordingly. For all layers a growth temperature of 1130 °C, growth pressure of 76 Torr, and total flow of 12 slpm was used. H₂ was the carrier gas. From bottom to top, TMGa, NH₃, and Cp₂Mg flows were 22 sccm, 1 slpm, and 60 sccm, respectively for the first layer; 44 sccm, 2 slpm, and 120 sccm for the second layer; 44 sccm, 2 slpm, and 120 sccm (and 6 sccm TMA) for the third layer; 22 sccm, 1 slpm, and 60 sccm for the last layer.

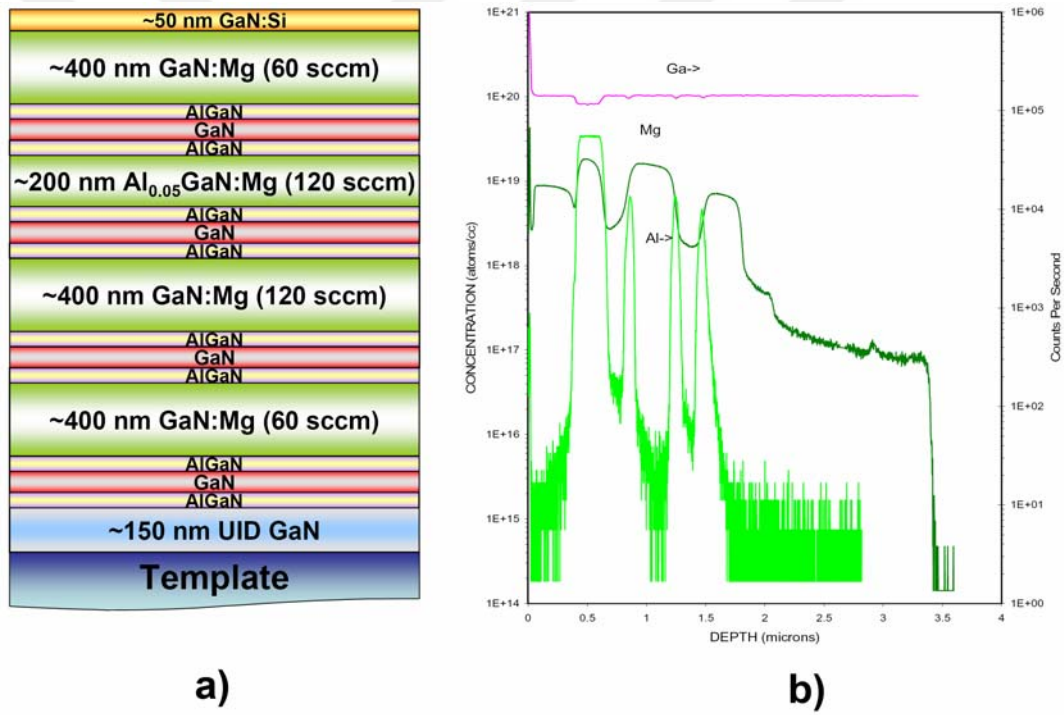


Figure 4.5 SIMS stack for various Mg doping levels in GaN and Al_{0.5}GaN a) sample stack structure, and b) SIMS profile of Mg concentration and Al and Ga counts in AlGaN matrix.

5% Al alloying in the third layer did not affect the amount of Mg concentration. With doubling growth rate (at constant molar ratios) Mg incorporation was doubled. The observed trends and the Mg incorporation levels were similar to the results from previous studies. The SIMS stack was an important reference point in determining the desired previously optimized growth conditions.

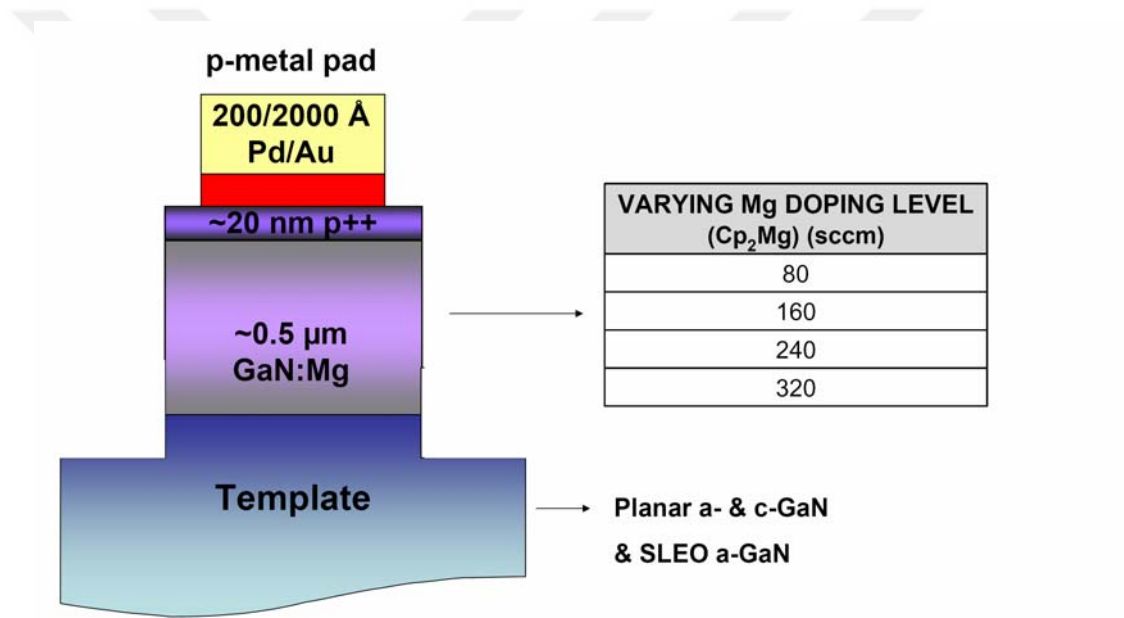


Figure 4.6 Schematic of Mg doped Hall and TLM sample structure and varying Mg doping levels.

To investigate the effect of reduced defect densities on electrical characteristics, ~0.5 μm GaN:Mg layers were grown by varying Cp₂Mg flows between 80 - 320 sccm and capped with 20 nm heavily doped p-GaN contact layer (see Fig. 4.6). To make a relative side-by-side comparison, SLEO a-GaN, planar a-GaN and planar c-GaN templates were co-loaded for growth. For all samples the

growth variables were kept constant at a growth temperature of 1130 °C, growth pressure of 76 Torr, 66 sccm TMGa, 6 slpm NH₃, and 12 slpm total flows. H₂ was used as carrier gas. After the growth, samples were annealed in N₂/O₂ ambient at 700 °C for 15 minutes to activate the p-doped layer. Then the processing flow in Appendix D was followed.

The SIMS data in Fig. 4.8 reveals the Mg incorporation in the three different templates. Fig. 4.7 demonstrates the trend between various Cp₂Mg flows and Mg incorporation. Accordingly, Mg incorporates in SLEO more than in planar a-GaN samples. The Mg incorporation levels were much lower (~6x) than expected for planar a-GaN films. Therefore, the material was extremely resistive. Hall or TLM measurements could not be carried out effectively.

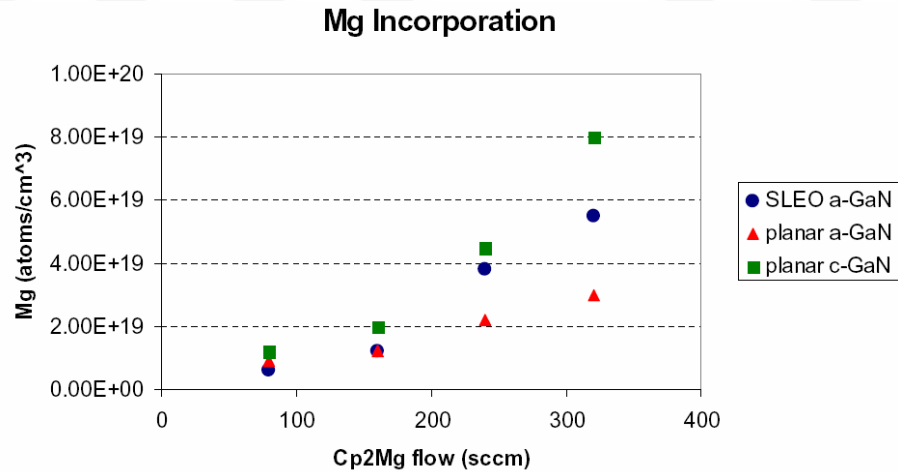


Figure 4.7 Mg incorporation for SLEO a-GaN, planar a-GaN and planar c-GaN, for various flow rates Cp₂Mg.

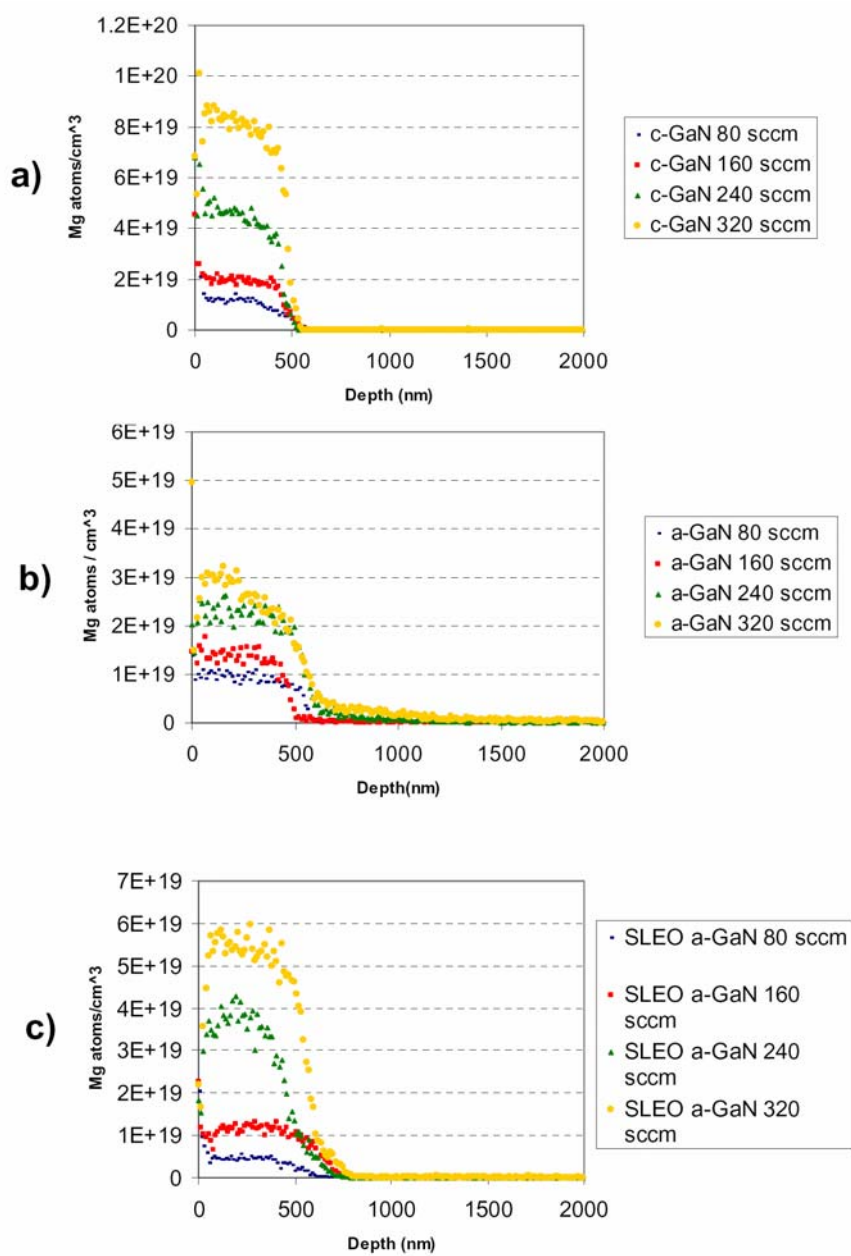


Figure 4.8 Mg incorporation SIMS data for: a) planar c-GaN, and b) planar a-GaN, and c) SLEO a-GaN at 80, 160, 240, and 320 sccm Cp_2Mg flow rates.

4.3. a-plane SLEO GaN 365 nm GaN/Al_{0.15}GaN LEDs

In the UV wavelength range, 370 nm and lower, as the bandgap gets wider the efficiencies start to suffer from defect densities, absorption, polarization and poor doping. For nonpolar III-Nitrides, these adverse effects are more pronounced due to higher defect densities, limited growth windows, and not well understood doping behavior. Therefore, there is no report on external quantum efficiency (EQE) of nonpolar UV LEDs at that wavelength, although there are numerous studies reporting EQE of polar UV LEDs at 365 nm [1-3]. In 2003 Chen et al. reported the first a-plane GaN/AlGa_N LEDs at 363 nm with high resistivity values [18].

In polar GaN/AlGa_N QWs, the separation of the wavefunctions due to the built-in electric field induces long radiative lifetime, which in turn leads to a charge accumulation in the wells. This capacitor effect is only partially compensated by the nonradiative recombination channels and it becomes increasingly important in wider wells, in which the separation of the wavefunctions is larger [19]. Therefore in polar QWs, well and barrier width becomes extremely important in influencing the quantum efficiencies. To determine the optimum well width several groups did extensive studies. Zeng et al.[20] varied the well depth of GaN/Al_{0.2}Ga_{0.8}N MQWs between 0.6 to 4.8 nm. These samples were grown by MOCVD on sapphire substrate with a fixed barrier width of 5 nm. They found out that well width between 1.2 and 4.2 nm provides the highest quantum efficiency, which can be attributed to the reduced piezoelectric effect and non-radiative recombination rate as well as the increased quantum well quality. The decreased efficiency for well widths below 1.2

nm is due to enhanced carrier leakage to the underlying GaN layers. Decreased quantum efficiency in MQWs with well depth > 4.2 nm was associated with an increased non-radiative recombination rate approaching the critical thickness of MQWs as well as a large red-shift due to the presence of strong piezoelectric field. The piezoelectric field causes spatial separation of the electron and hole wave functions and hence a large red-shift of the well emission peak with respect to the GaN epilayers as well as a reduction of the radiative recombination rate (or quantum efficiency). Similar research was done by Hirayama and Aoyagi [21] for a $\text{Al}_{0.53}\text{Ga}_{0.47}\text{N}/\text{Al}_{0.11}\text{Ga}_{0.89}\text{N}$ MQW structure with fixed barrier width of 8 nm. They found the optimum well depth for this compound structure to be ~3 nm.

The absence of polarization in nonpolar nitrides has been demonstrated by several groups [16, 22, 23]. In the case of nonpolar QWs, the transitions between conduction and valence bands occur with flat band conduction. The wavelength does not shift with change in current. Therefore, well and/or barrier width only determines the intensity of the emission peak and the lifetimes of the carriers. Time resolved photoluminescence studies of nonpolar GaN/AlGaN quantum wells are described in Appendix A in detail.

In order to compare defective planar films and reduced defect density films, and to make comparative analysis between polar versus nonpolar films, nonpolar SLEO a-GaN, nonpolar planar a-GaN, and polar planar c-GaN films were co-loaded for device growth. A well width of 3 nm was chosen as it was determined to be the optimum well width to minimize polarization effects in polar films, and the highest

intensity with the lowest radiative lifetimes were obtained because of better electron-hole wavefunction overlap in nonpolar a-GaN/AlGaN quantum wells. The barrier width of 10 nm was chosen with 15% Al content for good electrical confinement.

4.3.1. Experimental Procedure

4.3.1.1. Device growth

The LED structures were grown on ~ 1.5 μm nonpolar planar a-plane GaN, ~ 1.5 μm polar planar c-plane GaN, and ~ 20 μm MOCVD initiated HVPE coalesced nonpolar SLEO a-plane GaN templates. The size of the LEO stripes were 5 μm / 15 μm (window / wing) oriented along the m-direction as described in Chapter 3. Planar a-plane GaN films had threading dislocation (TD) density of $\sim 2 \times 10^{10} \text{ cm}^{-2}$ and stacking fault (SF) density of $\sim 3.5 \times 10^5 \text{ cm}^{-1}$. SLEO a-plane GaN films had threading dislocation (TD) density of $\sim 10^6$ - 10^7 cm^{-2} and stacking fault (SF) density of $\sim 10^3$ - 10^4 cm^{-1} . And the planar c-plane GaN films had TD density of $\sim 10^8 \text{ cm}^{-3}$. The device growth was carried out in a vertical close-spaced rotating disk 1x2'' Thomas Swan MOCVD III reactor. The structure consisted of ~ 0.5 μm GaN:Si ($\sim 2 \times 10^{19} \text{ cm}^{-3}$), 7 periods of GaN/Al_{0.15}GaN (3 / 10 nm) MQW active region, ~ 20 nm Al_{0.2}GaN barrier layer, ~ 200 nm Al_{0.05}GaN:Mg, and lastly, a ~ 20 nm heavily doped GaN contact layer. The device structure schematic is shown in Fig. 4.9.

In order to see the effect of Si doping in the barriers two types of LED structures were grown. One with lightly doped barriers at $2 \times 10^{18} \text{ cm}^{-3}$ to compensate background impurities and one with no Si doping in the barriers.

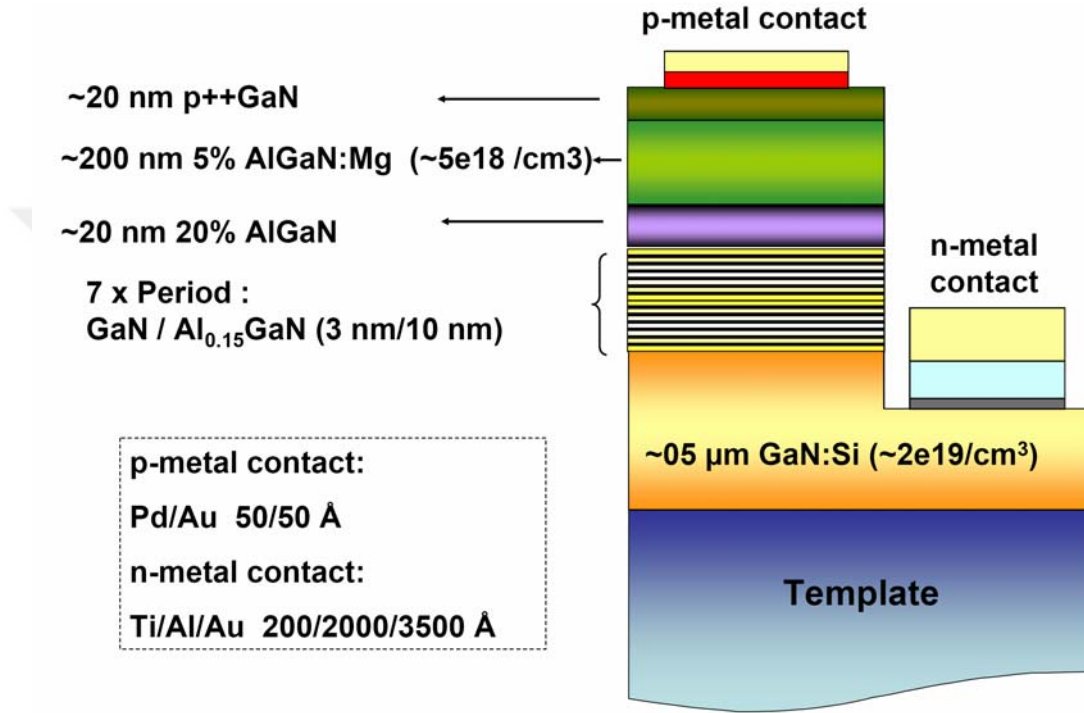


Figure 4.9 GaN / Al_{0.15}GaN LED device structure and metal contacts used.

4.3.1.2. Device processing

The devices were processed with a processing flow developed by Matt Schmidt. The details for the processing flow are given in Appendix C. Devices were annealed for p-activation with rapid thermal annealing (RTA) in N₂/O₂ ambient at 700 °C for 15 minutes. The Pd/Au (50 Å / 50 Å) p-metal contacts were patterned

by lithography. A mesa etch in Cl_2 based reactive ion etching (RIE) was followed by Ti / Al / Au (200 Å / 2000 Å / 3500 Å) n-metal contact lithography. Processing was finalized with Ti / Au (200 Å / 3000 Å) thick pad metal deposition.

4.3.1.3. Device testing

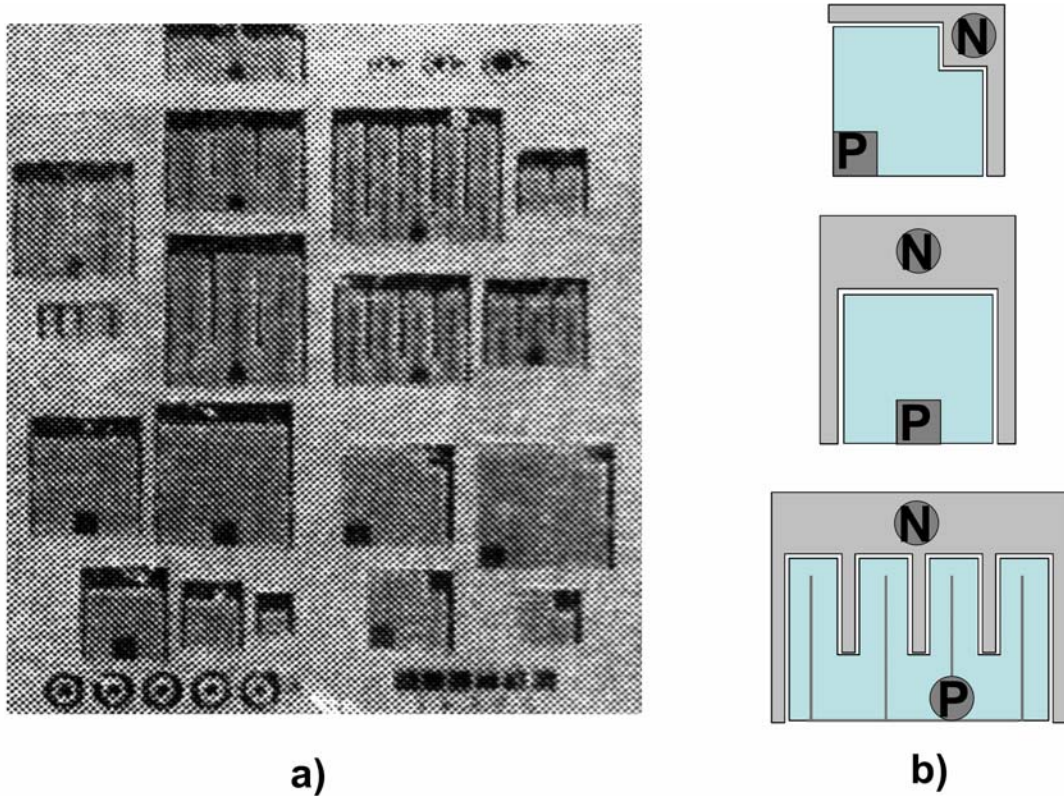


Figure 4.10 a) One die from the LED mask used in device processing, and
b) Three main types of devices with different contact geometry (from top to bottom); L-shaped, U-shaped, and interdigitated (E-shaped).

The LED mask was designed by Tom Katona [24]. There were three types of devices differing by their contact geometry; Interdigitated (E-shaped, or finger

pattern), U-shaped, and L-shaped. The interdigitated contact geometry was proposed to prevent current crowding and reduce the series resistance [25, 26]. Each contact geometry, there were three sizes of junction areas with $300 \times 300 \mu\text{m}$, $400 \times 400 \mu\text{m}$, and $500 \times 500 \mu\text{m}$ on the mask.

The devices were tested for I-V and E-L with DC at room temperature. The power measurements were done with AC at room temperature on unpackaged (on-wafer) LEDs. All device testing techniques were utilized to see the effect of defect densities, the effect of polarization fields, the effect of Si doping in the barriers, and the effect of the contact geometry.

4.3.2. Results

After testing over 200 SLEO a-GaN, 100 planar c-GaN, and 100 planar a-GaN LEDs, the power density results varied for planar a-GaN LEDs in the range of $0.02\text{--}0.05 \mu\text{W}$; for planar c-GaN LEDs between $0.3 - 0.5 \mu\text{W}$ (10 times higher) and for the SLEO a-GaN in the range of $2.5 - 6.5 \mu\text{W}$ (100 times higher).

In general, due to better current spreading, the Interdigitated (E-shaped) contact geometry devices yielded the best power, lowest forward voltage and lowest series resistivity results and then the U-shaped devices was the second best which was followed by the L-shaped devices.

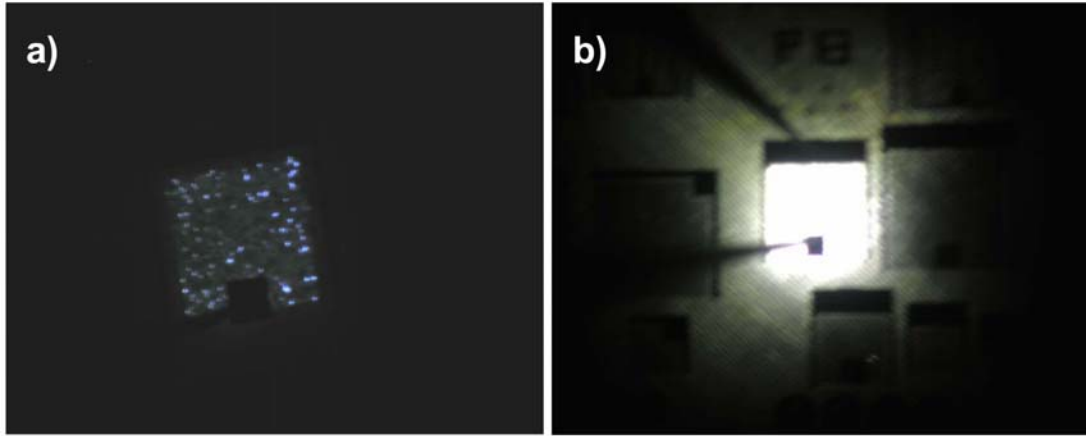


Figure 4.11 Light emission at 20 mA bias from U-shape contact geometry a) planar c-GaN, and b) SLEO a-GaN on wafer LEDs under microscope.

From Fig. 4.11 it is possible to see the difference in the color of emission (wavelength) and the intensity of emission. Planar polar c-GaN in Fig 4.11 a) exhibits defected dark areas, nonradiative recombination sites. It is easy to see the distinct and homogeneous emission from SLEO devices in Fig 4.11 b). Also comparing the two, although the c-GaN peak was measured at ~ 365 nm from EL spectra, the physical emission was close to violet due to combined effect of self-heating and intense broad absorption emission at ~ 370 nm. Due to tail edge, self-absorption emission, the peak emission was near violet for planar c-GaN.

Figure 4.12 shows the high quality SLEO LED with on-wafer $5\mu\text{W}$ emission power at 20 mA bias and room temperature.

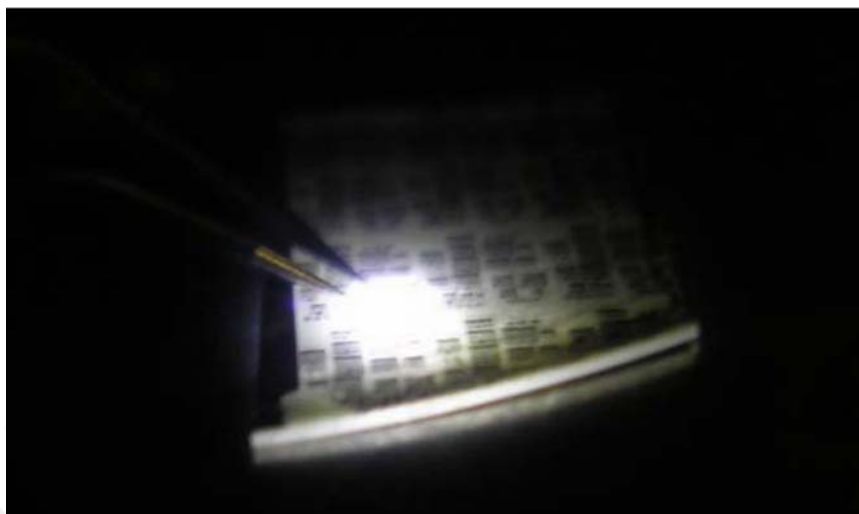


Figure 4.12 365 nm light emission to air at 20 mA bias from a-plane SLEO GaN/Al_{0.15}GaN unpackaged LED.

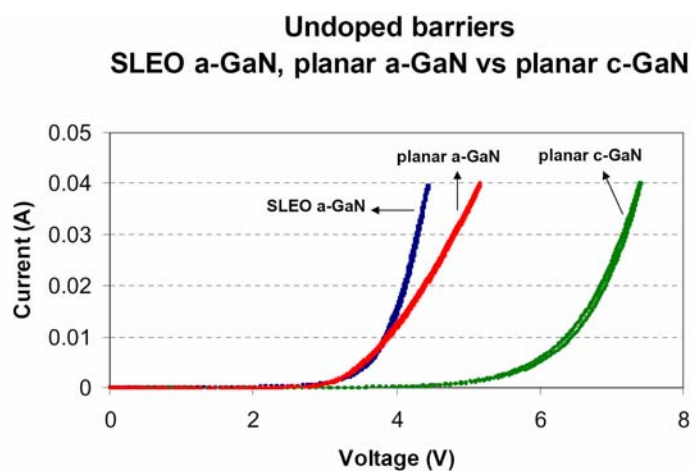


Figure 4.13 I-V characteristics for SLEO a-GaN, planar a-GaN, and planar c-GaN devices with undoped barriers for interdigitated

As shown in the combined I-V curves of the all material type LEDs (Fig. 4.13), planar c-GaN has a turn-on at 6.2 eV, both planar and SLEO a-GaN turned-on at around the same voltage; 3.5 eV and 3.75 eV, respectively. However, in planar a-GaN LEDs, the series resistance was the highest at $\sim 41.7 \Omega$ indicating presence of high defect densities, the SLEO LEDs had $\sim 16.65 \Omega$ resistance. Series resistance values for polar c-GaN LEDs fell in between these two values with $\sim 30 \Omega$. The resistivity values were directly proportional with the defect densities; as a-plane GaN having the highest (TDs $\sim 10^{10} \text{ cm}^{-2}$) and SLEO a-GaN with the lowest values (TDs $\sim 10^6\text{-}10^7 \text{ cm}^{-2}$), while planar c-GaN again falls in between (TDs $\sim 10^8 \text{ cm}^{-2}$).

In the next figure (Fig. 4.14) current voltage characteristics for different contact geometries with a contact area of $400 \times 400 \mu\text{m}$ are compared. The efficiency of LEDs decreases with increase in chip area because of increased heating and absorption effects. Therefore, the same contact area was compared for different contact geometries. The forward voltage and the series resistance for interdigitated devices were lower than L- or U shape devices. L-shape devices had $\sim 10\%$ higher series resistance than U-shape devices and U-shape devices had $\sim 10\%$ higher resistance than interdigitated ones. This was related to the current crowding effect reported by Katona et al. [24, 25, 26]. The interdigitated contact geometry assists in better current spreading and lower lateral resistance which eventually reduces the series resistance.

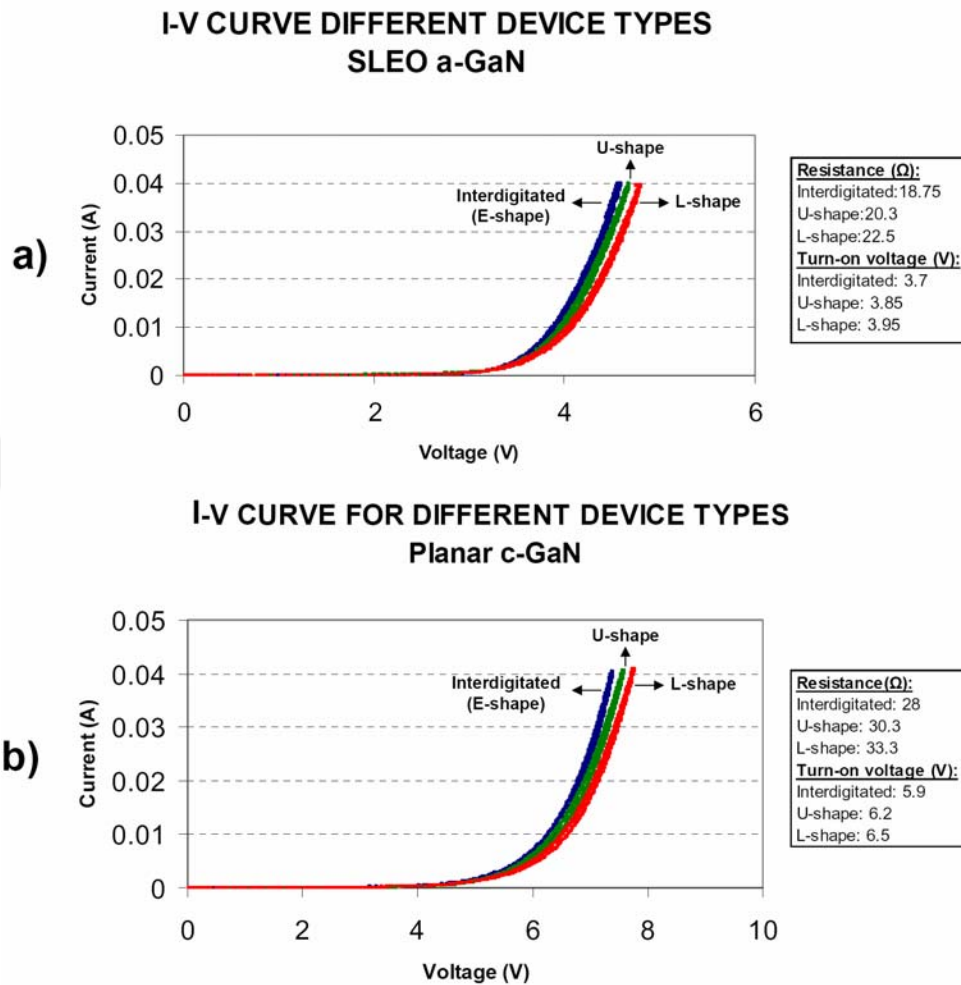


Figure 4.14 I-V curves for three main types of devices with $400 \times 400 \mu\text{m}$ junction area, resistance and turn-on voltage associated with each type of device for a) SLEO a-GaN and b) planar c-GaN LEDs with undoped barriers.

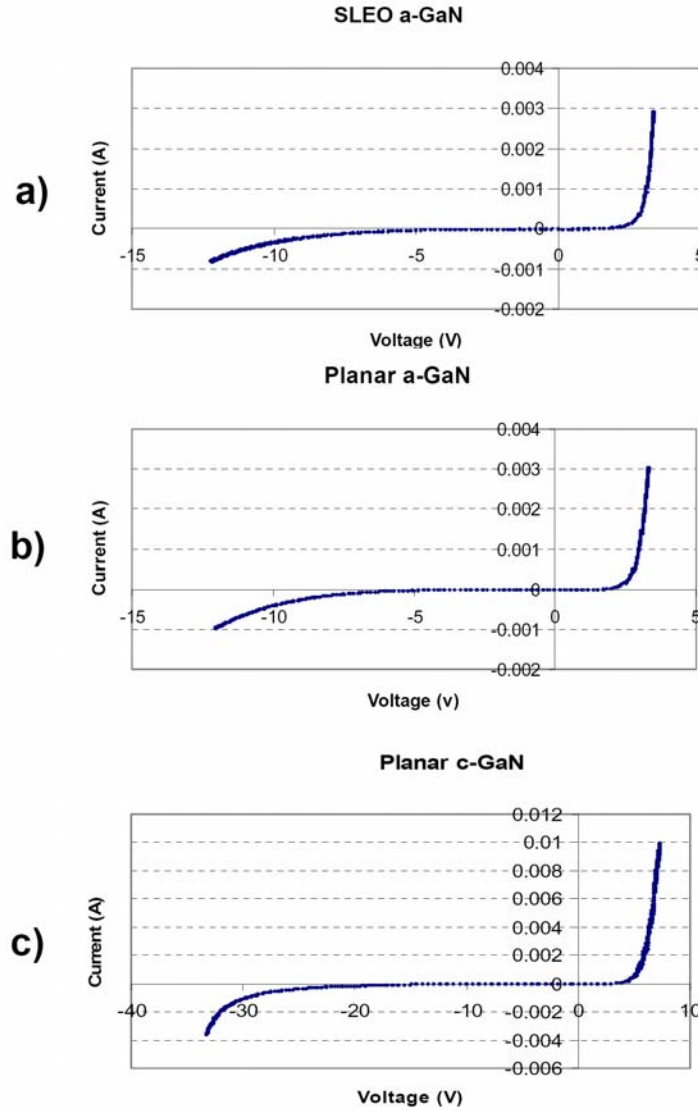


Figure 4.15 Reverse bias I-V characteristics for a) SLEO a-GaN, b) planar a-GaN, and c) planar c-GaN devices with undoped barriers.

Both nonpolar planar and SLEO LEDs had the breakdown voltages in between -7 – -9V while polar c-GaN had much larger breakdown voltages ~-24 – -30 V. Much speculation can be made about such low breakdown voltages observed for high quality SLEO templates. However none would be accurate, since the size of

the devices ($400 \times 400 \mu\text{m}$) was big enough to have regions clear of defects as well as regions with clustered defects within the same device area which would eventually affect the break-down characteristics. Kozody et al. [27] suggested a way to get around this problem by designing small enough devices in a size that it would less possibly include these clustered defects. In order to understand the leakage and break-down characteristics, one needs to follow such a method.

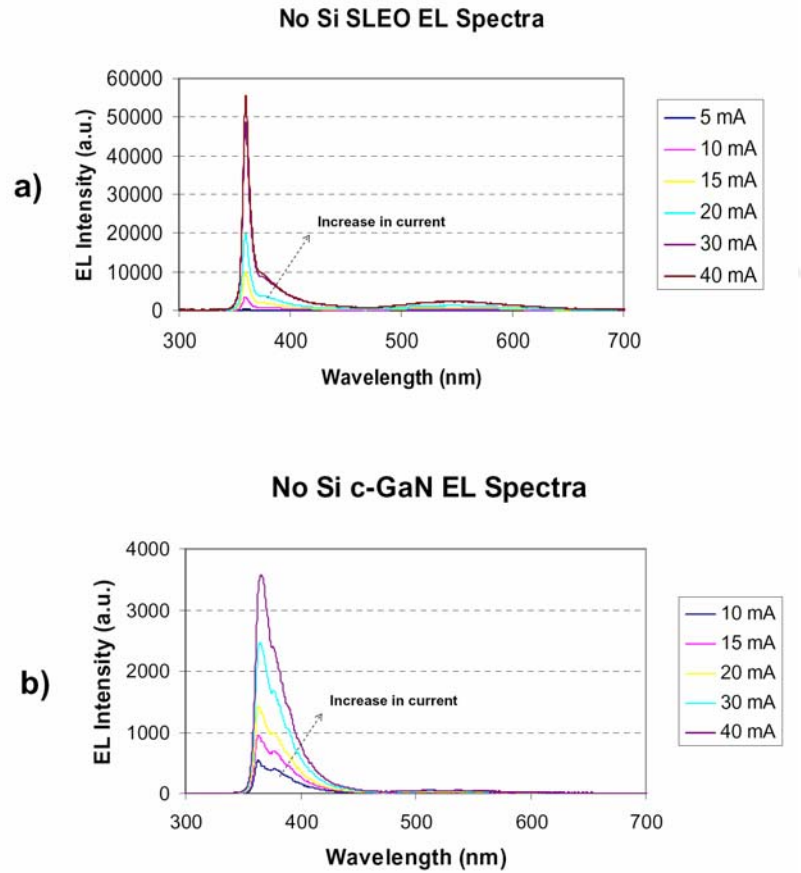


Figure 4.16 EL spectra at currents ranging between 5 – 40 mA for a) SLEO a-GaN, and b) planar c-GaN devices with undoped barriers.

In EL spectra (Fig. 4.16), other than GaN bandedge emission, two other peaks are present. One is deep yellow luminescence (YL) at ~ 2.2 eV and the other is emission at ~ 370 nm (~ 3.35 eV) which was associated with absorption and re-emission of MQWs in the underlying n-type GaN layers [28]. The self-absorption emission at 370 nm was in different shapes and intensity for a-plane SLEO GaN and c-GaN. This can be related to different GaN template thicknesses, presence of SiO₂ in SLEO and relative luminous intensity of these two materials with respect to the absorption peak. SLEO material has much higher GaN thickness, therefore there is more optical loss than in planar c-GaN. Even with these extra losses, SLEO LEDs have 15 times higher intensity and 6 times less FWHM values. On the other hand, SiO₂ scatters the light which may create higher light extraction efficiency.

As shown in Fig. 4.17, the c-GaN emission peak has shifted from 363 nm to 366 nm with a change in drive current due to the thermal effects. The emission wavelength at 360 nm for SLEO does not change with increase in drive current, which makes clear that the quantum well-band edge emission lacks polarization field effects and better heat sinking capabilities.

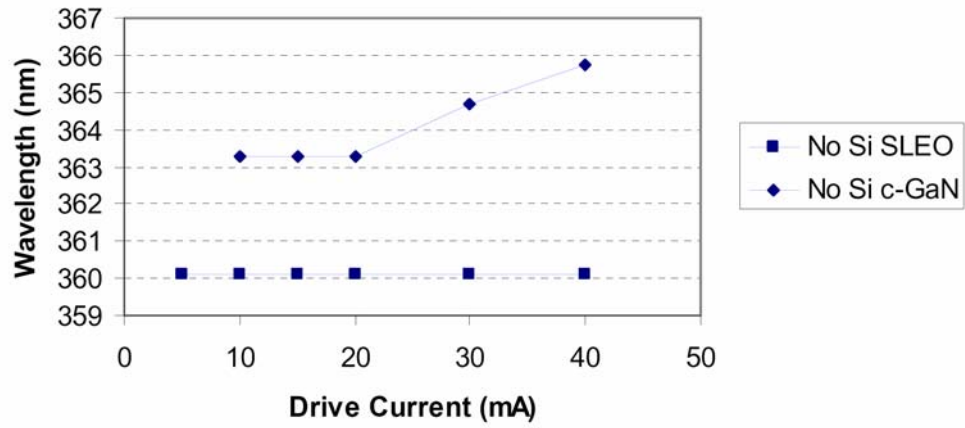


Figure 4.17 Change in emission wavelength with increase in drive current for SLEO a-GaN and planar c-GaN devices with undoped barriers.

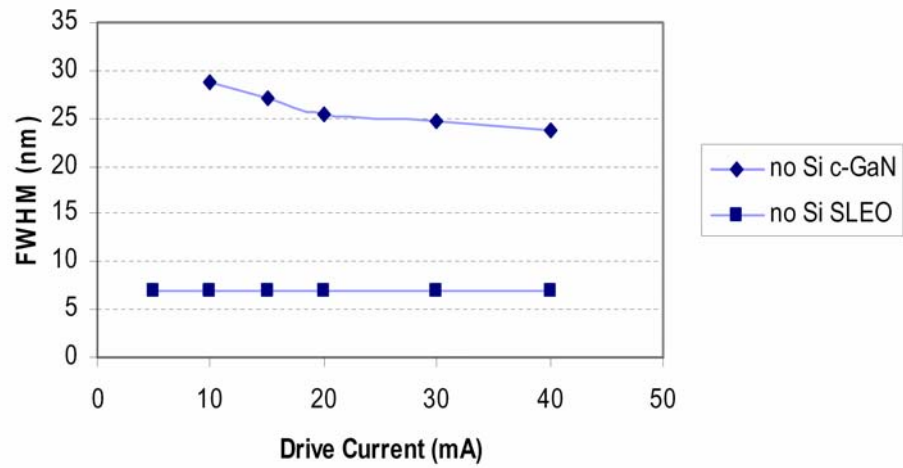


Figure 4.18 Change in FWHM of the EL emission with increase in drive current for SLEO a-GaN and planar c-GaN devices with undoped barriers.

With increase in drive current, the FWHM did not get affected for SLEO, which suggests that the device heating was not a big issue due to reduced internal resistance related with defects and large film thickness. However, for planar c-GaN FWHM value decreases with increase in drive current, which was the opposite of what was expected. This phenomenon is related with self-absorption emission at 370 nm. As the drive current increases, the bandgap emission intensity increases and with QCSE wavelength shifts from 363 to 366 nm, getting closer to the 370 nm emission peak. As the two peaks approach each other FWHM decreases. Therefore, the trend in FWHM was the combined effect of wavelength shift and relative intensity of bandgap emission versus self-absorption emission and can not be directly related to heat dissipation.

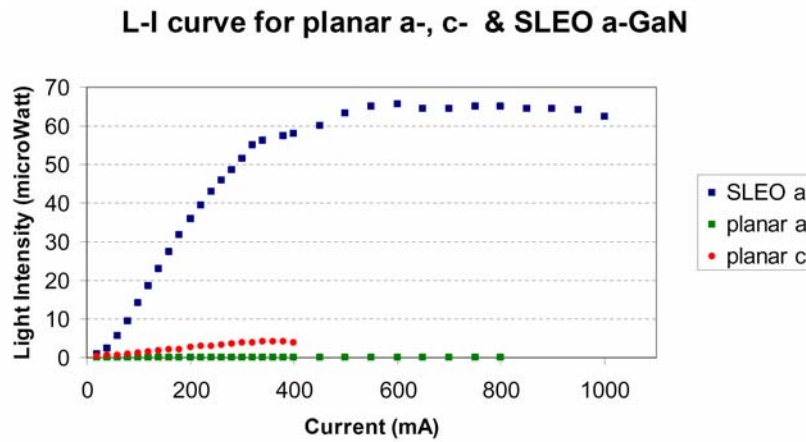


Figure 4.19 L-I characteristics of planar a-plane, planar c-plane, and SLEO a-plane GaN/Al_{0.15}GaN unpackaged LEDs.

The dependence of light intensity for SLEO a-GaN, planar a- and c-GaN LEDs on the drive current (L-I) curve is illustrated in Fig. 4.19. The drive current ranged from 20 mA to 1 A, depending on the material quality. For SLEO LEDs the output power at 20 mA was in the range of 2.5 – 6.5 μ W for various types of devices. For planar a-GaN films this value was 100 times lower, in the range of 0.02 – 0.05 μ W. For planar c-GaN films the output power was in the range of 0.3 – 0.5 μ W. Because the device structure and doping levels for this LED set were not optimized, the relative values have more significance than the absolute values.

Figure 4.20 demonstrates the effect of contact geometry on the performance of planar a-GaN LEDs. The interdigitated contacts exhibited the highest power output at 0.068 μ W and roll-off current at $\sim$ 400 mA, while for U-shape contacts these values dropped down to 0.059 μ W and $\sim$ 350 mA, respectively. For L-shape contacts the power out and roll-off current were 0.05 μ W and $\sim$ 400 mA, respectively. The higher roll-off currents can be directly attributed to lower device heating due to improved conductivity with better current distribution and lower series resistance. When there is heat in the device there is thermal impedance so there is relationship between the power and resistance. “ $P_{\text{without heating}} - P_{\text{with heating}}$ ” depends on the power dissipation in the device, defined as “ $I^2 \times R$ ” thus the higher the resistance, the higher the power dissipation. Diode, contact and spreading resistance are the major contributors for series resistance. In this case, the material quality is the same and so is the internal resistance. However, since the contact geometry, contact and spreading resistances are different, they contribute differently

in series resistance. Also, reduced output power for L-shaped devices can be attributed to a current crowding effect which prevents the uniform illumination throughout the surface.

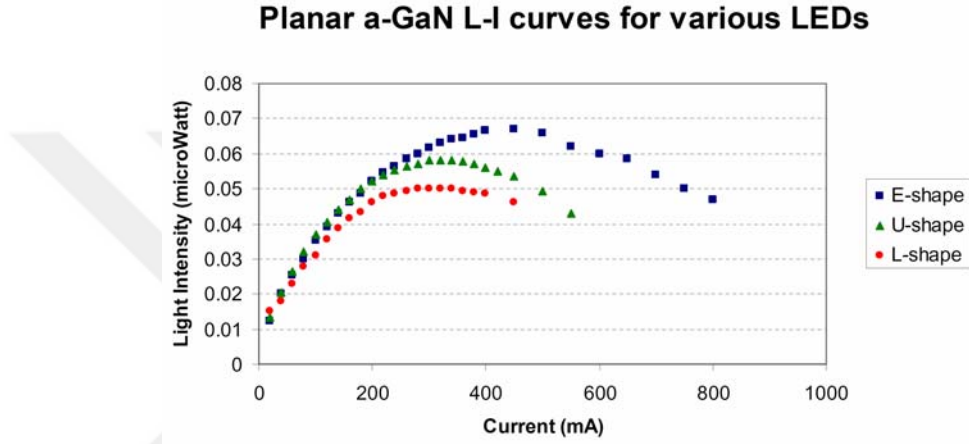


Figure 4.20 L-I characteristics of planar a-plane GaN/Al_{0.15}GaN unpackaged LEDs for Interdigitated (E-shape), U-shape, and L-shape devices with 400 x 400 μm junction area.

External quantum efficiency (EQE) is defined as:

$$\eta_{\text{ext}} = \eta_{\text{injection}} \times \eta_{\text{internal}} \times \eta_{\text{extraction}} = \frac{P / h \cdot \nu}{I / e}$$

where;

$\eta_{\text{injection}} \approx 1$ (which can be controlled with doping levels)

$$\eta_{\text{extraction}} = \frac{\text{\#of photons emitted to free space per second}}{\text{\#of photons emitted from active region per second}}$$

$$\eta_{\text{internal}} = \frac{\text{\#of photons emitted from active region per second}}{\text{\#of electrons injected into LED per second}}$$

$$= \frac{1}{1 + \frac{\tau_{\text{radiative}}}{\tau_{\text{non-radiative}}}} = \frac{P_{\text{int}} / h \cdot \nu}{I / e}$$

In order to figure out the internal quantum efficiencies, the devices should be packaged and all the light generated should be collected with an integrating sphere or time resolved photoluminescence experiments should be carried on the wells. Since the testing was done on an unpackaged wafer, only the EQE and the wall-plug efficiency values will be analyzed. The wall-plug efficiency is defined as:

$$\eta_{\text{wall-plug}} = \frac{P}{I \cdot V} \quad \text{where } P \text{ is power out and } I \cdot V \text{ is the electrical power provided}$$

to the LED which was calculated from $V = V_{\text{turn-on}} + I \cdot R_{\text{series}}$ for each device. The turn-on voltage for the devices is essentially the bandgap energy which was defined as “ $h \cdot \nu$ ” in external quantum efficiency equation previously. Therefore the wall-plug efficiency contains an extra series resistance dependent term in the denominator. If the device is an ideal LED with low series resistance, wall-plug efficiency gets closer to EQE, i.e. as $R \rightarrow 0$, $V \rightarrow V_{\text{turn-on}}$. As a result, although both EQE and wall-plug efficiencies are expected to follow parallel trends, wall-plug efficiencies will be lower than EQE values. This can be seen in the following Figures 4.21 b), 4.22 b), and 4.23 b) for SLEO a-, planar a- and planar c-GaN LEDs, respectively.

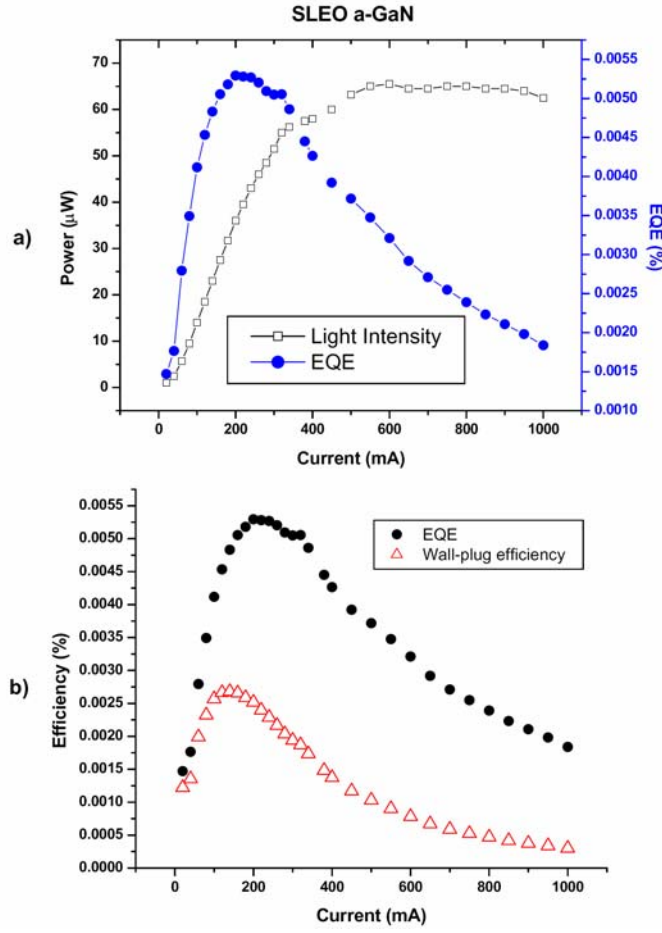


Figure 4.21 For on-wafer a-plane SLEO LEDs a) change in output power and external quantum efficiency, and b) change in external quantum efficiency vs. wall-plug efficiency with increase in drive current

For SLEO LEDs the highest EQE was 0.0055% at 200 mA after which the EQE started to decrease gradually with increasing drive current up to roll-off current of 1 A. The decrease in EQE and saturation of power output were related to heating. Since the internal resistance was relatively lower, longer decay range was observed

for SLEO LEDs compared with the planar counterparts. This EQE value is the first reported value for nonpolar UV LEDs.

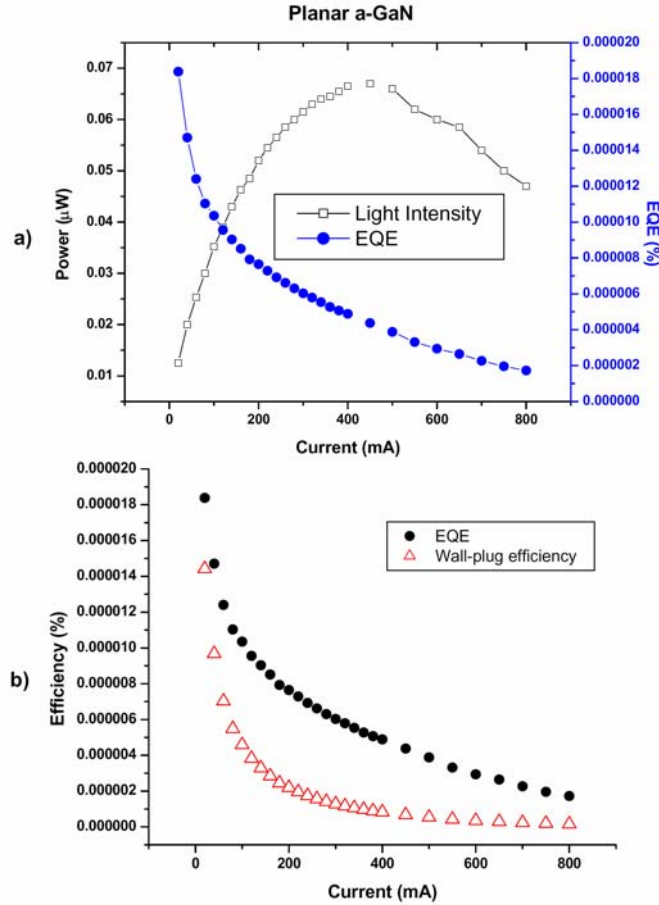


Figure 4.22 For on-wafer a-plane planar LEDs a) change in output power and external quantum efficiency, and b) change in external quantum efficiency vs. wall-plug efficiency with increase in drive current

It is shown in Fig. 4.22 that while planar a-GaN LEDs output power increases with increasing current, there is no observable peak for EQE due to high internal resistance related with high defect densities. The highest EQE obtained for planar a-GaN LEDs was 0.00002%, which was almost 275 times lower than what

was observed for SLEO LEDs. Considering the fact that both a-plane LEDs were free from polarization fields and both of them were grown and processed together, the tremendous amount of difference in EQE could come only from the presence of the defects. By just lowering the defect densities 3-4 orders of magnitude, it was possible to improve device efficiencies 275x.

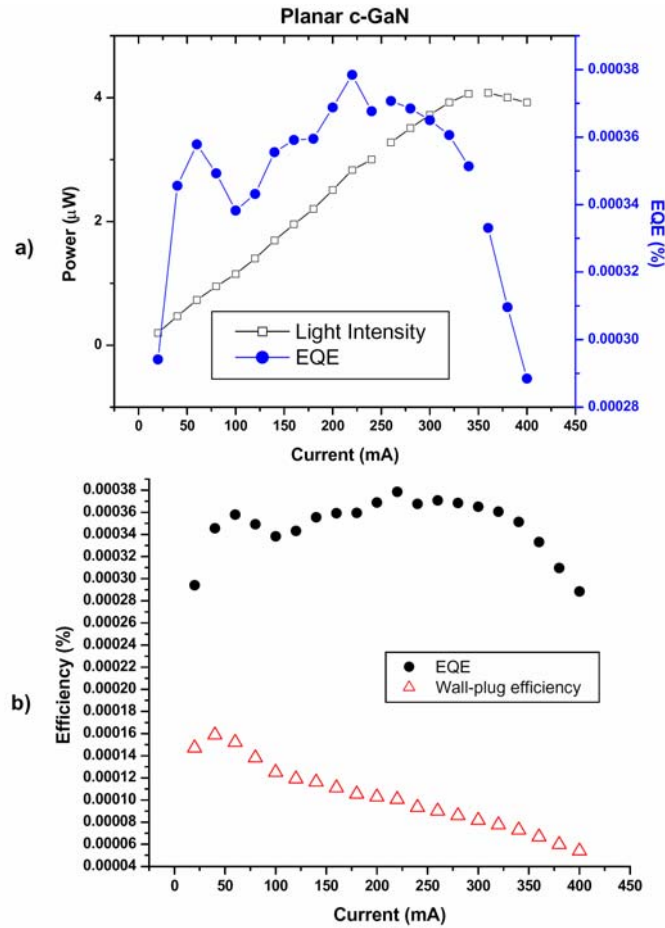


Figure 4.23 For on-wafer c-plane planar LEDs a) change in output power and external quantum efficiency, and b) change in external quantum efficiency vs. wall-plug efficiency with increase in drive current

The highest EQE obtained for polar planar c-GaN LEDs was at 200mA with 0.00038%, which was ~20x higher than planar a-GaN LEDs and ~15x lower than high quality SLEO LEDs. Although it is not very accurate to compare two different materials that are polar and nonpolar, considering the fact that c-GaN LEDs had QCSE but relatively lower dislocation densities than nonpolar planar a-GaN LEDs, the major contributor to 20x difference in EQE was the presence of defects. Also, both SLEO and planar c-GaN LEDs demonstrated similar trends in device efficiencies. However c-GaN LEDs heated at much lower currents and rolled-off at ~400 mA. This also indicates that defect reduction enhances the device lifetime. Another important difference between the SLEO and the planar c-GaN LEDs was the relative values of the EQE and wall-plug efficiencies. For planar c-GaN LEDs wall-plug efficiency values deviated from EQE values much more due to high series resistance.

4.3.3. Effect of Si doping in the barriers

Since the device structures were grown at low pressure (76 Torr), to compensate any impurity incorporation we introduced a very low level (0.1 sccm) of DiSi flow, corresponding to $2 \times 10^{18} \text{ cm}^{-2}$ Si concentration. Therefore self-compensation of Si was out of question. According to Fig. 4.24 and 4.25, barrier doping with Si did not affect the GaN emission wavelength for SLEO, while for planar c-GaN the wavelength was shifted to lower energy levels compared with the undoped barriers. In terms of wavelength shift with increase in drive current, although the wavelength

red-shifted only 1 nm at 40 mA for doped barriers, this shift was 3 nm for undoped barriers in the case of planar polar c-GaN samples. However both samples with doped and undoped barriers followed the self-heating related red-shifting trends. Self-absorption emission at 370 nm was broader and stronger when the barriers were

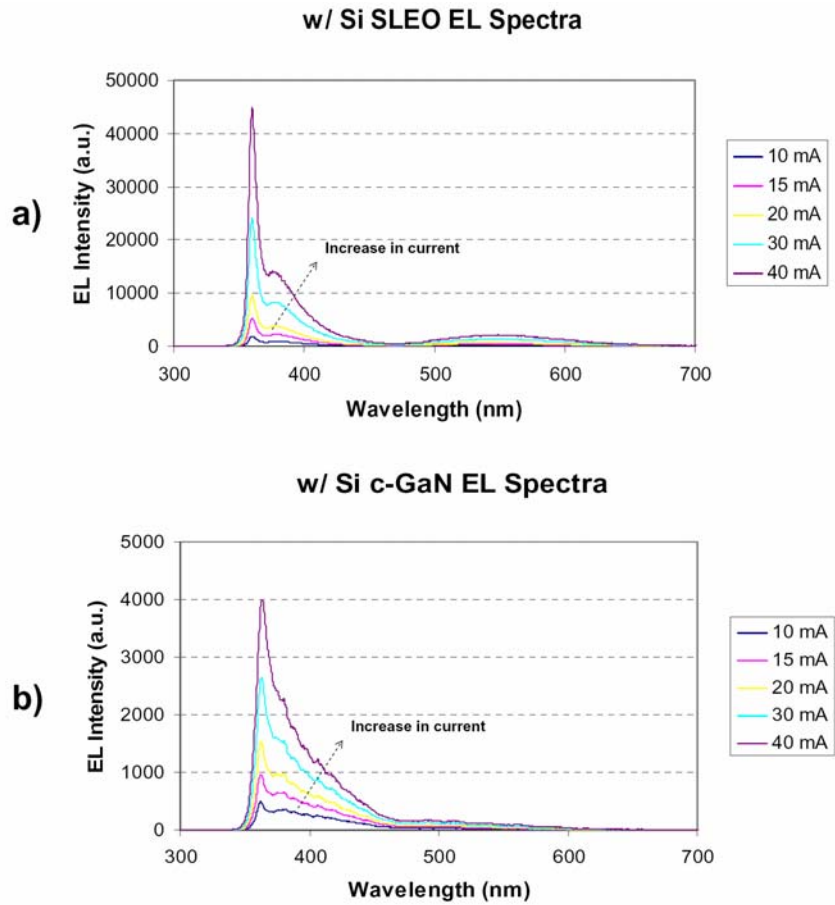


Figure 4.24 EL spectra at currents ranging between 10 – 40 mA for a) SLEO a-GaN, and b) planar c-GaN devices with Si doped barriers.

doped for both SLEO and planar c-GaN devices. This more effective absorption peak can be a consequence of reduced band-edge emission intensity, making the relative intensity of self-absorption emission to band-edge emission much more prominent. As a result, higher FWHM values were observed in doped barrier planar c-GaN samples (Fig. 4.26). Yellow luminescence (YL) at 2.2 eV seemed more dominant in the case of Si doped barriers for SLEO samples due to higher relative intensity with respect to lowered GaN band-edge emission. Also, YL emission for SLEO (that c-GaN lacks) can be explained by the lower surface temperature during growth in SLEO films. SLEO films were at least 20 microns thicker than planar c-GaN films, leading to poor thermal conduction to the surface. As a result, the actual growth temperature for SLEO samples was lower, which tends to allow more carbon and oxygen incorporation [11, 29, 30]. Si doping study explored by Keller et al [31], revealed that with increasing oxygen incorporation in the material, the distance of the donor-acceptor pairs decreases and the transition moves to higher energy, i.e. from 410 - 440 nm to lower wavelength, and oxygen acts as acceptor in the presence of Si doping. In the EL spectra, the observed second peak broadening may be related not only to self-absorption but also to oxygen related luminescence.

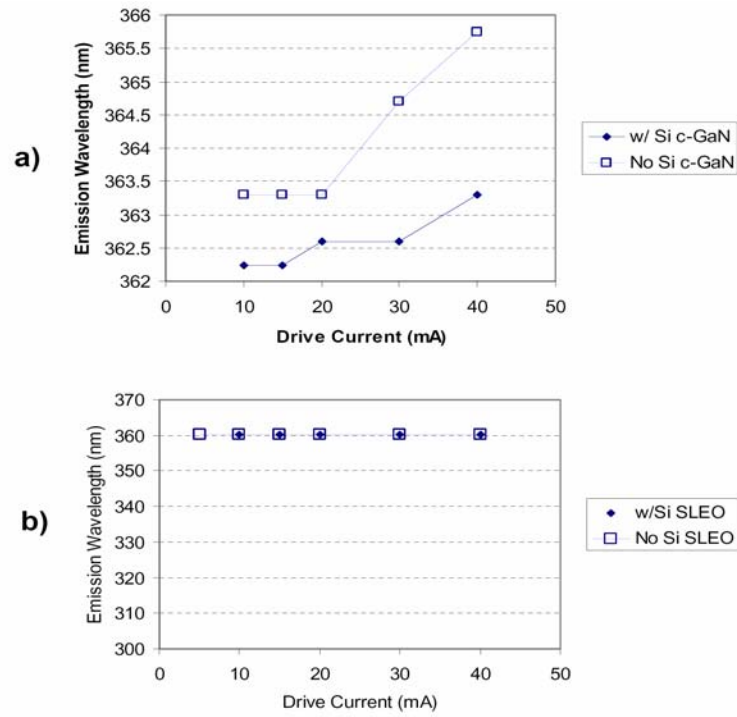


Figure 4.25 The effect of Si doping in the barriers on change in emission wavelength with increase in drive current for a) planar c-GaN devices, and b) SLEO devices.

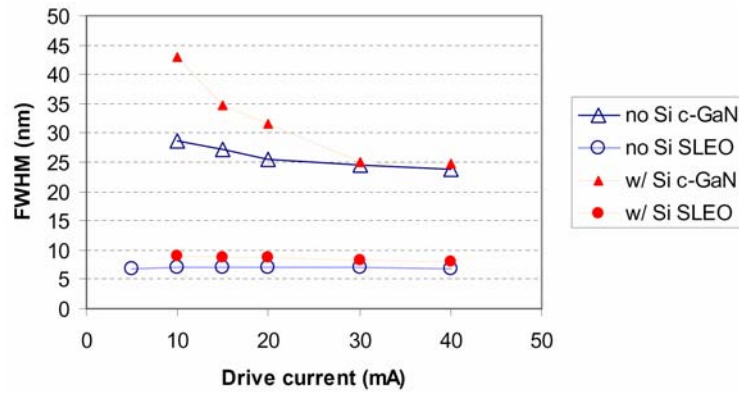


Figure 4.26 The effect of Si doping in the barriers on change in EL emission FWHM with increase in drive current for SLEO a-GaN and planar c-GaN devices.

Reduced EL intensity in barrier doped samples can be explained by surface roughening. Murakami et al. [32] and Nakamura et al [33] reported that with Si concentrations over $1 \times 10^{18} \text{ cm}^{-3}$ surface quality degrades forming V-shaped defects eventually degrading PL intensity.

Another reason for reduced EL intensity for the doped barrier samples can be insufficient Si doping levels. Interesting research has been done by Hirayama and Aoyagu [21] supporting this argument. They used $\text{Al}_{0.24}\text{Ga}_{0.76}\text{N}/\text{Al}_{0.11}\text{Ga}_{0.89}\text{N}$ MQW structures with doping levels in the barriers to compare the effect of n-type Si doping. They demonstrated that Si-doping is very effective enhancing the emission of AlGaIn QWs as long as the Si doping concentrations were in between 8×10^{18} - $5 \times 10^{19} \text{ cm}^{-3}$. They observed highest intensity at a Si-doping concentration of $2 \times 10^{19} \text{ cm}^{-3}$. Therefore there is an optimal doping level to accomplish emission enhancement.

Figure 4.27 shows the current voltage characteristics for the doped and undoped barrier samples. For both planar c-GaN and SLEO a-GaN samples the series resistance values doubled due to increased internal material resistance.

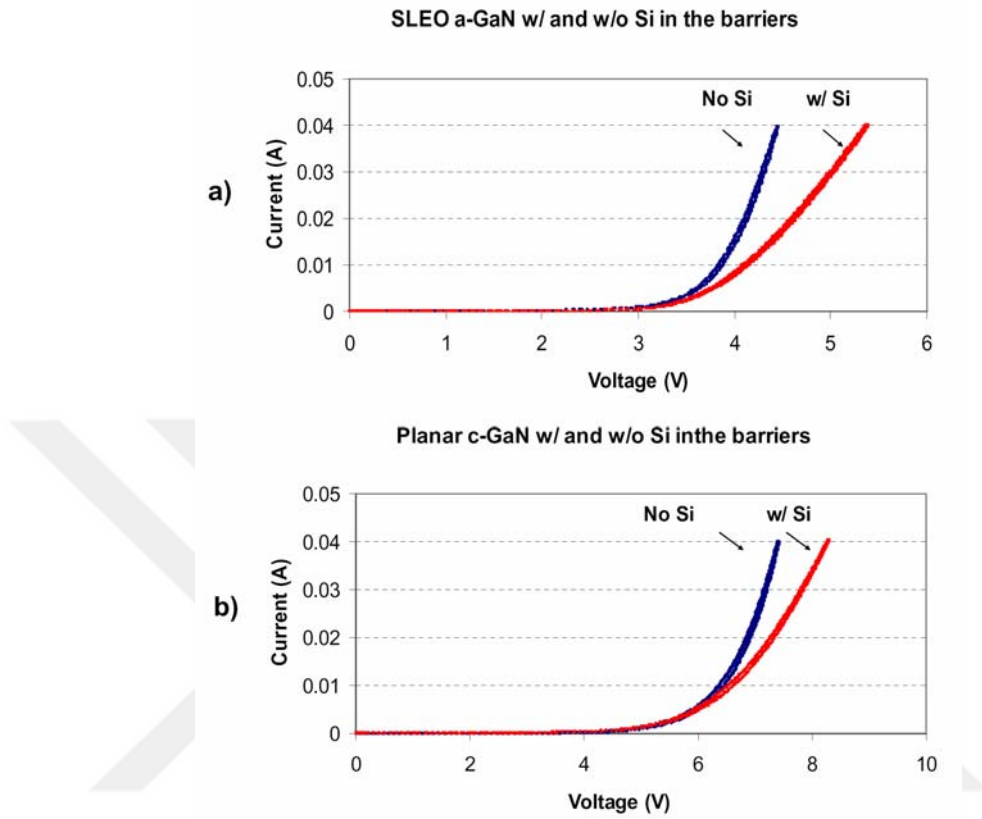


Figure 4.27 The effect of Si doping in the barriers on I-V characteristics for a) SLEO a-GaN, and b) planar c-GaN LEDs.

4.4. Conclusion

The two major challenges to improve the device performances can be summarized as eliminating polarization field and related effects, and reducing the defect densities in the material. Both of these challenges have been overcome by growing high quality reduced defect density (TDs of 10^6 - 10^7 cm⁻² throughout the material) nonpolar a-plane GaN templates using sidewall lateral epitaxial

overgrowth (SLEO) method described in Chapter 3. In this chapter, electrical and optical properties of this high quality material were investigated.

According to the Hall and TLM measurements SLEO material exhibited ~ 2 times higher electron mobilities with $\sim 10\%$ higher Si ionization at same level of doping. The anisotropic conductivity was found to be function of defect density.

The effect of defect densities, polarization fields, Si doping in the barriers, and the contact geometry on electrical and optical properties of devices were investigated by comparing LEDs with different defect levels, polar and nonpolar nature, doped and undoped barriers, and various device patterns.

Both electrical and optical performances of the devices with lower defect densities improved significantly. In order to be able to make a direct comparison between the planar films and the SLEO films, SLEO a-GaN (TDs $\sim 10^6$ - 10^7 cm $^{-2}$), planar a-GaN (TDs $\sim 10^{10}$ cm $^{-2}$) and c-GaN (TDs $\sim 10^8$ cm $^{-2}$) templates were co-loaded for device growth and processed together. The device structure and doping levels for this LED set were not optimized. Therefore, rather than absolute values, the relative values garner more significance. The SLEO LEDs exhibited high luminescence intensity (~ 10 - 15 x higher than planar c-GaN films) while emission from planar a-GaN films were extremely weak which made capturing EL emission extremely hard. For SLEO LEDs the output power at 20 mA was in the range of 2.5 – 6.5 μ W for various types of devices, for planar a-GaN films this value was in the range of 0.02 – 0.05 μ W, and for planar c-GaN films the output power was in the range of 0.3 – 0.5 μ W. The typical series resistance values for SLEO a-GaN, planar c-GaN, and planar a-GaN LEDs were ~ 15 Ω , ~ 30 Ω , and ~ 40 Ω , respectively.

With the high quality SLEO material, ~100 -300 times higher EQE and ~2.5-3 times lower series resistance values were obtained compared with high defect density planar films. Therefore, effective defect reduction along with elimination of the polarization fields is essential for improving upon the conductivity, doping ionization, electron mobility, radiative recombination, device lifetimes and so ultimately the UV LED performances. In order to realize the true potential of the nonpolar SLEO UV LEDs the device structure, doping levels, and mask design need to be optimized.

The effect of contact geometry was also investigated. The interdigitated contacts yielded the highest power out, lowest series resistance, and the best heat management properties compared with the U- and L-shaped contact geometries due to better current spreading, lower contact resistance, and alleviated current crowding.

Tables 4.2 and 4.3 summarize the effect of Si doping in the barriers. It was concluded that with introduction of Si at a level of $2 \times 10^{18} \text{ cm}^{-3}$, the material's internal resistance increases, translating to increase in the series resistance for both planar c-GaN and SLEO a-GaN LEDs. Si atoms act as scattering centers in the material instead of compensating impurities at such low concentration level. Also the surface quality degrades at low levels of Si concentration. As a result the EL intensity decreases.

Table 4.2 The effect of Si doping on Device performances of SLEO a-GaN LEDs

MATERIAL: SLEO a-GaN	With Si doping in the barriers	With NO Si doping in the barriers
Series resistance (Ω)	37	17
V_{TURN-ON} (eV)	3.9	3.75
V₂₀ – forward voltage (eV)	7.26	4.1
FWHM (nm) @ 20 mA (nm)	9.067	7.0682
EL Intensity @ 20 mA (nm)	9,492.9	19,848.9
Wavelength (nm)	360.13	360.13

Table 4.3 The effect of Si doping on Device performances of planar c-GaN LEDs

MATERIAL: PLANAR C-GaN	With Si doping in the barriers	With NO Si doping in the barriers
Series resistance (Ω)	57	30
V_{TURN-ON} (eV)	6.1	6.2
V₂₀ – forward voltage (eV)	7.26	6.83
FWHM (nm) @ 20 mA (nm)	31.6066	25.38254
EL Intensity @ 20 mA	1,530.9	1,429.37
Wavelength (nm)	362.59	363.3

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CHAPTER 5:

Conclusion and Future Work

5.1. Nonpolar Material Research

In Chapter 2 we showed that m-plane surfaces are more stable than a-plane. During optimization of m-GaN films to characterize the crystal quality, the full width at half maximum (FWHM) values of ω -scan X-ray rocking curves were measured for the $(1\bar{1}00)$ on-axis with the in-plane a- and c-axis coaxial with incident and scattered wavevectors, and for $(10\bar{1}2)$ off-axis. However it was recognized that the broadening of the $(1\bar{1}00)$ on-axis m-plane reflections parallel to the c-axis is sensitive to stacking disorders whereas for a- $(11\bar{2}0)$ plane the on-axis reflections parallel to c-axis are insensitive to stacking disorders. Therefore the FWHM values for m- and a-plane films could not be compared directly. The issue of peak broadening from stacking faults should be resolved.

Since the first device-quality smooth heteroepitaxial a-GaN films were obtained by Mike Craven [1, 2], the presence of high extended defect densities in nonpolar a-GaN has been the primary obstruction in obtaining high efficiency nonpolar devices. Conventional single-step LEO [3] and two-step LEO [4] methods have been utilized to reduce these defects, however due to limited growth window of a-plane films, full coalescence could not be achieved and when it was achieved, it was not reproducible. With a novel approach, SLEO, it was shown in Chapter 3 that

it is possible to coalesce these films while reducing the defects effectively, and also these results were reproducible.

Nonpolar m-plane GaN has one order of magnitude lower defect densities compared with a-plane GaN. According to the most recent single step LEO results for m-plane GaN grown over m-plane SiC by HVPE the TDs were reduced to $\sim 5 \times 10^6 \text{ cm}^{-2}$ from $4 \times 10^9 \text{ cm}^{-2}$, and the SFs as low as $3 \times 10^3 \text{ cm}^{-1}$ were achieved compared to $1 \times 10^5 \text{ cm}^{-1}$ for planar m-GaN in the wing region [5]. If SLEO technique was applied to m-plane GaN it would be possible to achieve the wing quality throughout the film. Another advantage of the m-plane GaN films is their wide growth stability window compared with their nonpolar counterpart a-plane films as confirmed in Chapter 2. Therefore, the SLEO method should be studied for defect reduction in m-plane films with MOCVD and HVPE or combination of both.

Other promising crystal orientations are the semipolar films such as $(10\bar{1}1)$ and $(10\bar{1}3)$. Recently Troy Baker et al. demonstrated the semi-polar plane growth on $(10\bar{1}1)$ and $(10\bar{1}3)$ oriented sapphire [6], and (100) and (110) oriented spinel [7]. They found out that the growth stability window for semi-polar GaN planes was large. Just like in nonpolar nitrides, the polarization related detrimental effects were alleviated on semi-polar surfaces. Also reported TD and SF densities were very similar to nonpolar GaN films. Therefore SLEO growth should be employed as a defect reduction technique for semi-polar surfaces as well.

For nonpolar a- $(11\bar{2}0)$ plane GaN, thus far only one stripe orientation, $\langle 1\bar{1}00 \rangle_{\text{GaN}}$, was tested with SLEO due to dislocation confinement under the mask with straight Ga and N face sidewalls. If the stripes were patterned along $\langle 0001 \rangle_{\text{GaN}}$ because of m-facet formation the dislocations would bend toward these free surfaces for conventional single-step LEO. If the $\langle 0001 \rangle_{\text{GaN}}$ orientation was used for SLEO, the bending dislocations may annihilate each other in the first stage of coalescence. It would be interesting to investigate the effect of another stripe orientation, $\langle 0001 \rangle_{\text{GaN}}$, on material quality to make a comparison between these two stripe orientations.

In Chapter 3 it was demonstrated that by reducing defect densities the growth stability window was enlarged. Even at atmospheric pressure a-plane growth was achievable on these high quality templates. Therefore higher growth pressures can be experimented with device growth in order to limit the impurity incorporation which in turn should improve the doping and carrier ionization efficiencies.

Due to higher thermal conductivity of GaN, the output power of the LEDs saturate at higher current injections [8]. Therefore producing freestanding a- or m-plane SLEO GaN by HVPE would benefit from excellent thermal conductivity along with the reduced defect densities.

Planar nonpolar films, both a- and m-GaN, have a complex nature of defect formation although most TDs forms along the growth direction with a screw component, there is a considerable amount of dislocations with mixed nature [1]. It

is important to understand the interaction of these defects during SLEO growth. The electrical conductivity and its' relation to defect structure in SLEO material should be understood well in order to model the devices grown on top. Therefore an extensive TEM study should be carried out to reveal the complete defect formation mechanisms in SLEO templates and then this should be followed by luminescence and electrical studies.

SLEO nonpolar films are an excellent candidate to study the impurity incorporation such as C, H, or O, and their interaction with the microscopic defects such TDs and SFs when compared with planar nonpolar films.

5.2. Nonpolar Device Research

By utilizing the SLEO technique in nonpolar planes, two of the most substantial challenges polarization and extended defects related to detrimental effects were overcome. There is still a vast area however to explore in nonpolar device research.

After the emergence of the SLEO technique presented in this dissertation, it is now possible to achieve ultimately high efficiency devices given that the proper growth and processing parameters are tuned in. The preliminary 360 nm UV LEDs presented in Chapter 4 were not optimized however it was sufficient to verify the improvement in device performance both electrical and optical by comparing defected planar LEDs and reduced defect density SLEO LEDs. As the results show, by utilizing SLEO grown nonpolar templates, we can achieve an increase in EQE by

~100-300x, a lower series resistance by ~2-3x, alleviated heating related issues, and potentially increased device lifetimes.

EQE is composed of two main components; internal quantum efficiency (IQE) and extraction efficiency. Therefore improvement in EQE can be accomplished by optimizing these two variables. The efforts toward eliminating polarization fields by growing on nonpolar surfaces, and reducing defect densities effectively by means of SLEO were to improve internal quantum efficiencies.

In conventional LED structures in order to improve the IQE further, the optimization of the active region such as using wider wells, reducing or increasing the number of wells, and optimization of the doping levels and the critical thicknesses of the n- and p-cladding layers are necessary. Another way of improving IQE is to dope the quantum wells with In, which would also prevent absorption, by taking advantage of carrier localization effect. As a result of spinodal phase decomposition within InGaN quantum wells In rich nanostructures forms which causes carrier localization effects and in turn enhances IQE [9, 10].

There are many ways to improve extraction efficiencies. The most well-known method is packaging with novel escape cone geometries that would enhance the light extraction. Also a carefully designed LED device mask such as interdigitated finger patterns or micro disks can enhance extraction efficiencies as well. In addition, transparent contacts such as ZnO would advance the light extraction from the top of the devices. Therefore any or all of these methods should be utilized in the SLEO devices.

In order to improve the extraction efficiencies for 365 nm and lower wavelength UV devices, the self-absorption issue should be resolved by removing the substrate and GaN template. The n-cladding layer can be grown with the addition of a few percent Al in GaN. Then sapphire can be removed by laser lift-off [11] and this can be followed by polishing off the underlying SLEO GaN template. By following a similar process Morita et al. [12] was able to increase the EQE to 44% in 360 nm GaN/AlGaN LEDs. The self-absorption issue becomes more significant at lower wavelengths, therefore the SLEO method should be developed for growing nonpolar AlGaIn or AlN films for deep UV devices. To improve the extraction efficiency creating controlled surface faceting or texturing can also be experimented with so that more surface area is created for light to escape [13.]. Another method to resolve the self-absorption issue is to grow DBR structures between the SLEO templates and device structures as Craig Moe et al. demonstrated to prevent absorption by SiC substrates in deep UV AlGaIn LEDs [14].

Finally, InGaIn based longer wavelength devices, LEDs and LDs, in the visible range of the spectrum should be grown on these high quality nonpolar SLEO templates. Previously we attempted to grow $\text{In}_{0.25}\text{GaIn}/\text{GaIn}$ LEDs on a-plane SLEO templates, also Matt Schmidt et al. experimented with LED growth in the same wavelength range. The results suggested that In prefers to incorporate in the defect regions and SLEO material has an inhomogeneous distribution of the defects although overall defect densities were significantly reduced on average throughout the film. While In clusters around these inhomogeneously distributed defect sites, no In is incorporated in high quality material regions which leads to

inhomogeneous emission spectra. Therefore the growth conditions should be optimized to enforce homogeneous In incorporation throughout the film.

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Appendix A: TIME RESOLVED PL STUDY

A.1. Introduction

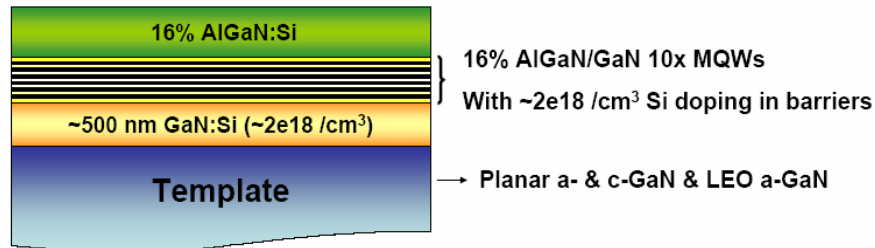
III-nitride devices are grown predominantly along the polar c- $\langle 0001 \rangle$ direction however due to a lack of center of symmetry in the wurtzite unit cell quantum wells grown along these orientations are subjected to the presence of spontaneous and piezoelectric polarization related electric fields. These fields would separate the electron and hole wavefunctions in the wells leading to a reduction in recombination efficiencies and oscillator strength, and an increase in radiative lifetimes ($\tau_{\text{radiative}}$). As mentioned throughout this thesis, nonpolar growth orientations of this material group along a- and m-directions have gained increased attention since 2000, after which it was proved that these nonpolar orientations lack such electric fields and their effects along the growth direction [1]. Another issue with III-nitrides and especially with nonpolar orientations is the presence of high defect densities. These defects, mainly composed of point defects, threading dislocations, and stacking faults, increase the nonradiative recombination centers and decrease the nonradiative lifetimes ($\tau_{\text{non-radiative}}$). These defects can although be significantly reduced by utilizing various lateral epitaxial overgrowth (LEO) methods. In this study time resolved photoluminescence (TRPL) studies were performed on planar polar c-plane and planar and low defect density nonpolar a-plane GaN/AlGaIn MQWs in order to fully understand the effect of growth

orientation and defect densities on the physical properties and improve upon the optical gain and quantum efficiencies of the 360 nm ultraviolet (UV) emitters. For applications such as chemical and biological sensors, solid-state lighting and displays, medical and healthcare, and defense UV light sources open up new possibilities. Therefore it is important to characterize the recombination dynamics of optical transitions and radiative recombination efficiencies of III-nitride UV photonic device structures. The time resolved optical studies are useful in providing necessary information regarding crystal quality, impurities, QW interface quality, and quantum efficiencies by means of optical transition dynamics, emission spectra, and emission characteristics. TRPL studies had been utilized primarily for material systems such as GaAs and its' alloys supporting longer wavelengths until the recent developments made in the large band-gap III-nitride material group. It is now possible to go down to 200 nm in spectral range with a few picoseconds (ps) time resolution. A TRPL system is composed of three main parts: a fast-pulsed laser, a monochromator, and a signal detector. The fast-pulsed laser employed while testing the structures presented in this chapter was a frequency tripled ps Ti:sapphire regenerative amplifier-pumped optical parametric amplifier, tunable through the visible spectrum and doubled to the ultraviolet. The TRPL signal was detected by a time-correlated single photon counting technique with a system response of 180 ps.

A.2. Experimental Procedures

A.2.1. Growth

GaN/AlGaIn multi quantum wells (MQWs) were deposited by metalorganic chemical vapor deposition (MOCVD) on co-loaded planar a-plane and c-plane GaN and lateral epitaxial overgrowth (LEO) a-GaN templates. LEO a-GaN templates were coalesced with hydride vapor phase epitaxy (HVPE) by Ben Haskell and LEO patterns were made of stripes with 2 μm window / 8 μm wing regions oriented along $\langle 1\bar{1}00 \rangle_{\text{GaN}}$. Prior to MQW region deposition a Si doped ($2 \times 10^{18} \text{ cm}^{-3}$) 0.5 μm GaN n-layer was grown. The MQW region consisted of 10 periods of GaN wells and $2 \times 10^{18} \text{ cm}^{-3}$ Si doped $\text{Al}_{0.16}\text{GaIn}$ barriers with varying well width between 3-20 nm at fixed barrier width of 10 nm and varying barrier widths between 5-15 nm at a fixed well width of 5 nm, as shown in Fig. A.1.1.



Varying well widths for 10 nm barriers (well/barrier-nm)					
3/10	5/10	7/10	12/10	15/10	20/10
Varying barrier widths for 5 nm wells (well/barrier-nm)					
5/5	5/8	5/10	5/12	5/15	

Figure A.1.1 GaN/AlGaIn MQW structure and varying well and barrier widths grown by MOCVD for PL and TRPL optical studies.

For all layers the growth pressure, growth temperature, NH_3 flow and total flow were 76 Torr, 1165 °C, 1 slpm and 10 slpm, respectively. For GaN and AlGaIn layers the TMGa flow was kept constant at 18 sccm, while for AlGaIn layers the TMAI flow was 23 sccm. The growth times for the wells and barriers were adjusted for varying widths.

A.2.2. Testing

Continuous wave (CW) photoluminescence measurements were taken using a 325 nm He-Cd laser at room temperature with excitation power density of 20 W/cm^2 .

Intensity dependent TRPL was carried out by Gregory A. Garret, H. Shen, S. Rudin and Michael Wraback from U.S. Army Research Laboratories. TRPL was acquired by gated down-conversion in a non-linear optical crystal with sub-picosecond resolution, and by time-correlated single photon counting (TCSPC) with a system response of 180 ps. Excitation laser pulses were derived from 250 kHz Ti:Sapphire regenerative amplifier-pumped optical parametric amplifier, tunable through the visible spectrum and doubled to the ultraviolet. Data were taken with pulses centered at 320 nm, just above the barrier band edge absorption (16% Al), and at 345 nm, just below the barriers, with fluences ranging from 2 to 200 $\mu\text{J}/\text{cm}^2$, or approximate free carrier areal densities per well of 2×10^{11} to $2 \times 10^{13} \text{ cm}^{-2}$. The spot size of the pump was much larger than that of the stripe pattern in the LEO sample, such that measurements are a weighted average of both wings and higher defect density windows. Time-integrated CW spectroscopy results were also provided.

The time resolved optical studies were performed to determine the effect of polarization, defect densities, well and barrier widths, pulse wavelength, and excitation intensity on recombination dynamics.

A.3. Results

The QWs were grown over planar a-GaN, planar c-GaN and HVPE grown LEO a-GaN templates with threading dislocation (TD) densities of $\sim 10^{10} \text{ cm}^{-2}$, $\sim 10^8 \text{ cm}^{-2}$, and $\sim 10^7 - 10^8 \text{ cm}^{-2}$ (in the wing region), respectively. Due to both polarity and defect density differences it would not be proper to compare planar a- and c-GaN samples, however defect densities for planar c-GaN and LEO a-GaN samples were similar. Any difference in luminescence characteristics between planar a- and LEO a-plane samples would be directly related to defect densities.

The direct comparison of room temperature CW-PL emission from planar and LEO a-GaN/AlGaIn QWs shows that one to two orders of magnitude reduction in TD density has a prominent impact on the luminescence performances. As can be seen from Fig. A.1.2 the PL intensity of LEO QWs was $\sim 20\times$ higher with respect to planar films. For LEO a-plane QWs, as the well width was lowered from 20 nm to 3 nm the PL intensity increased and the PL peak position blue-shifted due to better quantum confinement in the wells. The maximum intensity was achieved for 3 nm wide wells. On the other hand for planar a-plane films the maximum PL intensity was observed for 5 nm wide wells above which quantum confinement was reduced and below which defect related nonradiative recombination mechanics

dominated. Similar results for planar a-plane QWs were observed by Craven et al. [2].

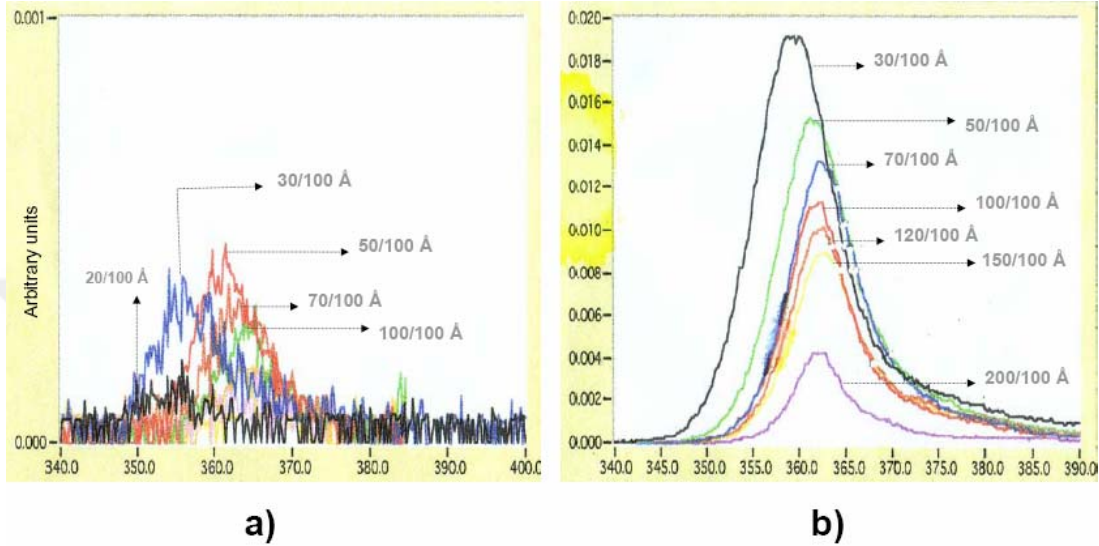


Figure A.1.2 CW-PL spectra for GaN/AlGaN MQWs grown on a) planar a-GaN templates, and b) LEO a-GaN templates.

Another CW measurement carried out by U.S. Army Research Lab revealed that although planar c-GaN and LEO a-GaN templates had similar TD densities, a-plane LEO MQWs yielded higher PL intensity most probably due to a lack of polarization related fields in the wells. The planar a-GaN/AlGaN QWs was brighter than planar c-plane QWs. On the other hand according to the TRPL results lifetime of c-plane was approximately an order of magnitude higher than a-plane. Accordingly even though having higher defect densities than planar c-plane, CW-PL was brighter on planar a-plane possibly because of surface roughness.

However the CW measurements can only offer average information while TRPL provides data beyond the capabilities of CW. Therefore to investigate the

effect of well width, barrier width, excitation power intensity, and pump wavelength on recombination dynamics TRPL studies were explored for polar and nonpolar QWs.

In order to improve upon device performances it is important to realize high internal quantum efficiencies. The internal quantum efficiency can be defined with two components: radiative ($\tau_{\text{radiative}}$) and nonradiative ($\tau_{\text{non-radiative}}$) recombination lifetimes

$$\eta_{\text{internal}} = \frac{1}{1 + \frac{\tau_{\text{radiative}}}{\tau_{\text{non-radiative}}}}$$

As $\tau_{\text{non-radiative}}$ increases or $\tau_{\text{radiative}}$ decreases it is possible to improve the internal quantum efficiencies. At room temperature recombination rates are dominated by nonradiative recombination transitions. Therefore short nonradiative lifetimes are favorable at room temperature. The total recombination is expressed as

$$\frac{1}{\tau} = \frac{1}{\tau_{\text{radiative}}} + \frac{1}{\tau_{\text{non-radiative}}} \Rightarrow \tau = \frac{\tau_{\text{radiative}} \cdot \tau_{\text{non-radiative}}}{(\tau_{\text{radiative}} + \tau_{\text{non-radiative}})}$$

The radiative recombination rate between carriers is defined as “ $R = B.n.p$ ” where B is the band to band transition coefficient, n is free electron concentration and p is free hole concentration. As the bandgap gets larger B gets smaller. Also the inverse of the “B.n” cross product can be defined as radiative lifetime ($\tau_{\text{radiative}} = (B.n)^{-1}$). Short recombination lifetime means that the nonradiative carrier recombination is dominant. Higher localization energy results in longer radiative

lifetimes. Lower localization energy excitons decays with radiative recombination. Radiative lifetime is expected to be shorter with free carrier recombination mechanisms. Therefore the total recombination lifetime should be related directly to exciton localization energy which is correlated with the activation energy E_0 . E_0 increases with increasing bandgap.

According to TRPL studies the a-plane LEO samples exhibited two decay times while planar films showed only one. The two separate decay times for LEO samples were attributed to emission from reduced defect density wing regions corresponding to longer decay times, and high defect density window regions. While the weak initial fast decay may reflect the quality of the material above the window region, the high quality of the MQW deposited on the larger wing regions of the LEO a-GaN is evidenced by the slow PL decay.

The well width dependence of decay times measured by time correlated single photon counting (TCSPC) for low pump intensity of $20 \mu\text{J}/\text{cm}^2$ and wavelength of 320 nm is displayed in Fig A.1.3. As a general trend for nonpolar a-plane QWs the PL decay times increased with increasing well width which is evidence of flat band conduction. For well widths 3 nm, 5 nm, 10 nm, and 20 nm, decay times were 371 ps, 420 ps, 585 ps, and 663 ps, respectively. The neighboring time integrated PL graph shows that as the lifetime decreases the intensity increases and blue-shifts with decreasing well width as was observed in CW-PL measurements previously.

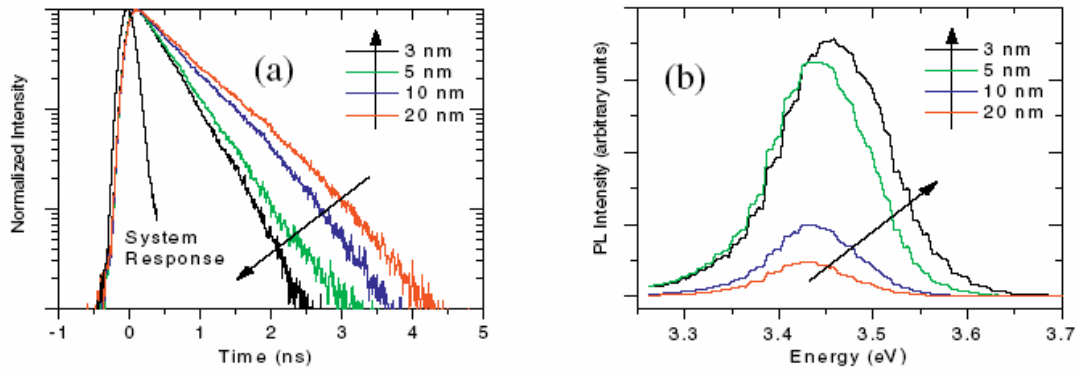


Figure A.1.3 a) Low intensity TRPL decays obtained by TCSPC, and b) time integrated PL spectra as a function of well width in a-plane LEO MQWs (Courtesy of Gregory Garret et al.[3]).

As opposed to nonpolar samples, in polar QWs the PL decay times declined with increasing well width due to electron hole wave function separation and reduced radiative recombination efficiencies as a result of QCSE. In polar structures the field can be screened by electron filling in the QWs leading to PL process with free electron-localized hole transition at the QW interfaces. Due to localized holes the radiative lifetimes would be higher than expected. The field screening and radiative lifetimes in polar QW structures can be controlled by varying the Al composition and well width as well as by different excitation densities and photexcited carriers.

High excitation densities saturate the defect related recombinations leading to stronger band edge emission. Fast lifetime decays with TCSPC as evidence, defect related nonradiative processes saturate even at low excitation density therefore these defect related nonradiative recombinations were not accountable for intensity dependent decays for nonpolar QW structures. In polar structures, due to QCSE, the

PL peak position shifts and shows dependence with change in excitation density. Due to screening of the built-in field at high excitation densities the emission from polar GaN wells are centered around 3.4 eV. On the other hand for nonpolar samples the peak position does not change with changes in excitation density. In this study the effect of excitation density on decay times and PL intensity with changes in well and barrier width for polar and nonpolar samples were the main focus. The predominant recombination at high excitation is through bimolecular processes with the saturation of nonradiative recombination channels. PL intensity should be linearly dependent on excitation power for nonpolar GaN wells [4].

At low pump intensity ($20 \mu\text{J}/\text{cm}^2$) the lifetimes for nonpolar LEO a-plane, polar planar c-plane, and nonpolar planar a-plane MQWs with 3 nm well width were 357 ps, 151 ps, and 4 ps, respectively. At high pump intensity ($200 \mu\text{J}/\text{cm}^2$) the lifetimes increased to 473 ps and 190 ps for nonpolar LEO a-plane and polar planar c-plane 3 nm MQWs, respectively. For the same 3 nm well width the PL intensity at $t=0$ for polar planar c-plane samples increased by a factor of 12.5 when the pump intensity was raised ~ 10 fold. However for the same increase in pump intensity, PL intensity of nonpolar LEO a-plane samples increased by a factor of 5.8. On the other hand decay time of both polar and nonpolar samples increased by the same factor of ~ 1.3 for ~ 10 fold increase in pump intensity. Therefore the source of intensity dependence in these two samples was different. For polar MQWs at higher pump intensity radiative lifetimes decrease by an increase in wavefunction overlap in the well and reduction in wavefunction penetration into the barriers leading to more effective polarization field screening with reduced recombination through barrier

and 2 DEG states. On the other hand for nonpolar MQWs at higher pump intensity radiative lifetimes increase by phase space filling of the single particle states with reduced binding energy and so the oscillator strength.

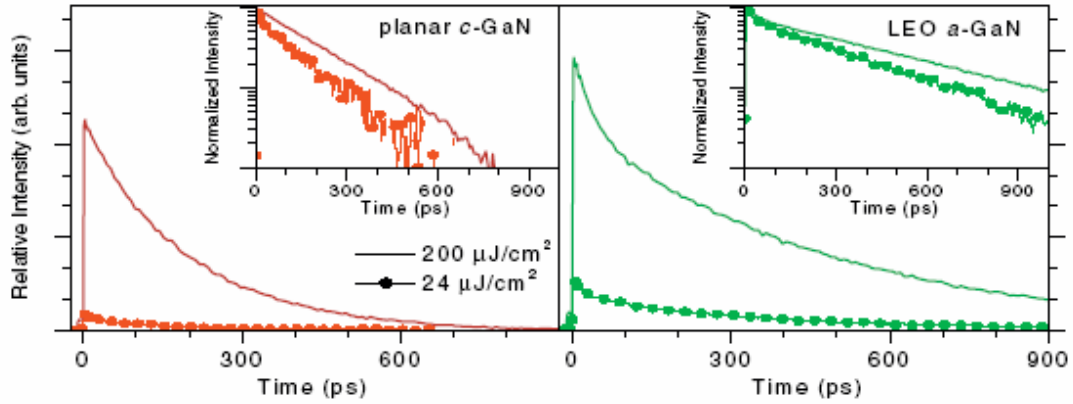


Figure A.1.4 Comparison of intensity dependent TRPL decays in GaN/AlGaN MQWs with 3nm well width deposited on planar c-GaN and LEO a-GaN templates (Courtesy of Gregory Garret et al.[3]).

The pump intensity dependence of the decay times for polar planar c-plane samples was attributed to QCSE. It was assumed that the dominant PL decay mechanism is nonradiative due to poor overlap of electron hole wavefunctions. Since the electron and hole wavefunctions separate in the wells, nonradiative recombination occurs through the barriers. At high carrier densities caused by high pump intensity, the field is screened and at $t=0$ the wavefunction is placed in the center increasing the PL intensity. After $t=0$ the luminescence starts to decay by various nonradiative mechanisms. Further evidence for this behavior can be found

in the comparison of the peak luminescence intensities for various wells at $t=0$ (Fig. A.1.5).

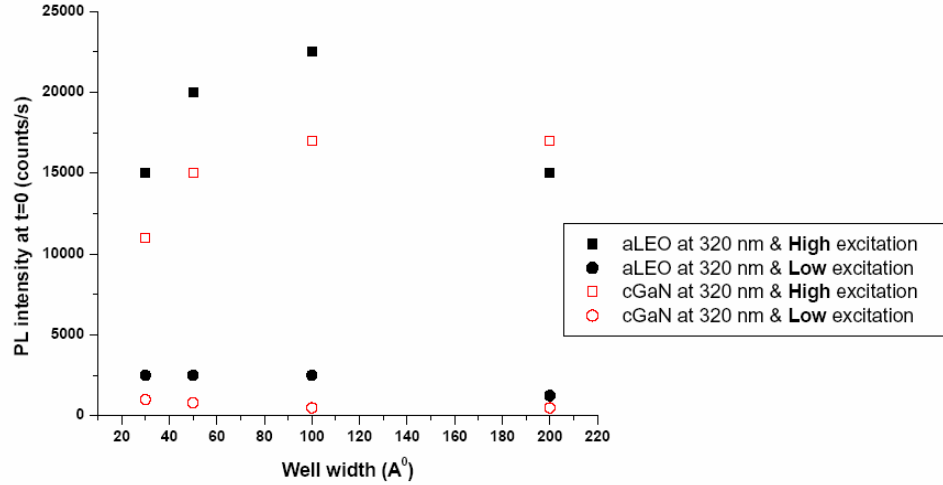


Figure A.1.5 PL intensity at $t=0$ in a-plane LEO and planar c-plane MQWs as a function of well width for low and high excitation power density.

For nonpolar a-plane MQWs as the well width decreased the radiative lifetime declined while all decay times were close to 500 ps of all well widths. As evidence at smaller well widths such as 3 nm the decay rate was faster, PL intensity was more intense and the pump intensity of PL intensity at $t=0$ was more sublinear due to better quantum confinement with higher oscillator strength and excitation binding energy (Fig. A.1.5 & Fig. A.1.6). From Fig. A.1.6, the pump intensity dependence of the decay times becomes smaller as the well width increases. With thicker nonpolar LEO a-plane 20 nm wells, the PL decay times were virtually independent of pump intensity, and PL intensity at $t=0$ had a linear relationship with

pump intensity as an evidence of lack of QCSE. This is a very important result since it shows that in the absence of electric field the wells can be grown thicker and still have a good wave function overlap without significant recombination through the barriers, thus improving radiative efficiency and luminescence lifetimes. At smaller well widths, wave function penetration into the barriers is still important and the intensity dependence at smaller well widths is an indication of the trap states saturation in the barriers. Therefore in order to achieve improved radiative efficiency and luminescence lifetime the wells should be grown thicker in flat-band conduction nonpolar QWs.

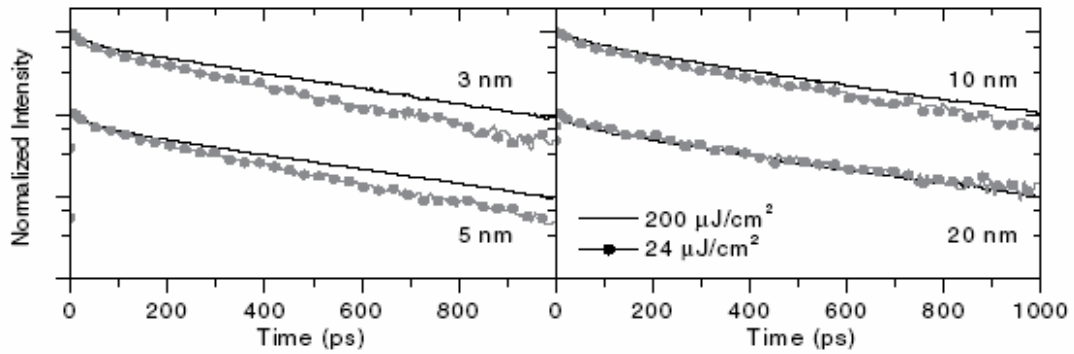


Figure A.1.6 Intensity dependence of TRPL decays in a-plane LEO MQWs as a function of well width (Courtesy of Gregory Garret et al.[3]).

In order to remove the wave function penetration into the barriers and saturation of traps in the barriers, the QWs were pumped right below the barriers at 345 nm so that the carriers were predominantly excited in wells. Since we were not saturating the trap states in the barriers directly at the smallest well width the intensity dependence was the largest (Fig. A.1.7). The lifetimes were shorter for 345

nm pumping compared with 320 nm pumping due to the diffusion of carriers from barriers into wells on a longer time scale when pumped at 320 nm. As the carriers recombine in the wells they are restored from the barriers by diffusing from barriers to the wells.

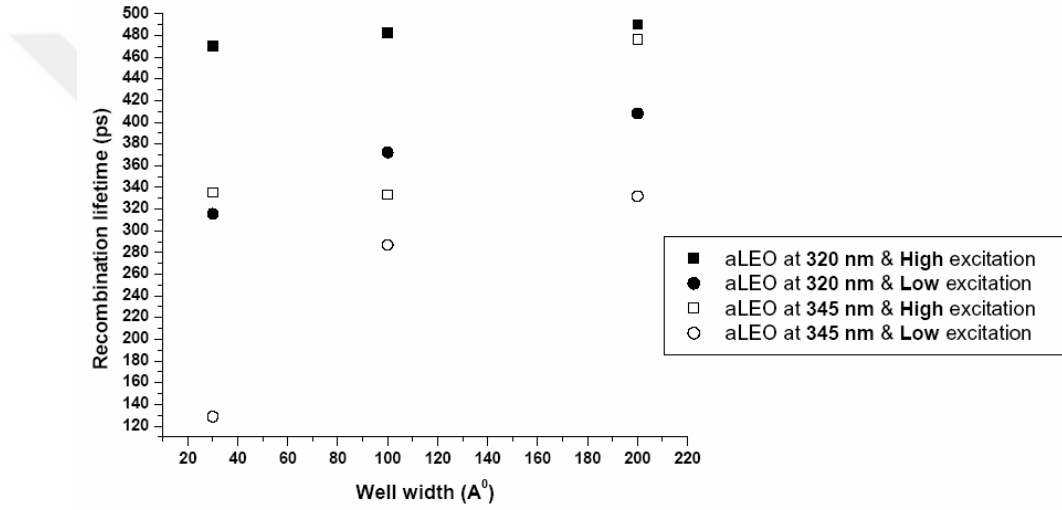


Figure A.1.7 Excitation wavelength dependence of TRPL decays in a-plane LEO MQWs as a function of well width for low and high excitation power density.

As shown in Fig. A.1.8 the variations of PL intensity as a function of well width at variable pump intensity can be associated more with the excitonic properties in the well than with increased non-radiative lifetimes.

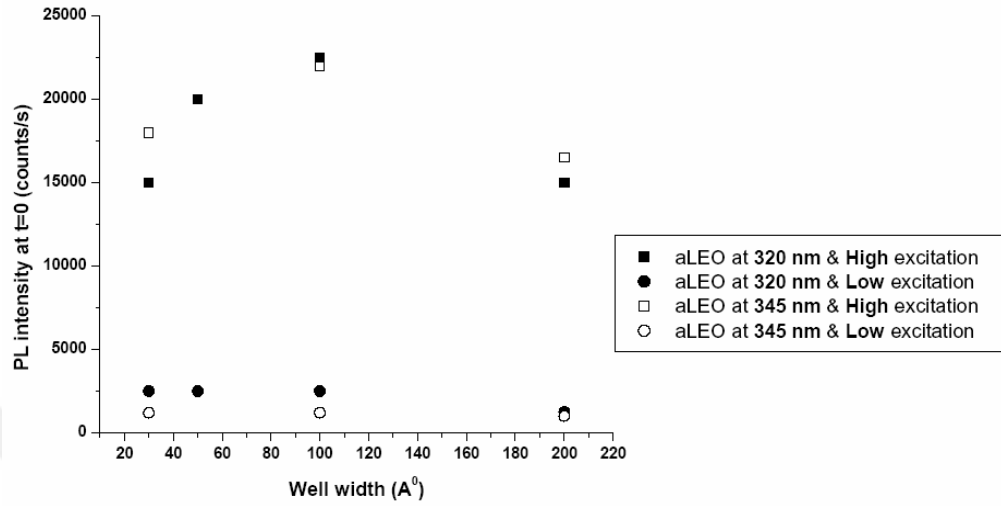


Figure A.1.8 Excitation wavelength dependence of PL intensity at $t=0$ in a-plane LEO MQWs as a function of well width for low and high excitation power density.

In order to see the effect of barrier width on intensity dependence of lifetimes the barriers were varied as 5, 8, 10, 12 and 15 nm for a constant well width of 5 nm. For above the barrier pumping (320 nm) the intensity dependence of a-plane LEO MQWs was similar for all widths except for 15 nm barriers in which case barriers exhibited less significant intensity dependence than expected because of the saturation effects in the wells which were associated with the diffusion of the excited carriers from barriers to wells at high intensities (Fig. A.1.8). Wider barriers have potentially more point defects with increased volume and so a larger number of nonradiative recombination sites, therefore at high pump intensities due to the carriers excited to barriers PL intensity declined with recombination of the carriers in the barriers. [5].

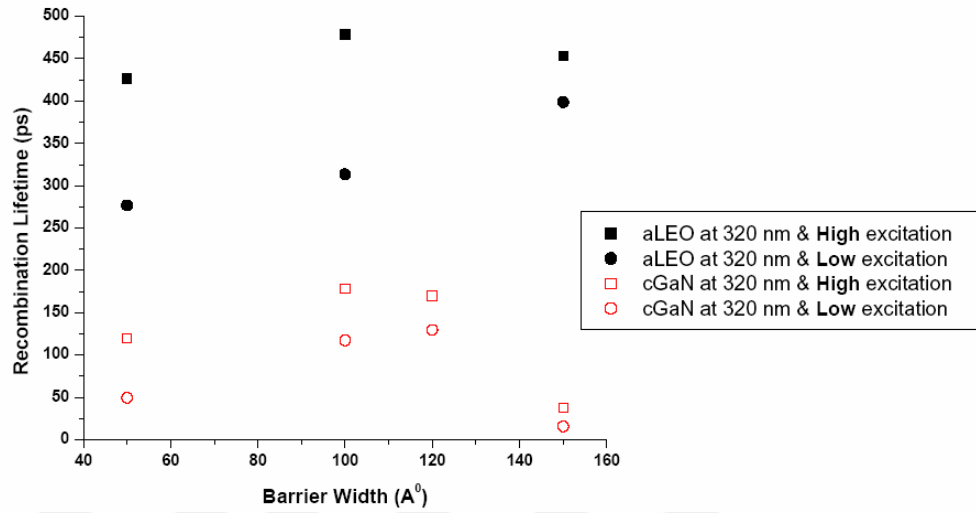


Figure A.1.9 Recombination lifetime in a-plane LEO and planar c-plane MQWs as a function of barrier width for low and high excitation power density.

A.4. Conclusion

Room temperature CW-PL and the time resolved optical studies were performed to determine the effects of polarization, defect densities, well and barrier widths, pulse wavelength, and excitation intensity on recombination dynamics.

Comparison of CW-PL emission from nonpolar planar and LEO a-plane MQWs showed that one to two orders of magnitude reduction in defect densities has a major impact on luminescence performances. With defect reduction it was possible to increase the PL intensities ~20x higher. For planar samples the highest intensity was observed at 5 nm while for LEO samples 3 nm wells exhibited the highest luminescence.

The PL lifetime of planar a-GaN was lower than planar c-GaN, and the LEO a-GaN was the highest. Distinct intensity dependence of the lifetimes for polar and nonpolar samples revealed that the source of recombination dynamics for these two samples were discrete. For polar MQWs at higher pump intensity radiative lifetimes increase with more effective screening while for nonpolar samples at higher pump intensity radiative lifetimes increase by phase space filling of the single particle states rather than nonradiative combinations. For the nonpolar a-plane LEO MQWs as the well width decreased PL intensity increased and lifetimes decreased due to enhanced quantum confinement with higher exciton binding energy and oscillator strength. Even with thick 20 nm wells, good wavefunction overlap without significant recombination through the barriers was achieved due to a lack of QCSE and flat band conduction. When the QWs were pumped at 345 nm below the barriers the pump intensity dependence decreased with increasing well width. Similarly when QWs were pumped at 320 nm above the barriers pump intensity dependency was reduced with increasing barrier width.

As a result reduced defect density nonpolar QWs are the ideal candidate for achieving high internal quantum efficiency devices with superior luminescent properties.

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Appendix B: SLEO PROCESSING FLOW

	Following process flow is base upon 5 micron mask openings and so minimum of 5 micron template thickness. In the case of 2 micron mask openings SiO ₂ deposition time, SiO ₂ etch time, and GaN etch time reduced with factor of 0.4-0.5.
1.0	Cleaning
	Soak in ACE and ISO (ultrasonic) for 4 min each
	Blow dry in nitrogen
	Bake dry @ 120° for >10 minutes
2.0	Deposit SiO₂ by PECVD
	Clean the chamber with no sample
	Evaporate 10,000Å SiO ₂ (x2) (by cooling off between two evaporation)
	Check the film thickness with filmetrics
3.0	Open Windows for SLEO Regrowth
	Bake @ 120° for >10 min
	Let HMDS sit on the surface
	Spin HMDS; 40s@6000rpm (x2)
	Spin photoresist AZ P4210; 45s@4000rpm
	Softbake; 95° for 1 min
	Expose for 18 s @ 7.5 W/cm ²
	Develop for in AZ 400K (1:4) for 50s
	Rinse in running DI for 30s
	Check in microscope
	Descum PEII chamber with no sample to clean
	Descum in PEII chamber (1min of O ₂ @ 300 mT, 100W)
4.0	SiO₂ Etch with ICP
	122 SiO ₂ cln et (dependin g on previous clean + 1 min coat)
	118 vertical SiO ₂ etch – over etch 10 min for 20,000 Å (etch rate ~2300 Å/min)
	121 O ₂ clean the chamber 20"

5.0	STRIPPING
	Soak in ACE and ISO (ultrasonic) for 4 min each (x2)
	N2 blow dry
6.0	GaN ETCHING with RIE#5
	Chamber clean
	-50sccm O2, 75 mT, 300 W, 10min
	-5sccm Cl2, 20sccm BCl3, 10 mT, 15 W, 3min
	-10sccm Cl2, 5 mT, 200 W, 2min
	Etch @ 10 sccm Cl2, 5mT, 200 W with He: 9.75 sccm cooling for 45 min (with etch rate ~1150 Å/s)
7.0	FINAL CLEANING
	Soak in ACE and ISO (ultrasonic) for 4 min each
	N2 blow dry

Appendix C: LED PROCESSING FLOW

	Regular LED Process
1.0	p-Activation
	ACE/ISO clean w/ ultrasonic 2 min each
	RTA 700C N2/O2 for 15 min.
2.0	p-Metal Contacts
	p-Metal Deposition (E-beam #3 or #4)
	-HCl:DI 1:3 dip for 60s, DI rinse for 60s
	-dry with N2 gun
	-deposit Pd/Au 50/50A
	p-Metal Photolithography
	-spin HMDS 40s @ 4k rpm
	-spin 4210 40s @ 4k rpm
	-soft bake 1 min @ 95C
	-expose for 18s @ 7.5 W/cm2
	-develop in AZ 400K (1:4) for 50s
	-rinse in DI for 45s
	-dry with N2 gun
	-inspect, develop more if necessary
	-30s O2 descum at 300mT/100W
	p-Metal Etch
	-dip in Au etch (approx. 5 - 10s)
	-rinse in DI for ~30 seconds
	-inspect, etch more if necessary
	-strip PR (ACE/ISO)
	-dehydration bake 5 min @ 120C
3.0	Mesa Formation
	Mesa Mask Photolithography (OPTIONAL)
	-spin HMDS 40s @ 4k rpm
	-spin 4210 40s @ 4k rpm
	-edge bead removal
	-soft bake 1 min @ 95C
	-expose for 18s @ 7.5 W/cm2
	-develop in AZ 400K (1:4) for 50s
	-rinse in DI for 45s

	-dry with N2 gun
	-inspect, develop more if necessary
	-hard bake 1 min @ 110C
	-15s O2 descum 300mT/100W
	Mesa Etch (RIE #5)
	-optional chamber clean (ONLY carrier wafer)
	-50sccm O2, 75 mT, 300 W, 10min
	-5sccm Cl2, 20sccm BCl3, 10 mT, 15 W, 3min
	-10sccm Cl2, 5 mT, 200 W, 2min
	-load sample onto carrier wafer
	-10sccm Cl2, 5 mT, 200W (etch rate ~1150A/m)
	-strip PR in ACE/ISO w/ ultrasonic for 4 min each (x2)
	-check etch depth with dektak
4.0	p-Contact Anneal
	p-Metal Anneal (RTA)
	-500C N2/O2 for 3 min
5.0	n-Metal Contacts
	n-Metal Mask Photolithography
	-spin HMDS 30s @ 3k rpm
	-spin AZnLOF2020 30s @ 3k rpm
	-edge bead removal
	-pre-exposure bake 90s @ 110C
	-expose for 10s @ 7.5 W/cm2
	-post-exposure bake 60s @ 110C
	-develop in AZ300-MIF for 60s
	-rinse in DI for 45s
	-dry with N2 gun
	-inspect, develop more if necessary
	-15s O2 descum 300mT/100W
	n-Metal Deposition (E-beam #3 or #4)
	-HCl:DI 1:3 dip for 60s, DI rinse for 60s
	-dry with N2 gun
	-deposit Ti/Al/Au 200/2000/3500A
	-liftoff in in 1165 at 80C
	- clean ACE/ISO w/ ultrasonic for 4 min each (x2)
	-dehydration bake 5 min @ 120C

6.0	Pad Metal
	Pad Metal Mask Photolithography
	-spin HMDS 30s @ 3k rpm
	-spin AZnLOF2020 30s @ 3k rpm
	-edge bead removal
	-pre-exposure bake 90s @ 110C
	-expose for 10s @ 7.5 W/cm ²
	-post-exposure bake 60s @ 110C
	-develop in AZ300-MIF for 60s
	-rinse in DI for 45s
	-dry with N ₂ gun
	-inspect, develop more if necessary
	-15s O ₂ descum 300mT/100W
	Pad Metal Deposition (E-beam #3 or #4)
	-deposit Ti/Au 200/3000A
	-liftoff in ACE/ISO w/ ultrasonic for 4 min each (x2)

Appendix D: HALL AND TLM PATTERNS PROCESSING

	P-DOPED SAMPLES
1.0	p-Activation
	ACE/ISO clean w/ ultrasonic 2 min each
	RTA 700C N2/O2 for 15 min.
2.0	p-Metal Contacts
	p-Metal Deposition (E-beam #3 or #4)
	-HCl:DI 1:3 dip for 60s, DI rinse for 60s
	-dry with N2 gun
	-deposit Pd/Au 200/2000Å
	p-Metal Photolithography
	-spin HMDS 30s @ 5k rpm
	-spin 4210 30s @ 5k rpm
	-soft bake 1 min @ 95C
	-expose for 20s @ 7.5 W/cm ²
	-develop in AZ 400K (1:4) for 50s
	-rinse in DI for 45s
	-dry with N2 gun
	-inspect, develop more if necessary
	-30s O2 descum at 300mT/100W
	p-Metal Etch
	-dip in Au etch (approx. 20-30s)
	-rinse in DI for ~30 seconds
	-inspect, etch more if necessary
	-strip PR and clean in ACE/ISO w/ ultrasonic 4 min each (x2)
3.0	Mesa Formation
	Mesa Mask Photolithography
	-spin HMDS 30s @ 5k rpm
	-spin 4210 30s @ 5k rpm
	-edge bead removal
	-soft bake 1 min @ 95C
	-expose for 20 s @ 7.5 W/cm ²
	-develop in AZ 400K (1:4) for 50s
	-rinse in DI for 45s
	-dry with N2 gun

	-inspect, develop more if necessary
	-30s O2 descum 300mT/100W
	Mesa Etch (RIE #5)
	-optional chamber clean (ONLY carrier wafer)
	-50sccm O2, 75 mT, 300 W, 10min
	-5sccm Cl2, 20sccm BCl3, 10 mT, 15 W, 3min
	-10sccm Cl2, 5 mT, 200 W, 2min
	-load sample onto carrier wafer
	-10sccm Cl2, 5 mT, 200W (etch rate ~1150A/m)
	-strip PR and clean in ACE/ISO w/ ultrasonic for 4 min each (x2)
	-check etch depth with dektak

	N-DOPED SAMPLES
1.0	n-Metal Contacts
	ACE/ISO clean w/ ultrasonic 2 min each
	n-Metal Mask Photolithography
	-spin HMDS 30s @ 3k rpm
	-spin AZnLOF2020 30s @ 3k rpm
	-edge bead removal
	-pre-exposure bake 90s @ 110C
	-expose for 10s @ 7.5 W/cm2
	-post-exposure bake 60s @ 110C
	-develop in AZ300-MIF for 60s
	-rinse in DI for 45s
	-dry with N2 gun
	-inspect, develop more if necessary
	-15s O2 descum 300mT/100W
	n-Metal Deposition (E-beam #3 or #4)
	-HCl:DI 1:3 dip for 60s, DI rinse for 60s
	-dry with N2 gun
	-deposit Al/Au 300/3000 A
	-liftoff in 1165 at 80C
	- clean ACE/ISO w/ ultrasonic for 4 min each (x2)
	-dehydration bake 5 min @ 120C

2.0	Mesa Formation
	Mesa Mask Photolithography
	-spin HMDS 30s @ 5k rpm
	-spin 4210 30s @ 5k rpm
	-edge bead removal
	-soft bake 1 min @ 95C
	-expose for 20 s @ 7.5 W/cm ²
	-develop in AZ 400K (1:4) for 50s
	-rinse in DI for 45s
	-dry with N ₂ gun
	-inspect, develop more if necessary
	-30s O ₂ descum 300mT/100W
	Mesa Etch (RIE #5)
	-optional chamber clean (ONLY carrier wafer)
	-50sccm O ₂ , 75 mT, 300 W, 10min
	-5sccm Cl ₂ , 20sccm BCl ₃ , 10 mT, 15 W, 3min
	-10sccm Cl ₂ , 5 mT, 200 W, 2min
	-load sample onto carrier wafer
	-10sccm Cl ₂ , 5 mT, 200W (etch rate ~1150Å/m)
	-strip PR and clean in ACE/ISO w/ ultrasonic for 4 min each (x2)
	-check etch depth with dektak