PROTON RADIATION AND THERMAL STABILITY
OF GALLIUM NITRIDE AND GALLIUM NITRIDE DEVICES

By

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by

Kimberly K. Allums
This document is dedicated to my Family and Friends. Thank you for your continued support of my endeavors and most of all me.
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By

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Chair: Cammy R. Abernathy
Major Department: Materials Science and Engineering

In today’s industry one can see a constant challenge to exceed the limits of
yesterday’s devices. For the last three decades, the III–V nitride semiconductors have
been viewed as highly promising for semiconductor device applications. The primary
focus of III–V nitrides, thus far, has been centered on light emitting diodes (LEDs),
injection lasers for digital data reading and storage applications, and ultra violet
photodetectors. Yet, another application is high-power electronic devices for space-borne
communications systems. It is expected that GaN-based devices will be more resistant to
radiation damage often encountered in space environments, though verification of this is
just now being undertaken. In particular, no information is yet available about the
sensitivity to radiation of devices using dielectrics such as MOSFETs. Similarly, very
limited data has been reported on the effects of high-energy protons on GaN based
devices of any type. For this reason the research presented in this dissertation was
undertaken to study the radiation and thermal stability of gallium nitride materials and
gallium nitride semiconductor diodes, with and without novel gate dielectrics such as, scandium oxide (Sc$_2$O$_3$) and magnesium oxide (MgO) and the ternary mix of magnesium calcium oxide (MgCaO).

It was found that though environmental degradation could be a problem for MgO dielectrics, the radiation exposure itself did not produce significant damage in either the Sc$_2$O$_3$, MgO or MgCaO dielectrics. Much of the minimal damage occurred in the GaN as shown by photoluminescence spectroscopy (PL).
CHAPTER 1
INTRODUCTION

In today’s industry, microelectronics has been largely based on silicon solid-state devices. As technology has improved, the demand for devices that can operate at higher temperatures and in more caustic environments has become the focus of several research areas especially military applications. As a result, compound semiconductors are becoming increasingly important because they do not possess the limitations of Si-devices and have direct bandgaps that allow them to be used in optical applications. Much of the focus of compound semiconductors has been on the III-V groups. Compound semiconductors, such as gallium arsenide (GaAs) and indium phosphide (InP), have higher carrier mobilities and lower-saturation electric fields than silicon semiconductor devices, and research based on these compound semiconductors have led to breakthroughs in device performance. For the last few decades the III-V nitrides, such as gallium nitride (GaN), have been viewed as highly promising for semiconductor device applications mainly because of a wider bandgap that will allow the semiconductor to overcome thermal and power handling limits of the GaAs and InP semiconductors. The primary focus of GaN applications thus far has centered on light emitting diodes (LEDs), injection lasers for digital data storage and ultraviolet photodetectors. Because of its large bandgap, $E_g$, GaN can operate at higher temperatures than its other semiconductor counterparts. However, the large $E_g$ also requires a large bandgap oxide in order to provide adequate carrier confinement in MOS-type applications. Various oxides were tested in previous studies but have been proven to possess many limitations as gate
dielectrics. Some alternative materials that are candidates for an optimum dielectric for GaN are magnesium oxide (MgO), and a ternary combination of magnesium calcium oxide (MgCaO). For use in wide bandgap semiconductor devices, the dielectric materials must also possess excellent thermal stability, both because of the high operating temperatures and the high processing temperatures needed for device fabrication. These oxides must also be radiation resistant for use in low orbit and aerospace applications. The ultimate goal of this project is to provide a high quality oxide that can improve the radiation hardness and thermal stability of GaN devices.
CHAPTER 2
BACKGROUND AND LITERATURE REVIEW

2.1 Introduction

This chapter discusses the fundamentals of field effect transistors (FET), metal oxide semiconductor capacitors, growth and characterization of gallium nitride, dielectric films and their properties, oxide growth processes and device applications. Also included is a literature review of the possible effects of radiation and the characterization methods used to determine the stability of the heterostructures in question.

2.2 Device Structures

A field effect transistor (FET) is a unipolar device where only one type of carrier takes part in the conduction process. The FET is a three-terminal device in which the current through two terminals is controlled by a voltage applied at the third. FETs are characterized by high input impedance since the control voltage is applied to a reverse biased junction either through a metal Schottky barrier or across an insulator [1]. Some advantages of FETs include higher switching speeds and higher cutoff frequencies than bipolar devices. There are various types of FETs such as junction field effect transistors (JFET) and metal semiconductor field effect transistors (MESFET), but the focus of this study will be metal oxide semiconductor field effect transistors (MOSFET) and metal oxide semiconductor (MOS) capacitors.

2.2.1 Metal Oxide Semiconductor Field Effect Transistor

The MOSFET is one of the most important devices for very large-scale integrated circuits such as microprocessors, and semiconductor memories and is also becoming an
important power device [2]. Similar devices to the MOSFET include the metal insulator semiconductor field effect transistor (MISFET) and the insulated gate field effect transistor (IGFET). For all of these device types, the gate region of the transistor determines the capabilities of the device. The uniqueness of the MOSFET relies on the oxide layer’s ability to prevent current flow from the semiconductor to the gate due to the high resistivity of the oxide. Figure 2-1 shows a general structure of a MOSFET. Ideally a MOSFET should possess high output current drive, $I_D$, high transconductance, $g_m$, stable threshold voltage, $V_T$, fast switching speed, high gate oxide breakdown voltage and low source/drain to body capacitance. Physically, the current drive is linearly related to gate width and as $I_D$ increases so does the capacitance.

There are two types of MOSFET devices, depletion mode and enhancement mode. In the depletion mode device, the material under the gate is doped in order to carry current. This device is in the “ON” state when there is no applied gate voltage. When there is a zero gate voltage, carriers are free to flow from the source to the drain in the MOSFET structure. As a negative voltage is applied to the gate contact, the area under the gate, called the channel, is gradually depleted of carriers. The depletion depth is increased until the flow from source to drain is stopped. This voltage is called the pinch off voltage, since it effectively pinches the channel shut. The transistor is now “OFF.” As the voltage across the source-drain is increased, it requires more gate voltage to successfully pinch-off the carrier flow. In the enhancement mode device, the material type under the gate is not doped; thus no channel is present and in an OFF state since there is no current flow. As with the depletion mode MOSFET, a positive drain voltage is still applied but there is also a positive gate voltage applied as well. This has the effect of
attracting free electrons towards the gate, thus creating a channel of free flowing electrons and the larger the positive gate voltage the wider the carrier channel of electrons. Figure 2-2 shows an example of the ON and OFF states of the enhancement mode device. Thus the maximum operating parameters of the device are determined by the amount of electric field that can be applied to the gate before dielectric breakdown occurs. Also, forward gate voltages can be used to increase the amount of current, which can be passed through the channel. Again a high dielectric breakdown field is required.

The MOSFET has several advantages over heterojunction type transistors, such as relative insensitivity to temperature during operation. Also MOSFET devices are expected to have a wide gate modulation range. This wide range is because the device turn-on is dependent on the dielectric thickness and is not limited to low turn-on voltages like those obtained when using Schottky metal contacts.

2.2.2 Metal Oxide Semiconductor Capacitors

The MOS capacitor (MOSC) is the most basic device used to evaluate the electrical properties of the oxide and the semiconductor. Throughout this study the MOS capacitor, also known as the MOS diode, is used for evaluation of the MOS structure. For use with GaN on sapphire substrate, the contacts are created front side. The process is done by etching the oxide to expose the underlying semiconductor material and then depositing ohmic and gate contacts. Figure 2-3 shows a cross section of the MOSC. The MOSC is operated by applying a potential difference across the gate and ohmic contacts. An understanding of MOSC behavior is obtained through the use of energy band diagrams and ideal capacitance-voltage curves.
2.3 Growth and Photoluminescence Spectroscopy of Gallium Nitride

One of the ways high quality gallium nitride is grown is via metal organic chemical vapor deposition (MOCVD). Trimethlgallium (TMG) and ammonia (NH₃) are used as source gases for Group III and V species to obtain c-axis oriented films of GaN grown on (0001) sapphire (Al₂O₃) wafers. In MOCVD the flow of the reactant gas sources is very critical to the GaN film growth and any slight change can alter the film quality. Hydrogen is normally used as a carrier gas and is flowed normal to the substrate surface to bring the reactant gases in contact with the substrate and to prevent thermal convection effects. MOCVD can be used to grow n-type and p-type GaN. Undoped GaN is naturally n-type due to nitrogen vacancies that occur in the film. Two types of GaN are generally mentioned when referring to n-type GaN, doped and unintentially doped. Silicon is normally used to doped GaN to increase the number of electron carriers within the substrate and the carrier concentration is typically $\sim 10^{19} \text{cm}^{-3}$ whereas unintentially doped GaN (known as u-GaN) has a typical intrinsic electron carrier concentration of $\sim 10^{16} \text{cm}^{-3}$. As well as being n-type, MOCVD GaN is normally grown with N terminated faces at the surface which tend to have dangling bonds that allow for the surface to be susceptible to contamination and impurities. This can lead to surface effects such as surface charging and fermi level pinning. P- type GaN is made by doping the GaN with magnesium, Mg, to create majority carriers of holes.

Gallium nitride can also be grown via molecular beam epitaxy (MBE) with either N terminated ([0001] direction) or Ga terminated ([000-1] direction) surfaces using sapphire substrates. MBE’s inherent control over growth parameters can be used to interrogate certain structural and electrical processes in the crystal. It has been successfully shown that GaN can be grown with Ga terminated surfaces (known as Ga-polar GaN), which
results in a smoother surface morphology and better quality films. The successful growth of Ga-polar GaN via MBE has a very narrow margin of error when it comes to the exact growth parameters and to assist with the correct termination many growths of Ga-polar GaN start with a MOCVD GaN template, which has the right orientation for continued growth of the GaN film by MBE.

Defects in GaN degrade device performance and longevity. Therefore it is important to have an understanding of the types of defects present and the effects they produce in GaN. Native defects in GaN tend to be Ga or N vacancies, impurities, dislocations and/or interstitial atoms. One non-destructive characterization used to determine the types of defects and their effects is photoluminescence spectroscopy (PL). These defects cause different energy or color bands in the PL spectra. By monitoring the intensity changes and wavelength peak shifts in these bands, it is possible to identify the types of defects that are present. Knowing this information concerning the defects can allow for treatments pre or post GaN growth to reduce the defects. Common color bands that show up in GaN are the ultraviolet band (UVL) at approximately 390-455nm (3.076eV - 3.257eV), blue band (BL) at approximately at 438 to 442nm (2.88eV – 2.90eV), green band (GL) at 492 to 577nm (2.5eV – 2.2eV), the much debated yellow band (YL) at 577 – 597nm (2.2eV – 2.3eV), and a red band (RL) at 688nm – 652nm (1.8eV – 1.9eV).

Each of these bands corresponds to an energy transition within the band gap of gallium nitride and depending on the energy or band associated with the transition, pertains to a particular type of defect within the material. The ultraviolet band (UVL) has shown up in undoped GaN as well as Mg-doped GaN. In u-GaN the main candidates for
shallow donors are SiGa and O_N. The assignment of shallow acceptor has been identified using optical detected magnetic-resonance (ODMR) as Si_N and in Mg-doped GaN, the shallow acceptors are MgGa and Si_N. The UVL bands in each type of GaN are very similar to each other indicating the different shallow acceptors manifest themselves similarly in GaN as a transition from a shallow donor to a shallow acceptor.

The blue band that occurs approximately 420nm to 490nm is observed in undoped, and Mg-doped GaN but only those grown via MOCVD or HVPE because the defect is not a native defect that is grown into the GaN film. The BL band has been defined in u-GaN as a transition from a shallow donor to deep acceptors at low temperatures and at elevated temperatures from conduction band to deep acceptor. In p-GaN the transition was described as a transition from deep donor to the shallow Mg_Ga acceptor. Quenching of the BL band occurs when holes from the acceptor level escape to the valence band and an observation of the BL band at low temperatures such as 15K enables the determination of the vibrational characteristics of the defects in its ground state.

The green band in GaN is usually coupled with the YL band and sometimes overshadowed by the intensity of the YL band. However, in high purity GaN a yellow-green band can be observed at room temperature. The shape and position of the band depends on the excitation intensity and excitation energy and can always be deconvolved into two bands. It is presumed that the YL and GL band are related to two charge states of the same defect, presumably the V_GaO_N complex.

Many researchers have disputed the attribution of the YL band in GaN and are still undecided as to the exact origins of the YL band. Although they have agreed that the YL band is not specific to a particular impurity defect, they have instead related the YL band
to a native defect that is grown into the gallium nitride and is specific to a type of transition with the band gap. It is also believed that in undoped GaN the $V_{Ga}O_N$ complex is responsible for the YL band and that $O_N$ and $C_{Ga}$ are shallow donors. It is still debated whether or not the YL band is a transition from the conduction band or shallow donor to a deep acceptor or as one researcher suggested [hoffman], a two stage process that involves a non-radiative capture of an electron by a deep double donor followed by a radiative recombination between the electron at the deep donor and a hole at a shallow acceptor. Whichever transition is responsible, thus far no model fully describes the defect or defects responsible for the YL band. What has been found about the YL band is that carbon doping and Si doping to a certain concentration enhance the YL emission. It is believed that the carbon and silicon sit upon Ga vacancies. For Si, the initial enhancement comes from inadvertent increase in the concentration of the $V_{Ga}$–related acceptors such as $V_{Ga}O_N$ and $V_{Ga}Si_{Ga}$ facilitated by the shift of the Fermi level to higher energies during growth. The activation energy of the $Si_{Ga}$ donor is 30meV. Although carbon does enhance the YL emission, the likelihood of a $C_{Ga}$ formation is very low.

The red band in u-GaN is the least studied and the least common of all the color bands that appear. The intensities are independent of temperature, saturate at low excitation, and possess long lifetimes. The transition is known to be shallow donor or conduction band to a deep acceptor at low temperatures and elevated temperatures respectively.

In Ga-polar GaN two color bands dominate within the low temperature PL spectra, a red band (RL2) and a green band (GL2). Both tend to have very weak emission and quench above 100K. These bands are not to be confused with the other RL and GL bands
mentioned. The bands are created by point defects that are fairly uniform throughout the bulk GaN and tend to dominate high-resistivity GaN \((n_0=10^{15} \text{ cm}^{-3})\). Due to internal transitions in the defects, the Gaussian shape of the bands indicate that in both cases the carrier is strongly localized at the defect providing a strong electron-phonon coupling.

In summary, determining the color spectra of PL is a very useful way to thoroughly characterize all types of gallium nitride films and to determine what defects are grown in and what defects are induced due to doping, impurities, and irradiation.

### 2.4 Dielectrics

#### 2.4.1 Ideal Dielectrics

Ideal dielectric materials are perfect insulators in which no mobile charged particles are present. The ability to store an electric charge is called capacitance [3]. When an insulator is placed between the conducting plates of a capacitor, the capacitance can increase significantly. Insulators have positive and negative charges in the form of the atom nucleus and the electron cloud, but these charges are bound to the atom or molecule and are not available for conduction [4]. Under the influence of an electric field, the nucleus and the electron cloud are displaced to a form a dipole [1]. Figure 2-4 shows the dipole formation in the presence of an electric field. The total effect of an electric field on a dielectric material is known as polarization. The ability of the material to resist the polarization of charge is described as the dielectric constant, \(\kappa\), which is the ratio of the permittivity of the material, \(\varepsilon_i\), to the permittivity of vacuum, \(\varepsilon_v\).

\[
\kappa = \frac{\varepsilon_i}{\varepsilon_v}
\]  

The dielectric constant can also be related to the internal field created within the material and the external field applied, through Equation 2-2 [5].
\[ E_{\text{internal}} = \frac{E_{\text{applied}}}{\kappa} \]  

(2-2)

The polarization, \( P \), of the material is related to the dielectric constant by equation \( 2-3 \), where \( \xi \) is the strength of the electric field (V/m). It can be assumed from this relation that the polarization increases as the electric field strength increases, until all the dipoles are aligned such that

\[ P = (\kappa - 1)\varepsilon_0 \xi \]  

(2-3)

There are various applications for dielectric materials. Passivation of high voltage junctions, isolation of devices and interconnects and gate insulation of field effect transistors are a few that are relevant for this discussion. For a material to be a successful dielectric it must meet certain criteria. Desirable characteristics include chemical stability over the life of the device, immobile charge traps (to avoid shorting and frequency limits) and a dielectric constant higher than that of the semiconductor (to avoid generating a high electric field in the dielectric). The dielectric/semiconductor interface is also an important focus of research in the area of device processing. The interface state density of carrier traps must be \(<\text{mid-}10^{11} \text{ eV}^{-1}\text{cm}^{-2} \) for a device to be considered successful. A good dielectric should possess low defect density, good barrier properties against impurity diffusion, high-quality interface with low state density and fixed charge and stability under irradiation. For use in wide bandgap semiconductor devices, the dielectric materials must also possess excellent thermal stability, since use at high operating temperatures is very desirable.

**2.4.2 Crystalline vs. Amorphous**

There are several reasons that both crystalline and amorphous dielectric layers are being investigated. The major concerns with MOS-type semiconductor devices are the
interface between the dielectric and substrate and current leakage in the oxide layer. Ideally the perfect dielectric would be a single crystal structure with the same symmetry and no lattice mismatch to GaN. The absence of lattice mismatch eliminates stress and defects at the interface. Realistically, using a dielectric material with small lattice mismatch to the substrate should reduce high defect densities at the interface. However, in some cases it may not be possible to eliminate a sufficient number of crystalline defects; thus the possibilities of using amorphous oxides will also be discussed.

Using a single crystal layer of the prospect oxides should in principle provide a high quality layer. However, even the small lattice mismatch of the two prospect oxides can still cause defects in the interface. These defects can propagate through the entire oxide creating traps or leakage paths. One problem is finding the right growth conditions that will produce a single-crystal and not a poly-crystalline structure. Polycrystalline layers at the interface could result in grain boundaries allowing high leakage current through the dielectric. In an amorphous arrangement the atoms are randomly distributed on the substrate. This eliminates the problem of strain and dislocations at the interface and in the dielectric. A truly amorphous layer would also stop any shortening of current through the oxide, because the electrons or holes would have no defect paths to follow. One potential problem is the possibility for formation of crystallites in the layer. At high operating temperatures, the crystallites may grow and coalesce to form grain boundaries and a polycrystalline-like structure, resulting in a very leaky and thermally unstable dielectric. Each type of oxide layer has advantages and disadvantages.

2.4.3 Present State of Dielectrics for Gallium Nitride

Several dielectrics have been tested for use on wide bandgap GaN semiconductor devices (Table 2-1). Gallium nitride research has resulted in long-lifetime, room
temperature operation of photonic devices. These include LEDs that cover the visible spectrum, laser diodes in the blue and blue-green regime, and UV detectors. These devices are just recently reaching production with problems still to be solved in the fields of n-Ohmic and p-Ohmic contacts, p-type doping issues, Schottky contacts, and dielectric materials. Also, with the lack of availability of high-quality GaN substrates, research an epitaxial growth and substrate selection is still ongoing. From material and processing advances learned from the photonics research, high-power and high-temperature switches have been realized. The following summarizes some of the dielectrics research to date.

2.4.3.1 Silicon oxide on GaN

Silicon oxide is a very attractive choice for a dielectric material since it is has been well studied and the processing is well established. Silicon oxide deposited by plasma enhanced chemical vapor deposition (PECVD) [6-9] has been reported to give interface state densities on the order of low 10^{11} \text{eV}^{-1} \text{cm}^{-2}. Silicon oxide deposited by electron beam (EB) evaporation on GaN has shown an interface state density of 5.3 \times 10^{11} \text{eV}^{-1} \text{cm}^{-2} [8]. After annealing the EB deposited SiO_x at 650°C, the valance band offset was measured to be 2\text{eV} and the conduction band offset was measured to be 3.6 \text{eV} [10]. The EB evaporated SiO_x shows a silicon rich stoichiometry when compared to the PECVD SiO_x.

There are several inherent problems with SiO_x as a dielectric material for wide bandgap semiconductors. The high D_{it} of SiO_x is due to uncontrolled oxidation of the surface [11]. The most significant limitation is that SiO_x has a dielectric constant (\varepsilon) of 3.9, which is significantly lower than that of GaN (\varepsilon = 8.9). This will create a very large
electric field in the dielectric, leading to further breakdown and increased power consumption.

2.4.3.2 Silicon nitride on GaN

The $D_{it}$ obtained from PECVD silicon nitride [8,9] has been reported as $6.5 \times 10^{11}$ eV$^{-1}$cm$^{-2}$. The Si$_3$N$_4$ MISFET, however, shows poor performance. Electrical measurements showed the MISFET structure had a large flat band voltage shift (3.07 V) and a low breakdown voltage (1.5 MV/cm). There was no microstructural analysis performed on the deposited Si$_3$N$_4$ films. SiN deposited by ECR plasma CVD showed a $D_{it}$ of $1 \times 10^{11}$cm$^{-2}$eV$^{-1}$ but had excess leakage current due to the small conduction band offset [11]. The ECR-CVD MIS diode showed a $D_{it}$ of $4 \times 10^{11}$ cm$^{-2}$eV$^{-1}$, fixed oxide charge of $1.1 \times 10^{11}$ cm$^{-2}$ and breakdown 5.7 MV/cm with a dielectric constant of 6 [12].

A unique dielectric structure of SiO$_2$/Si$_3$N$_4$/SiO$_2$ (ONO) was reported to have breakdown field strength of 12.5 MV/cm for temperatures as high as 300°C. The ONO structure was deposited by jet vapor deposition to a thickness of 10 nm /20 nm /10 nm. The multiplayer structure does allow for unique engineering of a dielectric, but multiple interfaces can lead to an extremely large number of interface state traps and increased processing. The $D_{it}$ for the ONO structure was reported to be less than $5 \times 10^{10}$ eV$^{-1}$cm$^{-2}$ with breakdown fields greater than 12 MV/cm [13].

2.4.3.3 Aluminum nitride on GaN

Aluminum nitride deposited by MBE and MOCVD has been used to create MISFET devices and insulated gate heterostructure field effect transistor (IG-HFET) devices [14,15]. The AlN MISFET structure grown at 400°C was polycrystalline. From x-ray reflectivity measurements, the AlN/GaN interface showed a roughness of 2.0 nm.
This may be due to the polycrystalline nature of the film or from intermixing of the AlN and GaN at the interface. The dielectric breakdown field was calculated to be 1.2 MV/cm. The AlN IG-HFET structure was grown at 990°C, forming a single crystal film of 4.0 nm. This device operated in depletion mode and had a pinch-off voltage of 0 V. Hexagonal aluminum nitride has a 2.4% lattice mismatch with hexagonal GaN on the (0001) plane. The 4.0 nm film thickness is greater than the critical thickness allowed for elastic deformation leading to threading dislocations forming from plastic deformation. Single crystal AlN and polycrystalline AlN films suffer from defects and grain boundaries that cause shorting.

### 2.4.3.4 Gallium oxide on GaN

Like Si, gallium nitride forms a native oxide. This oxide has been considered as a dielectric material, like the native oxide of silicon. Thermal oxidation has been studied using dry [16,17] and wet [18] atmospheres. Dry oxidation of GaN epilayers at temperatures below 900°C showed minimal oxidation. Dry oxidation at 880°C for 5 hours produced 1110 nm of β-Ga$_2$O$_3$ with a $D_{it}$ of $1 \times 10^{10}$ eV$^{-1}$cm$^{-2}$ and showed inversion [19]. At temperatures above 900°C, a polycrystalline monoclinic Ga$_2$O$_3$ formed at a rate of 5.0 nm/hr. This oxidation rate is too slow to be viable as a processing step. Wet oxidation of GaN also forms polycrystalline monoclinic Ga$_2$O$_3$, but at a rate of 50.0 nm/hr at 900°C. From cross-sectional transmission electron microscopy, the interface between the oxide and the GaN was found to be non-uniform. Scanning electron microscopy showed that both films were rough and faceted. Electrical characterization of the oxide showed a dry oxide dielectric field strength of 0.2 MV/cm and a wet oxide dielectric field strength of 0.05 to 0.1 MV/cm. The microstructure formed from this
process was shown by XRD to be a high temperature hexagonal phase [20]. Ga$_2$O$_3$, formed by this method passivates the surface [20-23] and has a $D_n$ of $10^{11}$ eV$^{-1}$cm$^{-2}$ for GaN MOS. A negative oxide charge as well as high capacitance and reduced reverse leakage where shown for thicker oxides grown by PEC [21]. Using PEC and a HeCd laser, a low reverse leakage current of 200 pA at 20Vm was achieved. For this oxide the forward breakdown, $E_{fb}$, was 2.8 MV/cm and the reverse breakdown, $E_{rb}$, was 5.7 MV/cm with a $D_n$ of $2.53 \times 10^{11}$ cm$^{-2}$eV$^{-1}$. The dielectric constant of Ga$_2$O$_3$ grown under these conditions was estimated to be 10.6 [24]. With amorphous GaO deposited by PEC, low leakage currents of $<10 \times 10^{-8}$ Å/cm at −15 V have been measured. Using Ga$_2$O$_3$ as both gate dielectric and passivation layer, a breakdown field of 0.4 MV/cm was observed. The bandgap, $E_g$, of Ga$_2$O$_3$ was measured to be 4.4 eV [25].

2.4.3.5 Silicon dioxide on gallium oxide on GaN

Depending on the growth technique, the interface between the SiO and the GaN can vary drastically. When SiO is deposited by RPECVD, a parasitic subcutaneous layer of native gallium oxide is grown on the GaN surface. This layer has been shown to have a direct effect on the device performance. When the thickness of the initial GaO layer is controlled by a pre-oxidation step the device characteristics improve markedly. Remote Plasma Assisted Oxidation first followed by RPECVD gave a lower $D_n$ and a smaller flat band shift over the RPECVD of the SiO$_x$ alone [26]. Real and ideal CV curves are nearly identical [27]. After an anneal in an RTA for 1 min at 900°C in Ar the $D_n$ is $2-3 \times 10^{11}$ cm$^{-2}$ [28,29]. Another group used a similar oxide growth technique and measured a $D_n$ of $3.9 \times 10^{10}$ eV$^{-1}$cm$^{-2}$ and a low leakage current [30, 31].
2.4.3.6 Gallium gadolinium oxide on GaN

Due to the recent success of gallium gadolinium oxide (GGG) as a dielectric in GaAs MOSFETs [32-37], attention has turned toward this as a dielectric material for GaN. The GGG dielectric was deposited on a GaN epilayer by EB evaporation of a single crystal GGG source [14]. The substrate temperature was 550°C. The interface roughness was calculated to be 0.3 nm from x-ray reflectivity. Metal oxide semiconductor (MOS) capacitors were formed and tested. A breakdown field of greater than 12 MV/cm was estimated. More recently, the thermal stability of the film and the interface has been proven to temperatures as high as 950°C and operation of a depletion mode MOSFET has been performed at temperatures up to 400°C [38]. The EB evaporated GGG stoichiometry is heavily dependant upon the substrate temperature. Changes in temperature lead to changes in the stoichiometry [39]. This limits the available microstructure obtainable within the stoichiometric limits of GGG.

2.4.3.7 Gadolinium oxide on GaN

GaN based MOSFETs have been made that used a stacked gate oxide consisting of single crystal gadolinium oxide and amorphous SiO₂ [40]. The gadolinium oxide provides a good oxide /semiconductor interface and the SiO₂ reduces the gate leakage current and enhances oxide breakdown voltages. The dislocations in the Gd₂O₃ film limit the breakdown field that can be sustained in the dielectric.

2.4.3.8 Scandium oxide on GaN

Scandium oxide grown by MBE has been used as a gate dielectric and passivation layer for GaN based devices. Scandium oxide has the bixbyite crystal structure, a reasonable band gap of 6.3 eV and a lattice mismatch to GaN of 9.2%. The scandium
oxide was grown by MBE using an RF oxygen plasma, substrate temperature of 650°C and an effusion cell temperature of 1130°C. This oxide showed inversion when used in a GaN gated diode [41]. This oxide was also grown under the same conditions except the substrate temperature was 100°C, which resulted in an interface state density of $5 \times 10^{11}$ eV$^{-1}$cm$^{-2}$ [42].

Scandium oxide has also been used as a field passivation layer for GaN devices. It has been shown to reduce the reverse leakage current and increase the $f_T$ and $f_{MAX}$. The passivation films have been grown with a substrate temperature of 100°C and a cell temperature of 1130°C [43]. Scandium oxide has better long-term stability than SiN$_x$ as a passivation film for GaN based HEMTs. It has been shown to dramatically reduce the gate lag problems due to surface states on AlGaN/GaN HEMTs [44].

2.5 Epitaxial Growth

The term “epitaxy” is Greek in origin, coming from the words “epi” meaning upon, and “taxis” meaning to arrange. Epitaxy is the process of controlled growth of a high-quality crystalline or amorphous layer of material on a substrate. There are two categories of growth for epitaxy: homoepitaxy and heteroepitaxy. Homoepitaxy is the growth of a crystalline layer that is the same as the substrate material while heteroepitaxy is the growth of a layer that differs from that of the substrate [1]. The growth method used in this study is Molecular Beam Epitaxy (MBE). It is important to select growth parameters that will produce the highest quality epitaxial layer for the GaN semiconductor, in order to reduce crystalline defects that would degrade the performance of the semiconductor device.
2.5.1 Molecular Beam Epitaxy

The growth of the epitaxial layer takes place due to reactions between the molecular beams coming from the sources and a crystalline surface held at suitable temperatures. The molecular beams are produced from solid element at sources heated in effusion cells. Growth begins when different atomic species are absorbed on the substrate surface and migrate to form the deposited layer. A common in-situ growth-monitoring technique is electron diffraction, commonly known as RHEED (Reflection High-Energy Electron Diffraction). In this method, high-energy electrons are diffracted off the growing surface and produce an image on a screen on the opposite side of the chamber [45] (Figure 2-5).

Although MBE has the capability for growing complex multilayers where precise control of dopant concentration, layer thickness and interface abruptness are required, there are also serious concerns. For example, due to the UHV environment sensitivity to contamination and replenishment of sources, long periods of system downtime can ensue.

2.5.2 Substrate Preparation

Before any epitaxial growth, the substrates received an ex-situ and an in-situ surface treatment to remove any contamination and the native oxide. The surface of the semiconductor must be as clean and planar as possible to ensure high quality dielectric film deposition. Surface contamination leads to impurities at the dielectric/semiconductor interface, which ultimately result in the formation of interface traps. Since gallium nitride substrates are not currently available, gallium nitride grown on sapphire wafers oriented <0001> were used. These will be referred to throughout this work as GaN substrates. Two different types of growth of the GaN substrates were employed in this work, MBE and metal-organic chemical vapor deposition (MOCVD). The MBE GaN substrates were grown in house (referred to as UF MBE GaN) and the
MOCVD substrates were either grown in house (referred to as UF GaN) or by Uniroyal Optoelectronics (UOE). From AFM, there is a large difference in surface roughness between the two types of GaN. The MOCVD substrates are 1-3 nm RMS roughness and the MBE substrates are ~6 nm RMS roughness (Figure 2-6).

The GaN substrates received an *ex-situ* treatment consisting of a 3-minute etch in (1:1) hydrochloric acid: water, followed by a DI rinse and blown dry by nitrogen. This was used to remove any organic residue from the surface. Then a 25-minute exposure to ozone produced by an ultraviolet lamp in a UVOCS UVO Cleaner model number 42-220 was used to oxidize the carbon on the surface and create a thin native oxide layer. Next, the substrates received another etch in buffered oxide etch for 5 minutes to remove the native oxide. This is shown by observing the RHEED pattern produced from the surface. The RHEED pattern produced by the native oxide is more diffuse than the pattern produced by the buffered oxide etched surface (Figure 2-7).

The GaN substrates were then mounted to molybdenum blocks using indium solder, and then loaded into the load-lock of the MBE. Room temperature RHEED measurements showed a reasonably clean (1x1) surface (Figure 2-7). Two different crystal directions are observed in RHEED to create a more complete understanding of the surface. Here, the <1-100> and the <11-20> directions are observed. An *in-situ* thermal treatment was employed to further remove any oxide or contamination left on the surface. The substrates were heated to 700°C in vacuum and no overpressure of nitrogen was used. The RHEED patterns recorded at this temperature indicate a sharp (1x3) pattern (Figure 2-8).
2.6 Types and Effects of Radiation on Microelectronics

In the past decades most radiation testing has been performed on Si devices. Currently there is very limited information on radiation effects on GaN devices. The objective is to investigate the stability of the oxide, the interface between the oxide and semiconductor, and the operational characteristics of the MOS capacitor.

In order to understand the relevant applications for radiation hard devices one must first understand the origins and effects of the space radiation and terrestrial radiation environment. There are various types of radiation that affect electronic devices such as gamma rays, x-rays, protons, neutrons and other subatomic radioactive particles. Much of this radiation comes from solar cosmic rays, galactic cosmic rays that originate outside the solar system, and other planetary environments. The earth’s magnetic field shields a region of near earth space from these particles but they easily reach polar regions and high altitudes such as geosynchronous orbit (35,800km) in which satellites travel [46,47].

Around the earth lies a radiation environment known as the Van Allen belts. These belts are compromised of trapped subatomic particles that can affect electronic devices operating at high-altitudes.

Gamma rays are electromagnetic radiation emitted by radioactive decay. These rays are shorter in wavelength than ultra violet and are far higher in energy. For experimental use, common sources of gamma rays are Cobalt 60 and Cesium 137. Effects of gamma rays tend to occur over long terms and can cause changes in electrical operational characteristics.

X-rays are created from high-speed electrons that after impinging on a metal release energy in the form of an electromagnetic wave. Unlike gamma rays, x-rays consist of a mixture of different wavelengths. In addition to originating from solar flares
and other cosmic occurrences, x-rays are also produced by nuclear explosions. After a nuclear explosion, much of the kinetic energy of the fission fragments is converted to internal excitation and radiation. Some 70-80% of the total energy of this material is emitted as thermal electromagnetic radiation in the soft x-ray region. This can produce ionizing effects on MOS devices. Radiation of this type can be found at satellite orbit altitudes as well.

Neutrons are also a concern with radiation environments. Neutrons are released following nuclear fission and can travel large distances even at sea-level and at atmospheric pressures. The neutrons can produce recoil protons by elastic collisions in hydrogen rich materials, therefore ionizing the material or electronics of interest.

Each of these types of radiation can damage or upset electronic devices but in comparison to the amount of protons in the environment they make up a small percentage. Generally, protons trapped in the earth’s magnetosphere have energies up to 800 MeV. They primarily originate from galactic cosmic rays, which come from outside the solar system. These protons are concentrated in a small area known as the South Atlantic Anomaly. The radiation belt dips into the earth’s atmosphere due to the earth’s tilt on its axis. This causes concern for electronic devices that will operate in or near the region. Hence the focus of this study, effects of proton radiation on gallium nitride devices. Proton radiation causes ionization in electronic devices. This involves creating electron-hole pairs, Frenkel defects, and bulk semiconductor defects [48]. Proton radiation can also cause traps to form at the interface of the oxide and substrate. These various radiation-induced defects can cause current leakage through the oxide, displacement or removal of semiconductor atoms, build up of trapped charge and shifts in
operational parameters of the electronic device. Previous studies on silicon devices have shown radiation resistance under the mentioned environments. Gallium nitride is expected to be even more radiation resistant or rad-hard than silicon. In the course of this study 40MeV\((\pm 2\%)\) and 10MeV \((\pm 10\%)\) energies of protons will be examined at 5\(\times\)10\(^9\) cm\(^2\) total particle dose. Although lower energy protons can be blocked by shielding, it is useful to use lower energy protons in order to look at the type of damage caused by radiation to the device or material. As technology improves, device features get smaller and use lower energy to operate. This can cause sensitivity to various impinging particles, which can damage the functionality of the device. The key to reducing the radiation sensitivity of the device may rely on finding an oxide layer that can remain virtually unaffected while protecting the underlying semiconductor material and interface.

In addition to permanent degradation of the device, an electrical device can also experience a number of single event effects (SEE) due to total dose ionization of the material or device. Soft errors are single event upsets (SEU). Events of this type can be corrected by reprogramming or resetting the device. This is usually apparent in memory circuits. It can also result in performance degradation if the error rate is too high. Hard errors, which are not correctible, include single event latchup (SEL), burnout (SEB), and gate rupture (SEGR). If a hard error occurs a circuit element can be physically damaged.

2.7 Previous Radiation Studies

In recent years, several studies have been done on radiation of various GaN devices. Radiation types have included gamma rays, electrons and low-energy protons.

Gamma irradiation at total fluences of 600 Mrads was studied by Luo et al. [49]. At this high dosage the AlGaN/GaN HEMT showed a 45% change in the
transconductance, $g_m$, for varied gate lengths and widths. At a lower dose of 300 Mrad, minimal changes in the electrical characteristics were observed.

Look et al. [50] conducted an electron radiation study. Energies ranging from 0.7 to 1 MeV were used to induce removal of nitrogen or gallium atoms. Fluences ranging from 1 to $7 \times 10^{16}$ cm$^{-2}$ were noted. They concluded that nitrogen Frenkel pair formation was occurring, resulting in the formation of shallow donors and shallow or deep acceptors at the same rate. Look’s group also concluded that annealing produced Frenkel pair recombination. This work also supports the donor nature of N vacancies in the GaN.

The effect of proton radiation on AlGaN/InGaN/GaN LEDs was reported by Osinski et al. [51]. This group used a beam energy of 2 MeV with a total fluence of $1.68 \times 10^{12}$ protons/cm$^2$. They found a 40% reduction in the output power of the LED even though the I-V curves of the devices showed very little change. The optical properties of the irradiated area almost returned to normal when the sample temperature was lowered to ~15 K. Their conclusion was that the proton dosage did not degrade the single quantum well LEDs and the optical properties remained practically unchanged.

Another group, Khanna et al. [52], studied photoluminescence of proton irradiated GaN to determine its radiation resistance. Proton beams with an energy of 2 MeV and varying fluences were used. As expected, they found that GaN was much more radiation resistant than GaAs. This was determined from PL intensity levels. A reduction in the intensity of the dominant peak in the PL spectrum of irradiated GaN was noted, implying that midgap states were created. They attributed the intensity loss as a result of trapped carriers at radiation-induced defect sites.
In a study of annealing behavior of proton radiated AlGaN/GaN HEMTs, they found remarkable recovery of the HEMT DC performance. A fluence of $1 \times 10^{14}$ cm$^{-2}$ at an energy of 1.8 MeV was used. After radiation of the HEMT the saturation of the I-V curves was reduced from 260 mA/mm to about 100 mA/mm. As with other studies the reduction was attributed to radiation induced traps resulting in removal of free carriers. Transconductance of the device was also reduced from 80 mS/mm to 26 mS/mm. However the study showed a gradual increase of electrical characteristics with increasing annealing temperatures. At a temperature of 800$^\circ$C, the saturation and transconductance returned to 220 mA/mm and 56 mS/mm respectively [53].

An experiment conducted by Emtsev et al., studied radiation induced defects of n-type GaN and InN. 1.0 to 1.5 $\mu$m thick layers of hexagonal n-GaN and InN were grown by MOCVD and plasma–assisted MBE techniques. After pre-radiation characterization by XRD and Raman spectroscopy, the samples were radiated with protons of 150keV and then annealed at 50$^\circ$C or 100$^\circ$C for 20 minutes in nitrogen. For the InN, the group found that the increase in electron concentration was most likely due to the production of radiation–induced defects with the shallow donor states. Since the production rate did not change over a wide range of proton dosage, $1 \times 10^{15}$ to $1 \times 10^{16}$cm$^{-2}$, the defects are believed to be native defects that can be attributed to immobile nitrogen vacancies in the InN. Annealing temperatures up to 100$^\circ$C show no change in electron concentration or electron mobility but did show a pronounced decrease of 30% in electron concentration between temperatures of 250-300$^\circ$C. At elevated temperatures the annealing behavior becomes complicated in the sense that a reverse annealing stage of electron concentration and mobility takes place at approximately 400$^\circ$C. At 500$^\circ$C little change in either
concentration or mobility was noted but the parameters were higher than the non-irradiated layers of InN. N-GaN: Si showed a substantial decrease in concentration of charge carriers. It was found that the electron removal rate was dependent on doping level of the GaN. After annealing to 200°C, there is a noticeable decrease in electron mobility but no significant changes in electron concentration. At temperatures of 300-400°C the mobility continues to drop but at 600°C the electron concentration and mobility recover substantially. It is noticed that the mobility of the charge carriers becomes even larger than the initial measured mobility of the n-GaN. Based on this study the group concluded that in InN, nitrogen vacancies were the likely cause of the increase of free electrons in the irradiated InN. For n-GaN they concluded that the production rate of native defects appears to be Fermi-level dependant and that two main recovery stages of electron concentration were found at intervals of 300-400°C and 500-600°C [54].

Gaubas et al., studied the radiation effects of semi-insulating layers of GaN grown on bulk n-GaN/sapphire substrates. The samples were irradiated with 10keV X-ray dose of 600Mrads and 100keV neutrons with fluences of 5e10^{14} and 10^{16} cm^{-2} respectively. A set of wafers and diode structures were irradiated then characterized post radiation using Photoluminescence spectroscopy (PL), non-invasive microwave absorption (MVA) and contact photoconductivity (CPC). The MVA method is based on a pump-probe technique with optical excitation and microwave absorption by the free carriers, the CPC relies on measuring the photocurrent decays. It was found that the radiation induces an increase in the non-radiative trap density, which resulted in a significant decrease in the PL intensity of the blue, yellow and UV bands. The effects of the disorder caused by the radiation
manifested themselves in the long tail CPC measurements as well as MWA decays with a
time stretching factor of 0.07 [55].
Table 2-1 Properties of Dielectrics Previously Studied for Use with GaN

<table>
<thead>
<tr>
<th>Material</th>
<th>Bandgap (eV)</th>
<th>Dielectric ($\varepsilon$)</th>
<th>Melting Point (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlN</td>
<td>6.2</td>
<td>8.5</td>
<td>3273</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>5.75</td>
<td>12</td>
<td>2319</td>
</tr>
<tr>
<td>Ga$_2$O$_3$</td>
<td>4.4</td>
<td>10</td>
<td>2013</td>
</tr>
<tr>
<td>Gd$_2$O$_3$</td>
<td>8.5</td>
<td>11.4</td>
<td>2668</td>
</tr>
<tr>
<td>Ga$_2$O$_3$(Gd$_2$O$_3$)</td>
<td>4.7</td>
<td>14.2</td>
<td>2023</td>
</tr>
<tr>
<td>SiO$_x$</td>
<td>8.9</td>
<td>3.9</td>
<td>1993</td>
</tr>
<tr>
<td>Si$_3$N$_4$</td>
<td>5.0</td>
<td>7.5</td>
<td>2173</td>
</tr>
<tr>
<td>MgO</td>
<td>7.3</td>
<td>9.8</td>
<td>3073</td>
</tr>
<tr>
<td>Sc$_2$O$_3$</td>
<td>6.3</td>
<td>11.4</td>
<td>2678</td>
</tr>
</tbody>
</table>
Figure 2-1 Typical MOSFET
Figure 2-2 Cross-section illustration of an enhancement mode MOSFET. A) At $V_G=0$ the device is “OFF” B) At $V_G<0$ the device is “ON”. Notice the conduction channel of electrons created with the application of positive gate voltage.
Figure 2-3 Cross section of a MOS capacitor.

Figure 2-4 Dipole formation in the presence of an electric field.
Figure 2-5 Sketch of Riber MBE used for oxide growth (after Gila)
Figure 2-6 AFM images of as received GaN. A) MOCVD GaN from Epitronics B) as received MBE GaN from SVT.
Figure 2-7  Examples of GaN surfaces before growth. A) The UV-ozone treated surface of GaN. B) Buffered oxide etched surface of GaN.

Figure 2-8  Photos of RHEED indicating a (1x3) pattern. A) <11-20> crystal direction. B) <1-100> crystal direction.
CHAPTER 3
EXPERIMENTAL APPROACH

3.1 Alternative Dielectrics

Because of the various limitations of the previously studied dielectrics, alternative dielectrics are being actively sought. Scandium oxide, magnesium oxide and the ternary combination of magnesium calcium oxide show promise as candidate dielectrics. MgO alone as a gate insulator has proven to have a low Dit, but is thermally and structurally unstable in normal atmosphere. The possible solution to this problem would be to cap the MgO with a more stable oxide layer or combine the magnesium with another dielectric material add stability to the oxide. Hence, the purpose of using a magnesium ternary oxide capped with thermally and structurally stable scandium oxide as dielectric.

The reasons for selecting these particular oxides are as follows:

• Lower lattice mismatch to the GaN semiconductor

• Wide bandgap

• Thermal stability

• High dielectric constant

• Desirable band alignment with GaN

Each of the proposed dielectrics have bandgaps that are substantially larger than the bandgap of GaN. In addition to withstanding high operating temperatures, the dielectrics should also be able to withstand irradiation of protons that are found in caustic environments of space and low earth orbits.
3.2 Oxide Growth Parameters

3.2.1 Scandium Oxide Growth

The Sc$_2$O$_3$ samples were grown using the Riber 2300 MBE equipped with a RHEED system. Scandium oxide exists in a bixbyite structure and was grown using a standard effusion cell filled with Sc operating at temperatures from 1130°C to 1170°C [56,57]. Oxygen was supplied from a RF plasma source which was kept at 200 watts forward power. The chamber pressures ranged from $1 \times 10^{-4}$ Torr to $5 \times 10^{-4}$ Torr. The substrate temperature was varied from 100°C to 600°C. Higher growth temperatures produce larger grain sizes. Electron microscopy shows a smooth interface between the Sc$_2$O$_3$ and the GaN substrate.

3.2.2 Magnesium Oxide Growth

The magnesium oxide samples were grown from 99.99% pure magnesium and Knudsen cell temperatures ranging from 350°C to 400°C. Substrate temperatures between 100°C and 340°C were used. The oxygen was provided by an Oxford RF plasma source at 300 W forward power. Oxygen pressure was varied from $8 \times 10^{-6}$ up to $1 \times 10^{-5}$ Torr. In all cases, the sample rotation was kept at 15 rpm.

3.2.3 Magnesium Calcium Oxide Growth

The magnesium calcium oxide samples were grown from the same Mg sources as that used for the MgO with the addition of 99.999% pure calcium, Ca, from another Knudsen cell with temperatures ranging from 450°C to 500°C. Substrate growth temperatures were between 100°C and 300°C. Two growth methods were used: 1) continuous where all shutters open at once and exposed to the substrate and 2) digital alloying where alternating layers of MgO and CaO are deposited. Changing the flux of
the metal sources during a continuous growth or changing the timing of the shutter sequences during digital growth varied the composition of the film. Oxygen pressure was held at 8x10^{-6} Torr and the RF plasma source. As in the MgO growth, the sample rotation was kept at 15 rpm.

### 3.3 Materials Characterization

#### 3.3.1 Auger Electron Spectroscopy

Auger Electron Spectroscopy (AES) uses a focused electron beam to create secondary electrons near the surface of a solid sample [58]. Auger electrons are able to characterize the elemental composition and at times, the chemistry of the surface of samples. When combined with ion sputtering to gradually remove the surface, AES can similarly characterize the sample in depth, allowing microanalysis of three-dimensional regions of the solid samples. Auger is normally non-destructive except during depth profiling and for materials which are sensitive to the e-beam. The main use of AES is to discover the elemental composition of inorganic materials or interface compositions. (Figure 3-1)

#### 3.3.2 Atomic Force Microscopy

Atomic Force Microscopy (AFM) is a real space imaging technique that can produce topographic images of a surface with atomic resolution in all three dimensions [58]. Atomic Force Microscopy is a very powerful imaging system since it can study insulators, semiconductors, conductors, and transparent as well as opaque materials. Surfaces can also be studied in liquid or in ultra high vacuum and the system uses a sharp tip mounted on a flexible cantilever. When the tip comes within a few angstroms of the sample’s surface, the repulsive van der Waals forces between the atoms on the tip and
those on the sample cause the cantilever to deflect. Only with an unusually sharp tip and a flat sample is the lateral resolution truly atomic. (Figure 3-2)

3.3.3 Transmission Electron Microscopy

One of the most powerful microstructural analysis techniques available is transmission electron microscopy (TEM). From TEM, not only can the microstructure of an epitaxial film be determined, but also detailed analysis of defects in the film, atomic imaging of the interface, and an accurate calculation of lattice spacing is determined. TEM uses a beam of electrons that pass through and interact with a very thin sample to form an image on the other side of the sample (Figure 3-3) [58]. The interactions between the atoms in the sample and the electrons produce the contrast seen in the image. One of the major drawbacks of TEM is the sample preparation required to obtain the images. The sample must be cut, polished, and thinned to electron transparency (~100 nm) via hand polishing and ion beam milling or by using a focused ion beam (FIB) system. This is especially difficult for the nitride materials due to their hardness. The FIB used to make these sample was a FEI Strata DB (Dual Beam) 235 FIB. A JOEL 200CX operating at 200 keV was used for film analysis and JOEL 2010FX operating at 400 keV was used for high-resolution analysis of the interface.

3.3.4 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is another technique used to give detailed material characterization. It uses a rastered electron microscope to highly magnify the image of the surface of the material [58]. The SEM works by scanning a fine probe of electrons over the surface of the specimen under vacuum conditions. As electrons penetrate and react with the surface, an emission of electrons or photons from the surface is given off. An appropriate detector can then collect the emission and the output can be
used to modulate the brightness of a cathode ray tube (CRT), whose inputs are in sync with the voltages rastering the electron beam. The image is produced on the CRT and every point the beam strikes on the sample is mapped directly onto a corresponding point on the screen. (Figure 3-4)

3.3.5 X-Ray Reflectivity

X-ray Reflectivity (XRR) is a non-destructive technique used to study the structure and the organization of materials, which are grown as thin films. The range of XRR includes the submicronic and atomic scales [59]. It can be used to determine the electron density profile (EDP) and the roughness of interfaces. By measuring the diffuse x-ray scattering, it can show the interfacial roughness between successive layers. The typical wavelength of the x-rays are 0.1 nm, which is a very high frequency. As a consequence, the x-ray interaction with matter can be described by an index of refraction, which characterizes the change of direction of the x-ray beam when passing from air to a material. The reflected intensities will be confined in a direction symmetrical from the incident x-rays beams. This technique is also very sensitive to any defects of flatness, which makes rough surfaces undesirable when performing this technique. The main advantages of XRR are the abilities to determine the surface and interface roughness, the layer thickness, EDP and the structural arrangements of complex films.

3.3.6 Photoluminescence

Photoluminescence (PL) measures physical and chemical properties of materials by using photons to induce excited electronic states in the material and analyzing the optical emission as the states relax [58]. The spectral distribution and time dependence of the emission are related to electronic transition probabilities within the sample, and can be used to provide qualitative and sometime quantitative information about chemical
composition, structure (i.e. interfaces, bonding, disorder), impurities, and energy transfer. This technique will allow useful observation of radiation effects to the interface between the dielectric and semiconductor. Figure 3-5 shows a setup of the PL system that is used in this study. A helium-cadmium laser is used as the excitation source and scanning parameters were from 340nm to 800nm.

3.3.7 Hall Effect

The Hall Effect is used to determine carrier concentration of the GaN substrate before and after thermal anneals and proton irradiation. The hall effect is a result of a magnetic field being applied perpendicular to the direction of the motion of charged particles. The magnetic field exerts a force on the current and causes the charged carriers in the current to split and gather at polar opposite sides of the sample. This enables the calculation of majority and minority charge carriers. Any change in the concentration in the carrier numbers can also be observed. The Hall voltage $V_H$ is represented by

$$V_H = \frac{(R_H I_x B_z)}{d}$$  \hspace{1cm} (3-1)

Where $R_H$ is the Hall coefficient, $I_x$ is the current in the x-direction, $B_z$ is the magnetic field in the positive z-direction and $d$ is the thickness of the sample [4].

3.3.8 Current - Voltage Analysis

A Hewlett Packard semiconductor parameter analyzer, HP4145B, was used to take I-V measurements of the MOS devices. The upper and lower current limits were set and swept from negative to positive. The range of the voltage was increased incrementally until forward and reverse breakdown were observed. Figure 3-6 depicts an ideal I-V curve for MOS devices. Note the sharp upward slope of the curve. The reasoning for this is “turn on” of the device. Once enough voltage is applied to the gate, current is conducted across the dielectric. The breakdown field strength is given by Equation 3-2.
\[ E_{BD} = \frac{V}{t} \quad (3-2) \]

Where \( t \) is the thickness of the oxide, \( V \) is the voltage at the compliance current, normally 1 mA/cm². A low breakdown field of the oxide is undesirable in this study, since it indicates weakness of the dielectric material.

### 3.1.9 Capacitance – Voltage Analysis

Capacitance-Voltage measurement is a critical analysis of electrical devices. C-V gives information about the fixed oxide charge, interface state density, border trap density and mobile ion density through graph plots or mathematical computations. All C-V data was obtained using an automated HP4284A LCR connected to a LabVIEW™ base PC. The LCR meter supplies a voltage signal of superimposed analog current (AC) and direct current (DC). Ideal C-V curves for n-type MOS capacitors are shown in figure 3-7. The C-V curves are frequency-independent in accumulation and depletion but at the onset of inversion the curves become strongly frequency-dependent. Minimizing the interface state density, \( D_{it} \), is also an important aspect of dielectric materials. A high interface state density of an oxide negates its usefulness as an insulating material for the semiconductor.

### 3.2 Diode Fabrication

Once the oxides were epitaxially deposited by MBE, the structures were fabricated into MOS diodes. Scandium oxide was etched using either an Inductively Coupled Plasma (ICP) or using a hot wet etch of \( \text{H}_2\text{SO}_4 \) for 6mins. Magnesium Oxide was etched using a 2% solution of phosphoric acid and DI H₂O for 20 seconds. The Magnesium Calcium Oxide was etched using the same 2% Phosphoric Acid/DI H₂O solution. Ohmic contacts consisted of 200Å Ti/700Å Al/400Å Pt/1000Å Au and were deposited by e-beam evaporation. Gate contacts were Pt/Au, 200Å and 1000Å respectively. No post anneal
was performed after deposition of the contacts. The finished MOS capacitors are shown in figure 3-8.

3.3 Proton Radiation Setup and Facility

For proton radiation the samples were taken to the Texas A&M Cyclotron. The oxide and device samples were radiated at doses of $5 \times 10^9 \text{ cm}^{-2}$ at energies of 10MeV and 40MeV under a vacuum in the $10^{-4} \text{Torr}$ range. The chamber takes at least six minutes to pump down before radiation can start. The samples were affixed to glass slides and then placed on the target mounting in the testing chamber as shown in figure 3-9.

The target mount frame can be adjusted in x, y, and z directions and rotated by computer control. The size of the exposed area is controlled by a shutter like aperture that can be adjusted horizontally and vertically to insure beam isolation to the desired target area. Beam uniformity and dosimetry are monitored by an array of five plastic scintillators located upstream from the target chamber. Beam uniformities of 95% or better can be achieved. Outside of the testing chamber, an electronic diagnostic setup is available for data acquisition, in-situ or ex-situ testing (depending on the device) and quick target changes (Figure 3-10).
Table 3-1 Proposed Oxide Properties.

<table>
<thead>
<tr>
<th></th>
<th>Bandgap (eV)</th>
<th>Dielectric Constant (ε)</th>
<th>Melting Point (K)</th>
<th>Mismatch to GaN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sc₂O₃</td>
<td>6.3</td>
<td>11.4</td>
<td>2678</td>
<td>9.2%</td>
</tr>
<tr>
<td>MgO</td>
<td>7.3</td>
<td>9.8</td>
<td>3073</td>
<td>-6.5%</td>
</tr>
<tr>
<td>MgCaOᵃ</td>
<td>7.4</td>
<td>10.8</td>
<td>2370</td>
<td>-</td>
</tr>
<tr>
<td>GaN</td>
<td>3.4</td>
<td>9.5</td>
<td>2773</td>
<td>–</td>
</tr>
</tbody>
</table>

a. composition of approx 50/50
Figure 3-1 Auger Electron Spectroscopy set-up.
A piezoelectric scanner which moves the tip over the sample (or the sample under the tip) in a raster pattern

A feedback system to control the vertical position of the tip

A way of sensing the vertical position of the tip

A sharp tip

A course positioning system to bring the tip into the general vicinity of the sample.

A computer system that drives the scanner, measures data and converts the data into an image.

Sample

Figure 3-2 Atomic force microscope (after K.K. Harris 2000).
Figure 3-3 TEM setup used to image atomic layers at the film/substrate interface.
Figure 3-4 SEM operation. Electron beam is rastered over the sample producing secondary electrons (after S.M. Donovan 1999).
Figure 3-5 Photoluminescence setup
Figure 3-6 Ideal MOS I-V curve

Figure 3-7 Ideal C-V curves of n-type MOS (after Johnson)
Figure 3-8 Finished MOS capacitors design.
Figure 3-9  Test Chamber for Radiation. A) Mounted samples in test chamber. B) Shutters of beam.
Figure 3-10 Radiation Test Facility at Texas A&M University.
Various types of GaN samples were tested pre- and post-proton radiation using photoluminescence (PL). The effects of rapid thermal annealing (RTA) were also investigated. The types of gallium nitride that were tested included n-type GaN, a high resistivity GaN (known as u-GaN), MBE grown GaN, and p-type GaN. PL was used to determine the initial luminescence state of the GaN and monitor changes in the luminescence profile after proton radiation and thermal annealing. RTA was used to determine if the radiation damage to the sample could be corrected by heating the sample to various temperatures.

4.1 Characterization of MOCVD N-type GaN

As grown commercial MOCVD GaN, supplied by Uniroyal Optoelectronics (UOE), was probed using PL. Figure 4-1 shows the initial GaN luminescence profile at room temperature (300K) and low temperature (15K). At 362nm(3.42eV), the primary transition or bandedge of the GaN is noted in addition to a defect band in the 450nm to 700nm range, which is related to various midgap defects in the gallium nitride. This yellow defect band gives n-GaN its distinctive luminescence color. Although the origin of this yellow band is heavily debated, the defect origins are most likely grown-in nitrogen vacancies. Notice at 15K the large defect emission from 450nm to 700nm has been suppressed. According to Khanna et al., the suppression of the defect band at low
temperatures is a result of reduced non-radiative transitions and a lack of available phonons [52].

The UOE n-GaN samples were irradiated at two different energies of 10MeV and 40MeV with a total particle dose of $5 \times 10^9 \text{cm}^{-2}$, which equals approximately 10 years of mission time in space applications. Figures 4-2 and 4-3 show the post radiation damage to the GaN in comparison to the initial scan at 300K and 15K respectively. At 300K a significant decrease in luminescence intensity is noticed as well as a slight shift in the bandedge peak to 354nm. Post radiation at 15K shows an increase in the defect band of the profile which can be attributed to the increased amount of defects within the bulk material that have now become radiative centers for electron–hole recombination. The bandedge is significantly decreased also due to the introduction of non-radiative defects. Also due to the radiation damage the UOE n-GaN samples luminescence a pale green color and not the customary yellow that characterizes GaN. Based on previous work done by other groups, the reason for this decrease in intensity and change in color is an increase in lattice defects.

After a year, the samples were rescanned with PL to determine the luminescence profile. Figure 4-4 shows a comparison to the initial GaN scans and the post radiated GaN. After one year, the defect luminescence returns to its pre-irradiated state. Similarly, there is a partial though not complete recovery of the bandedge emission. This change in the profile from the immediate post radiation scans implies either long life-time traps have relaxed or surface carrier loss has been suppressed via oxidation or adsorption of adventitious species such as carbon. The samples still luminescence with a
pale green-yellow and the PL profile was not completely returned to the original scan which shows that not all the changes caused by irradiation were corrected.

In order to determine if the radiated samples could be returned to their initial profile by thermal treatment, the UOE n-GaN samples were annealed using an RTA setup with varying temperatures ranging from 200°C to 900°C and held for 60 seconds at each temperature. After each temperature anneal, the GaN samples were rescanned using PL. In order to better monitor the changes in the n-GaN induced by annealing, a non-irradiated n-GaN sample from the same wafer as the irradiated samples was also annealed and PL scanned. Figures 4-5 through 4-7 shows a comparison of the annealed radiated and non-radiated samples of n-GaN. After annealing at 200°C a significant increase in PL intensity was observed for all samples but at 300°C the intensity decreases well below the original level. It seems unlikely that diffusion of nitrogen and gallium atoms to vacancies or interstitial sites within the GaN lattice has occurred at such a low temperature. Thus, it is possible that some surface phenomena are responsible, though the exact mechanism is as yet unknown. At higher temperatures of 400°C to 900°C, no large changes to the PL profiles in either intensity or bandedge shifts were observed. Based on figure 4-8, the GaN has reached a fairly steady configuration of defects within the lattice and that no more recovery or improvement of the samples can be achieved without completely damaging the GaN substrate. Figure 4-8 is a calculated trend of the annealed sample changes based on ratio comparisons of normalized bandedge peaks of all annealed GaN to the GaN control peak intensity of 1.4812. After 600°C, there is a gradual decrease in bandedge intensity indicating that further annealing is damaging the optical properties of the GaN.
After reviewing the results of the first UOE n-GaN, a further study of other types of GaN with different growth recipes and growth systems was undertaken in order to further investigate the phenomena at 200°C. Another UOE n-GaN sample from another wafer, which will be referred to as the 2nd UOE n-GaN sample, was scanned with PL, radiated at 10MeV and 40MeV and annealed from 200°C to 900°C to determine the 2nd UOE n-GaN trend. Figure 4-9 shows the pre and post radiation PL profiles of the 2nd UOE n-GaN. In comparison, both UOE n-GaN samples show the same basic trends in the radiation and thermal experiments. Figures 4-10 through 4-12 show the radiation and thermal effects on PL profiles for the 2nd UOE n-GaN sample. All of the data from the 2nd UOE n-GaN sample was collected immediately after radiation. As noted before, after annealing at 200°C a large increase in the PL intensity is observed. Thus far, this phenomenon has shown to be unique to all UOE GaN (n-GaN and u-GaN) samples and was not observed for any of the other GaN types, which will be discussed in later sections. This phenomenon could have various reasons, such as sample preparation, growth parameters for UOE GaN, surface effects or material doping, which are unknown and proprietary information at the present time. In figure 4-13, the normalized bandedge peak intensity versus annealing temperature is shown for the 2nd UOE n-GaN sample. The trend in the plot generally follows the first UOE n-GaN with no significant changes in the profiles after 400°C. For both samples, annealing was not effective in completely restoring the spectra to the pre-irradiated profile, and after annealing at 900°C the luminescence ratio is almost equal to that of the radiated sample ratio. At the higher temperatures, it is likely that nitrogen is being driven out of the sample and leaving behind nitrogen defects that essentially creates non-radiative centers.
In addition, n-GaN grown by an MOCVD system at the University of Florida (UF) was also studied and underwent the same experiments as the UOE n-GaN to explore the potential variability in MOCVD GaN. The GaN grown at UF sample will be referred to as UF n-GaN throughout the paper. Figure 4-14 shows the pre and post radiation PL scans, like the UOE n-GaN, irradiation of the UF n-GaN produced a significant drop in PL intensity. Figures 4-15 through 4-17 shows the results of rapid thermal annealing from 200°C to 900°C for each UF n-GaN sample. Figure 4-18 plots the bandedge peaks normalized to 25.069 in intensity. Unlike the UOE n-GaN, the non-irradiated UF n-GaN shows a significant drop in both bandedge and defect luminescence for all annealing temperatures. Annealing of the irradiated material produces little to no improvement in the intensity of the PL.

4.2 Characterization of MOCVD U-type GaN

As with the n-GaN samples, a set of UOE verses UF u-GaN was tested and compared. Generally u-GaN has lower luminescence intensity due to a reduction in free carriers. Figure 4-19 shows the changes in the PL scan after proton radiation at 10MeV and 40MeV. Notice that there is a severe reduction in the already low peak intensity. The broadness of the bandedge peak indicates the presence of defects in the gap of the GaN. Figures 4-20 through 4-22 depicts the effects of thermal annealing of the samples. In each of the samples there is an increase at 200°C, which, as mentioned before in previous section, is mostly likely surface effects of the GaN since the intensity continuously drops with increasing annealing temperatures beyond the temperature. Normalizing the bandedges peak intensity to the control sample bandedge shows the trend of increasing and decreasing intensity with annealing temperature (figure 4-23). At 400°C there is a severe decrease in intensity and no additional drastic change or recovery of the samples.
The UF u-GaN shows higher peak intensity than the UOE u-GaN possibly indicating a better quality sample. Figure 4-24 shows the pre and post radiation scans of the UF u-GaN and in figures 4-25 through 4-27 the thermal annealing is shown. The main trend of both the UOE u-GaN samples and the UF u-GaN is that at 400°C the control samples and the radiated samples are about the same in peak intensity and continue in a linear fashion to 900°C. Figure 4-28 exhibits this trend in the UF u-GaN. Radiation reduced the bandedge but only slightly reduced the defect luminescence. As with the UF n-GaN, annealing at all temperatures reduce the PL intensity.

4.3 Characterization of MBE Grown Ga-Polar GaN

MBE gallium nitride is known to be highly resistive mostly likely due to self-compensation of the Ga and exhibit for weak PL emissions due to lack of free carriers available in the samples for emission transitions. Figure 4-29 shows the comparison of the pre and post radiation scans of the Ga-polar GaN sample. The bandedge peak after irradiation is no longer visible in the PL scan mostly indicating a large increase in optically active defects in the samples or degradation of the surface. After annealing each sample (figures 4-30 through 4-32), there was a slight increase in the peak intensity at the higher temperatures but it never recovers to the original peak intensity. Figure 4-33 shows the normalized bandedge peak intensities to the control versus the annealing temperatures.

4.4 Characterization of MOCVD P-type GaN

The comparison of UF MOCVD grown p-type GaN was studied with PL, annealing, and Hall to determine the behavior of the sample after proton irradiation and thermal testing. The samples were taken from the same wafer but one half of the wafer received a post activation anneal of the Mg implantation at 750°C for 30 seconds in the
RTA. The as grown p-GaN will be referred to as P-GaN and the part of the wafer that received the post growth activation anneal will be referred to as Activated P-GaN. In figure 4-34 is the PL comparison of each control sample. Figures 4-35 and 4-36 shows the pre and post PL scans of the p-GaN and activated p-GaN samples. Note the difference in peak intensity of the control samples and then the observed decrease in intensity of the radiated p-GaN samples but the increase in the activated p-GaN samples. It is possible that the radiation in the activated samples has caused ionization of the Mg acceptors. Figures 4-37 through 4-38 show the PL scans after annealing each of the p-GaN (un-activated and activated) control samples and figures 4-39 and 4-42 captures the trend of the 10MeV and 40MeV radiated p-GaN (un-activated and activated) samples. Each of the samples experienced a sharp decline in intensity at 400°C and again at 600°C. They each increased in peak intensity at 900°C which mostly likely can be attributed to defect annealing. Each of the un-activated p-GaN samples generally follows the same trend with or without radiation, whereas the activated p-GaN shows a larger spread in the ratios of the normalized bandedge peaks (figures 4-43 and 4-44). Figure 4-45 compares the two plots of bandedge peaks versus annealing temperature where it exhibits the difference in the changes of the samples. The as grown p-GaN look much more linear than the activated p-GaN.

4.5 Summary

Based on the data collected, some general trends for the GaN can be noted. First is that radiation of the samples decrease the PL luminescence intensity. Second, an increase of PL intensity at 200°C has shown to be unique to the UOE GaNs and as-grown P-GaN. After approximately 400°C, the drastic decline in peak intensity stopped and became less
significant in change with higher temperatures. These defects become non-radiative centers and trap carriers that contribute to optical transitions in the bandgap of the GaN.
Figure 4-1 Initial PL scans of non-radiated 1st UOE n-GaN at temperatures 300K and 15K.
Figure 4-2 Room Temperature PL analysis of 1st UOE n-GaN before and after radiation.
Figure 4-3 15K PL analysis of 1st UOE n-GaN before and after radiation.
Figure 4-4 Comparison of 300K 1st UOE n-GaN PL scans, pre-radiation, immediately post radiation, and post radiation 1 year later.
Figure 4-5 PL analysis of annealed un-radiated 1st UOE n-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-6 PL analysis of annealed 10MeV radiated 1st UOE n-GaN A) PL Spectra B) Bandedge inset (below).
Figure 4-7 PL analysis of annealed 40MeV radiated 1st UOE n-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-8 Normalized bandedge peaks of all annealed 1st UOE GaN to the 1st UOE GaN control peak intensity of 1.4812 vs. the annealing temperatures. 0 temperature indicates the initial state of the GaN.
Figure 4-9 Pre and post radiation PL analysis of 2nd UOE n-GaN.
Figure 4-10 PL analysis of un-radiated annealed 2nd UOE n-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-11 PL analysis of annealed 10MeV radiated UOE n-GaN. A) PL Spectra B) Bandedge inset
Figure 4-12 PL analysis of annealed 40MeV radiated UOE n-GaN. A) PL Spectra B) Bandedge inset
Figure 4-13 Normalized bandedge peaks of annealed 2\textsuperscript{nd} UOE n-GaN to the 2\textsuperscript{nd} UOE n-GaN control peak intensity of 12.354 vs. the annealing temperatures.
Figure 4-14 Pre and post radiation PL analysis of UF n-GaN.
Figure 4-15 PL analysis of annealed un-radiated UF n-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-16 PL analysis of annealed 10MeV radiated UF n-GaN. A) PL Spectra B) Bandedge inset
Figure 4-17 PL analysis of annealed 40MeV radiated UF n-GaN. A) PL Spectra  B) Bandedge inset
Figure 4-18 Normalized bandedge peaks of annealed UF n-GaN to the UF n-GaN control peak intensity of 25.069 vs. the annealing temperatures.
Figure 4-19 Pre and Post Radiation of UOE u-GaN.
Figure 4-20 PL analysis of annealed un-radiated UOE u-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-21 PL analysis of annealed 10MeV radiated UOE u-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-22 PL analysis of annealed 40MeV radiated UOE u-GaN. A) PL spectra B) Bandedge inset.
Figure 4-23 Normalized bandedge peaks of annealed UOE u-GaN to the UOE u-GaN control peak intensity of 0.1162 vs. the annealing temperatures.
Figure 4-24 Pre and post radiation PL analysis of UF u-GaN
Figure 4-25 PL analysis of annealed un-radiated UF u-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-26 PL analysis of annealed 10MeV radiated UF u-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-27 PL analysis of annealed 40MeV radiated UF u-GaN. A) PL Spectra B) Bandedge inset.
Figure 4-28 Normalized bandedge peaks of annealed UF u-GaN to the UF u-GaN control peak intensity of 1.2997 vs. the annealing temperatures.
Figure 4-29 Pre and Post Radiation PL analysis of MBE Ga-polar GaN.
Figure 4-30 PL analysis of annealed un-radiated MBE Ga-polar GaN. A) PL Spectra B) Bandedge inset.
Figure 4-31 PL analysis of annealed 10MeV radiated MBE GaN. A) PL Spectra B) Bandedge inset.
Figure 4-32 PL analysis of annealed 40MeV radiated MBE GaN. A) PL Spectra B) Bandedge inset.
Figure 4-33 Normalized bandedge peaks of annealed MBE Ga-polar GaN to the MBE Ga-polar GaN control peak intensity of .01016 vs. the annealing temperatures.
Figure 4-34 Comparison of PL scans of as grown P-GaN and Activated P-GaN.
Figure 4-35 Pre and post radiation PL analysis of as-grown P-GaN.
Figure 4-36 Pre and post radiation PL analysis of Activated P-GaN.
Figure 4-37 PL analysis of annealed as-grown un-radiated P-GaN.
Figure 4-38 PL analysis of annealed un-radiated Activated P-GaN.
Figure 4-39 PL analysis of annealed 10MeV as-grown P-GaN
Figure 4-40 PL analysis of annealed 40MeV as-grown P-GaN.
Figure 4-41 PL analysis of annealed Activated P-GaN radiated at 10MeV.
Figure 4-42 PL analysis of annealed Activated P-GaN radiated at 40MeV.
Figure 4-43 Normalized bandedge peaks of annealed as grown P-GaN to the as grown P-GaN control peak intensity of 2.7808 vs. the annealing temperatures.
Figure 4-44 Normalized bandedge peaks of annealed Activated P-GaN to the Activated P-GaN control peak intensity of .42224 vs. the annealing temperatures.
Figure 4-45 All Normalized P-GaN bandedge peaks to control peaks of 2.7808 and .42224 respectively.
CHAPTER 5
RADIATION AND THERMAL STABILITY OF NOVEL OXIDES ON GALLIUM NITRIDE

Dielectrics on GaN were tested pre- and post-proton radiation using, XRR and PL. The effects of rapid thermal annealing (RTA) were also investigated. PL was used to determine the initial luminescence state of the GaN. GaN has a distinct luminescence profile, changes in which can be monitored by observing the peak intensity changes and shifts in the peak wavelengths. XRR was used to monitor the morphological changes between the interface of the dielectric and the GaN. Observations of the periodic oscillations and amplitude of the oscillations can show roughing at the interface due to particle bombardment by the radiation. Annealing was used to determine if the damage to the sample could be corrected by heating the sample up to various temperatures. After analyzing the results of the GaN samples, three different oxides of ~400Å Sc$_2$O$_3$, ~200 Å MgO with a ~200 Å Sc$_2$O$_3$ capping layer, and ~200 Å MgCaO with a ~200 Å capping layer were epitaxially grown on UOE n-GaN to determine if the oxide layers would protect the interface between the oxide and the GaN as well as reduce the bulk damage to the GaN. The oxide/GaN samples were initially characterized with PL and XRR. The samples were then irradiated with protons at two different energies of 10MeV and 40MeV with a total dose of $5 \times 10^9 \text{cm}^{-2}$ and were then re-characterized using the same techniques.
5.1 Characterization of Sc₂O₃ on GaN

Figures 5-1 shows the initial PL and XRR profiles of the Sc₂O₃/GaN. The starting roughness at the interface of the oxide and semiconductor and the surface of the oxide are ?? and ?? respectively. In figure 5-2 a noticeable decrease in bandedge intensity is observed for both radiation energies for the Sc₂O₃/GaN sample but an increase in the defect density intensity for the sample irradiated at 40MeV. The PL scan taken at 15K shows an increase in the defect band emission and a large bandedge intensity increase. The XRR data in figure 5-3 shows the reduction in periodicity and amplitude of the XRR scan relative to the initial scan, which indicates roughening of the Sc₂O₃/GaN sample interface, due to particle bombardment of the proton radiation. The oxide sample exhibited the same pale green color as the non-oxide/GaN.

The samples were then scanned a year later to determine any changes in the PL profiles. Figure 5-4 shows a comparison of the initial, immediate post radiation, and post radiation one year later. As with the non-oxide GaN, these samples also showed that some relaxation had occurred within the samples. Notice the return of the peak intensity in comparison to the immediate post radiation scans, however, the sample did not completely return to its initial as grown state. Also, the oxide/GaN samples still luminesced a pale green-yellow color indicating that the samples had not completely recovered from the damage received from the irradiation.

The samples were subjected to annealing to determine weather any more recovery of the damage could be seen. A companion non- radiated sample was subjected to the same annealing temperature ranges of 200°C to 900°C in order to compare them to the irradiated annealed samples to determine how much recovery or damage is done by just annealing alone. Figures 5-5 through 5-7 show the PL scans Sc₂O₃/GaN samples after
annealing from 200°C to 900°C. Notice the large increase in the scans at 200°C and then a decrease at 300°C. The Sc$_2$O$_3$/GaN PL scans show an increase from 200°C to 400°C in both bandedge intensity and defect density intensity. The increases appear to be approximately the same for each annealed temperature, which can possibly indicate that annealing is producing more defects in the sample. This phenomenon was also seen in the non-oxide GaN, which indicates that this is possibly a surface effect of the GaN. After annealing to 400°C, the samples were rescanned using XRR to look at the effects the annealing had on the interfaces. Figures 5-8 and 5-9 shows the comparison of the initial XRR scans to the post-radiated post-annealed scans. Annealing the samples to 400°C has shown further roughening of the interfaces of the oxide to the GaN substrate.

After annealing the samples to 400°C, they were then scanned by XRR to look at the effects of just annealing on the interfaces of the samples. XRR showed a loss of periodic oscillations as shown in figure 5-7 and as with the radiated samples, the interfaces have roughened with annealing. Results of roughening are current leakage via interface state traps once the oxide/GaN is fabricated into a MOS device, which shortens the gate breakdown voltage of the device.

### 5.2 Characterization of Sc$_2$O$_3$/MgO on GaN

Figures 5-11 and 5-12 shows the initial PL and XRR profiles of the Sc$_2$O$_3$/MgO/GaN. For the Sc$_2$O$_3$/MgO samples, both showed a decrease in bandedge and defect density intensity at both radiation energies at room temperature but at 15K there is a marked increase in the intensity of the bandedge and defect densities. Figure 5-13 shows the damage to the interface via loss of oscillations at 40MeV. The MgCaO/Sc$_2$O$_3$ sample displayed a decrease in bandedge and defect density at both energies at room temperature and low temperature of 15K as shown by figure 4-16. For the MgO/Sc$_2$O$_3$,
the PL scans are virtually identical indicating that there is no significant change has taken place to change the luminescence profile. However, the XRR scan does show roughening of one or more of the samples has occurred.

5.3 Characterization of Sc$_2$O$_3$/MgCaO on GaN

Figures 5-1 and 5-2 show the initial PL and XRR profiles of the Sc$_2$O$_3$/MgCaO/GaN. In comparison to the other oxide samples the PL scan shows a slight difference in the defect density peaks, noted by the more narrowed spikes in the 450nm to 700nm range. A possible reason for this is the lattice matching between the MgCaO/GaN interface. This is also evident in the XRR comparison of the pre and post irradiation data in figure 4-17. The XRR profile has some change at the interface but not as much as with the other less latticed matched oxides.

In figure 4-30 the post annealed PL scans of MgCaO/Sc$_2$O$_3$ show identical scan at 200°C and 300°C but reveals an increase in both bandedge and defect densities at 400°C. This is most likely caused by the creation of defects and traps since both peaks have increased about the same. The XRR scan shows a decrease in the oscillations, which as stated before indicated roughening at one or more the interfaces.

5.4 Summary

The roughening of the oxide/GaN interface suggests that operating temperatures of any device fabricated from the MgO and MgCaO oxides maybe be limited. As with the non-oxide GaN samples the irradiation of the oxide samples still significantly decreases the bandedge emission and increases the defect emission. The radiation also causes roughening of the oxide/semiconductor interface and roughening at the surface. After one year, the samples show some relaxation of long life-time traps that degrade the PL emission, as was shown with the non-oxide GaN. The effect of annealing on the oxide
samples also causes roughening at the surface and interface in the MgO and MgCaO samples. Of the three different oxides, Sc$_2$O$_3$ shows the least amount of change after annealing to 400°C. The MgO with capping layer shows the most change based on the XRR scans that was done implying that although the capping layer has slowed degradation of the MgO, there is still some hydrolyzing of the MgO occurring. Radiation of the samples makes them more susceptible to annealing damage because of the defects already present. As stated in earlier sections, the purpose of the alternate dielectrics is to find one that allowed for elevated operating temperatures.
Figure 5-1 Preliminary Sc$_2$O$_3$/GaN A) PL spectra and B) XRR scan.
Figure 5-2 Sc$_2$O$_3$/GaN PL analysis. A) PL at 300K. B) PL at 15K.
Figure 5-3 Sc$_2$O$_3$/GaN XRR scans showing the change in oscillations.
Figure 5-4 Sc$_2$O$_3$/GaN PL scans of pre-radiation, immediate post radiation and post irradiation 1 year later. A) PL spectra B) Bandedge inset.
Figure 5-5 PL scans of samples annealed non-radiated Sc$_2$O$_3$/GaN to 900C. A) PL Spectra B) Bandedge inset
Figure 5-6 Sc$_2$O$_3$/GaN post radiation anneals to 900°C for radiation energies of 10MeV. A) PL spectra B) Bandedge inset.
Figure 5-7 Sc$_2$O$_3$/GaN post radiation anneals to 900°C for radiation energies of 40MeV.
A) PL Spectra  B) Bandedge inset.
Figure 5-8 Sc$_2$O$_3$/GaN XRR scans of unradiated annealed sample at 400C.
Figure 5-9 Sc$_2$O$_3$/GaN XRR scans pre and post radiation annealed at 400°C.
Figure 5-10 Normalized bandedge peaks of annealed Sc$_2$O$_3$/GaN to the Sc$_2$O$_3$/GaN control peak intensity of .5139 vs. annealing temperature.
Figure 5-11 Preliminary Sc2O3(20nm)/MgO(20nm)/GaN PL spectra and XRR. A) PL spectra B) XRR scan.
Figure 5-12 Sc2O3(20nm)/MgO(20nm)/GaN PL scans. A) PL at 300K B) PL at 15K.
Figure 5-13 Sc2O3(20nm)/MgO(20nm)/GaN XRR scan showing the change in oscillations at 40 MeV.
Figure 5-14 Sc2O3(20nm)/MgO(20nm)/GaN PL scans of pre-radiation, immediate post radiation and post irradiation 1 year later. A) PL spectra B) Bandedge inset.
Figure 5-15 PL scans of samples annealed non-radiated Sc2O3(20nm)/MgO(20nm)/GaN to 900°C. A) PL Spectra B) Bandedge inset.
Figure 5-16 Sc2O3(20nm)/MgO(20nm)/GaN post radiation anneals to 900°C for radiation energies of 10MeV. A) PL spectra B) Bandedge inset.
Figure 5-17 Sc2O3(20nm)/MgO(20nm)/GaN post radiation anneals to 900°C for radiation energies of 40MeV. A) PL spectra B) Bandedge inset.
Figure 5-18 Sc2O3(20nm)/MgO(20nm)/GaN XRR scan of annealed sample.
Figure 5-19 Sc2O3(20nm)/MgO(20nm)/GaN XRR scans pre and post radiation annealed at 400°C.
Figure 5-20 Normalized bandedge peaks of annealed Sc2O3(20nm)/MgO(20nm)/GaN to the Sc2O3(20nm)/MgO(20nm)/GaN control peak intensity of .21556 vs. annealing temperature.
Figure 5-21 Preliminary Sc$_2$O$_3$(20nm)/MgCaO(20nm)/GaN. A) PL spectra B) XRR scan.
Figure 5-22 Sc2O3(20nm)/MgCaO(20nm)/GaN PL scans. A) PL at 300K B) PL at 15K (bottom).
Figure 5-23 Sc2O3(20nm)/MgCaO(20nm)/GaN XRR scan showing the change in oscillations.
Figure 5-24 Sc2O3(20nm)/MgCaO(20nm)/GaN PL scans of pre-radiation, immediate post radiation and post irradiation 1 year later. A) PL spectra B) Bandedge inset.
Figure 5-25 PL scans of samples annealed non-radiated Sc2O3(20nm)/MgCaO(20nm)/GaN to 900C. A) PL Spectra B) Bandedge inset.
Figure 5-26 Sc2O3(20nm)/MgCaO(20nm)/GaN post radiation anneals to 900°C for radiation energies of 10MeV. A) PL spectra B) Bandedge inset.
Figure 5- Sc2O3(20nm)/MgCaO(20nm)/GaN post radiation anneals to 900°C for radiation energies of 40MeV. A) PL spectra B) Bandedge inset.
Figure 5-28 Sc2O₃(20nm)/MgCaO(20nm)/GaN XRR scan of annealed sample.
Figure 5-29 Sc2O3(20nm)/MgCaO(20nm)/GaN XRR scans pre and post radiation annealed at 400°C.
Figure 5-30 Normalized bandedge peaks of annealed Sc2O3(20nm)/MgCaO(20nm)/GaN to the Sc2O3(20nm)/MgCaO(20nm)/GaN control peak intensity of 1.2968 vs. annealing temperature.
CHAPTER 6
PROCESSING AND THERMAL STABILITY OF MOS DEVICES

The following sections will discuss the processing of novel gate dielectric semiconductor devices and the thermal stability of the fabricated MOS devices.

6.1 Processing of Novel Oxide/Gallium Nitride Devices

Through much trial and error a process for fabricating single structure and stack structure MOS diodes was found. A mask set was designed to be used for a variety of MOS diodes. The mask set was developed using Microsoft PowerPoint that was reduced to MicroFiche to acquire the desired devices sizes. Figure 6-1 shows the basic mask design, which had a standard ohmic pad and two different gate pad sizes of 50µm and 100µm. Two different methods of processing the MOS devices were studied.

1. A process in which ohmic contacts were deposited before oxide growth on GaN.
2. A process in which ohmic contacts were deposited after dielectric growth on GaN.

The advantage of putting ohmic contacts on the GaN before oxide growth, allows for annealing of the ohmic contacts without causing thermal damage to the oxide. However, once the ohmic contacts are on the GaN and annealed, the substrate should ideally go thru a dilute standard MBE substrate cleaning before growth of the oxide can be done. The problem that can arise with the standard cleaning, it is possible damage to the ohmic contacts via etching of the metal from the buffered oxide etch used to clean the substrate surface. A simple ohmic contact study was performed to verify which conditions would result in the best ohmic contacts. Four ohmic samples with the following conditions were tested:
1. RTA Anneal at 750°C for 30 seconds
2. No Pre Clean, in-situ MBE Anneal at 700°C for 5 minutes facing sources
3. Pre Clean with UV-Ozone and dilute BOE, in-situ MBE anneal at 700°C for 5 minutes facing sources.
4. UV-Ozone Treatment, in-situ MBE anneal at 700°C for 5 minutes facing sources.

Figure 6-2 shows the IV results for each of the conditions. Based on the results the ohmic sample that received just the UV-Ozone treatment, showed a more linear profile after annealing and proved to be the better condition for ohmics before oxide growth. One problem that does exist with this approach is making contact with the electrical probes through the oxide to the ohmic pad. Hence it was necessary to find an etching recipe that would work for the stack structures. Table 6-1 shows the recipes used to etch each oxide that was studied. The stack structure of Sc$_2$O$_3$/MgCaO or Sc$_2$O$_3$/MgO is dry etched through the scandium oxide and then wet etched through the MgO or MgCaO. For this study all the diode samples were processed post oxide growth on u-GaN substrates.

6.2 Proton Radiation Effects of GaN MOS Diodes

Radiation effects of different oxides and oxide structures were tested to determine which oxide would be the least susceptible to proton bombardment. Figures 6-3 and 6-4 show the IV and CV behavior of the Sc$_2$O$_3$/GaN sample before and after radiation. Although there is a decrease the turn-on has also become more abrupt. The CV shows approximately a positive 1V flatband voltage shift possibly indicating an increase in fixed oxide charge. In figures 6-5 and 6-6, the electrical behavior of the MgO stack structure oxide is presented. Both IV and CV show degradation of the electrical characteristics of the sample. In the current-voltage plot there is a decrease in breakdown voltage and in capacitance-voltage, there is a small amount of change post irradiation that is affecting
the fixed oxide charge. For the MgCaO stack structure to samples with different oxides
thicknesses where grown and tested to determine which thickness of MgCaO was better
to have at the interface of the GaN device. As it is shown in figure 6-7, the sample with
the 5nm MgCaO showed better breakdown than the 35nm MgCaO. From this point the
5nm MgCaO with capping layer was investigated for the radiation and thermal annealing.
Figure 6-8 shows the IV breakdown of the sample after 10MeV irradiation. As expected
the breakdown decreases. This is keeping with the model that irradiation of GaN devices
still experience some degradation after exposure to protons. Figure 6-9 shows the CV
characteristics of the ternary oxide. As noted there is a large increase of forward flatband
voltage shift, which has resulted in a poor CV curves. Inaccurate or poor data does not
allow for calculations of interface state density. Interface state densities(Dit) of the
irradiated sample are as listed in table 6-2. In figure 6-10, the Dit plots for the Sc$_2$O$_3$/GaN
and the Sc$_2$O$_3$/MgO/GaN samples. The degradation of the ternary oxide was too poor to
resolve a dit measurement from.

6.3 Effects of In-situ and Ex-situ Thermal Annealing

Scandium oxide was tested to determine the electrical characteristics for use as a
capping layer for MgO and MgCaO. Figure 6-11 shows the IV results of the Sc$_2$O$_3$/GaN
during heating on the hot chuck of the probe station. The measurements were taken as the
sample was heated from 100°C to 300°C, typical operating temperatures for GaN devices.
Notice that while the sample is hot, the breakdown is less than when the sample as been
cooled back to room temperature. The figure also shows an increase in breakdown with
after the final cooling back to room temperature after being heated to 300°C. After 100°C
no relevant CV data could be collected as shown in figure 6-12.
A significant problem with MgO that has been observed in the past, is the slow degradation of the oxide in air. Over a period of time the oxide begins to deteriorate through the formation of hydroxyl species. Hence the reason for using Sc$_2$O$_3$ as a capping layer to the MgO, to prevent the degradation of the sample while it is exposed to air, resulting in 8nm of Sc$_2$O$_3$ capping layer and 40nm of MgO on u-GaN. This sample was grown at a low oxygen pressure of .2 torr$^{-1}$, which has shown to have better electrical characteristics than oxides grown at higher oxygen pressures. Figure 6-13 shows the thermal behavior for IV testing of the sample. Thermal cycling of the sample decreases its breakdown field and during heating, there was no relevant CV during the heating of the sample. This implies that there are long-time traps that are not reaching equilibrium while the sample is heated. Figure 6-14 shows the post heating CV behavior. Post 100$^\circ$C there is some slight improvement of the flat band voltage shifts but this worsens as the sample is heated and cooled again over 200$^\circ$C to 300$^\circ$C, as seen by the forward shift in flat band voltage.

Magnesium calcium oxide (MgCaO) was grown to determine the electric characteristics since MgCaO has a lattice match that is very close to the lattice of GaN. The idea behind this is that the better the lattice matching the smaller interface defect density. High interface defect densities create current leakages, which affect the threshold gate voltage, $V_{th}$. All devices using MgCaO are capped with scandium oxide to prevent degradation of the MgCaO. The most critical layers of the dielectric occur several monolayers at the interface. 5nm of MgCaO followed by 35nm Sc$_2$O$_3$ was grown on one sample and 35nm of MgCaO with 5nm Sc$_2$O$_3$ cap was grown on the second sample. Both of the samples were fabricated into MOS diodes and tested. As shown previously in
figure 6-7 the current-voltage measurements of the two different dielectric thicknesses of the MgCaO. From the data shown the MgCaO breakdown voltage for both samples is roughly .75 to 1 V. The low breakdown suggests that even thought the oxide is maor lattice matched there is still a lot of current leakage through the oxide. Two possible reasons are:

1. Defects in the oxide: changes in the epitaxial growth process may improve the interface, which could in turn improve the breakdown voltage.

2. Residual water: since it is known that MgCaO readily hydrolyzes in water, care should be taking during the fabrication process to dehydrate the sample via low temperature furnace bakes after water is used on the sample.

These few changes could very well make a significant difference in the electrical measurements of the MgCaO devices. The sample with the 5nm MgCaO oxide was further tested during and after heating on the hot chuck probe station. Figures 6-15 through 6-17 show the results of the IV and CV profiles of the 35nm Sc₂O₃/5nm MgCaO/u-GaN sample. As shown in the IV graph, after the initial decrease in breakdown voltage the sample under goes very small changes upon continued thermal cycling. The CV shows shifts in the flat band voltage indicating a change in fixed oxide charge. Figure 6-18 shows a separation of heated CV and cooled room temperature CV for better comparison purposes.

Forming gas (H₂+N₂) anneals were also performed on each sample in an RTA at 400°C for 30 seconds. The purpose was to determine if the anneal would improve the samples characteristics. Figures 6-18 to 6-20 present the CV behavior post forming gas anneal. Of the 3 samples the the 35nm Sc₂O₃/5nm MgCaO/u-GaN showed a decrease in fixed oxide charge based on the negative flat band voltage shift from the initial CV curve. But the calculated Dit for the forming gas anneals show just the opposite for the samples.
Dit indicates that the Sc$_2$O$_3$/MgO/GaN is the better sample but judging by the shape of the CV curve this shouldn’t hold to be true. This type of error can be attributed to errors in measurements or calculation of the interface state density. The Dit of each annealed sample is shown in figures 6-21 to 6-22.

6.4 Summary

In short radiation and annealing of the MOS diodes does degrade the samples, just as it does with the PL in the gallium nitride materials. It also causes a forward flatband voltage shift in the CV curves, indicating higher fixed oxide charge and a decrease in IV breakdown.
Table 6-1 Oxide Etchants and Condition.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Wet Etch Solution</th>
<th>Dry Etching Recipe</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sc$_2$O$_3$</td>
<td>-</td>
<td>ICP CL Etching</td>
<td>20mins</td>
</tr>
<tr>
<td>MgO</td>
<td>2% Phosphoric Acid/DI Water</td>
<td></td>
<td>15 to 17 secs.</td>
</tr>
<tr>
<td>MgCaO</td>
<td>2% Phosphoric Acid/DI Water</td>
<td></td>
<td>15 secs.</td>
</tr>
<tr>
<td>Sc$_2$O$_3$/MgCaO or MgO</td>
<td>2% Phosphoric Acid/DI Water</td>
<td>ICP CL Etching through Sc$_2$O$_3$</td>
<td>-</td>
</tr>
</tbody>
</table>
### Table 6-2 Dit Values at .4eV for Oxide/GaN Devices

<table>
<thead>
<tr>
<th></th>
<th>Dit @ .4eV</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>40nm Sc$_2$O$_3$/GaN</strong></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>5.955E11</td>
</tr>
<tr>
<td>10MeV Radiation</td>
<td>1.03E12</td>
</tr>
<tr>
<td>Forming Gas Anneal @ 400C</td>
<td>7.82E11</td>
</tr>
<tr>
<td>In-situ 100C</td>
<td>1.065E12</td>
</tr>
<tr>
<td>Post 100C</td>
<td>6.949E11</td>
</tr>
<tr>
<td><strong>2nm Sc$_2$O$_3$/40nm MgO/GaN</strong></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>2.33E11</td>
</tr>
<tr>
<td>10MeV Radiation</td>
<td>5.288E11</td>
</tr>
<tr>
<td>Forming Gas Anneal @ 400C</td>
<td>1.03E11</td>
</tr>
<tr>
<td>Post 100C</td>
<td>2.79E11</td>
</tr>
<tr>
<td>Post 200C</td>
<td>2.65E11</td>
</tr>
<tr>
<td>Post 300C</td>
<td>2.39E11</td>
</tr>
<tr>
<td><strong>35nm Sc$_2$O$_3$/4nm MgCaO/GaN</strong></td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>2.21E11</td>
</tr>
<tr>
<td>Forming Gas Anneal @ 400C</td>
<td>1.17E12</td>
</tr>
<tr>
<td>In-situ 100C</td>
<td>6.39E11</td>
</tr>
<tr>
<td>Post 100C</td>
<td>1.99E11</td>
</tr>
<tr>
<td>In-situ 200C</td>
<td>5.30E11</td>
</tr>
<tr>
<td>Post 200C</td>
<td>8.65E11</td>
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<tr>
<td>In-situ 300C</td>
<td>6.289E11</td>
</tr>
<tr>
<td>Post 300C</td>
<td>1.29E12</td>
</tr>
</tbody>
</table>
Figure 6-1 Finished diode mask design.
Figure 6-2 Ohmic IV plots taken from samples receiving various treatments.
Figure 6-3 Current density – Voltage behavior of Sc₂O₃/GaN after 10MeV irradiation.
Figure 6-4 Capacitance – Voltage plot of Sc$_2$O$_3$/GaN after 10MeV irradiation.
Figure 6-5 Current density – Voltage behavior of Sc$_2$O$_3$/MgO/GaN after 10MeV irradiation.
Figure 6-6 Capacitance –Voltage plot of Sc$_2$O$_3$/MgO/GaN after 10MeV irradiation.
Figure 6-7 Comparison of Current Density-Voltage results of different $\text{Sc}_2\text{O}_3/\text{MgCaO}/\text{GaN}$ thickness.
Figure 6-8 Capacitance-Voltage plot of Sc$_2$O$_3$/MgCaO/GaN after 10MeV irradiation.
Figure 6-9 Capacitance-Voltage plot of Sc$_2$O$_3$/MgCaO/GaN after 10MeV irradiation.
Figure 6-10 Dit of post 10MeV radiation. A) Sc$_2$O$_3$/GaN B) Sc$_2$O$_3$/MgO/GaN
Figure 6-11 Current Density – Voltage behavior of Sc$_2$O$_3$/GaN during and after thermal annealing.
Figure 6-12 Capacitance – Voltage plot of Sc$_2$O$_3$/GaN during and after heating.
Figure 6-13 Current Density – Voltage behavior of Sc$_2$O$_3$/MgO/GaN during and after heating.
Figure 6-14 Capacitance – Voltage breakdown of Sc$_2$O$_3$/MgO/GaN after heating.
Figure 6-15 Current Density –Voltage behavior of Sc$_2$O$_3$/5nm MgCaO/GaN during and after heating.
Figure 6-16 Capacitance-Voltage plot of Sc$_2$O$_3$/MgCaO/GaN during and after heating.
Figure 6-17 Capacitance-Voltage plot of Sc$_2$O$_3$/MgCaO/GaN during and after heating. A) CV of in-situ testing. B) CV of samples cooled to room temperature.
Figure 6-18 Capacitance –Voltage plot of Sc₂O₃/GaN after forming gas anneal at 400°C.
Figure 6-19 Capacitance–Voltage plot of Sc$_2$O$_3$/MgO/GaN after forming gas anneal at 400°C.
Figure 6-20 Capacitance-Voltage plot of Sc$_2$O$_3$/MgCaO/GaN after forming gas anneal at 400$^\circ$C.
Figure 6-21 Dit of Annealed Sc$_2$O$_3$/GaN. A) Probe station thermal anneals. B) Forming Gas anneal.
Figure 6-22 Dit of Annealed 2nm Sc$_2$O$_3$/40nm MgO/GaN. A) Probe station thermal anneals. B) Forming gas anneal.
Figure 6-23 Dit of Annealed 35nm Sc$_2$O$_3$/5 nm MgCaO/GaN A) Probe station thermal anneals. B) Forming gas anneal.
CHAPTER 7
SUMMARY AND DISCUSSION

7.1 GaN vs. Oxide/GaN

Based on the data collected, some general trends for the GaN can be noted. First is that radiation and annealing of the samples with and without oxides, decrease the PL luminescence intensity, except the activated P-GaN. Radiation of the activated P-GaN sample enhances the PL emission. Second, an increase of PL intensity at 200°C has shown to be unique to the UOE GaNs and activated P-GaN. After approximately 400°C for non-oxide samples, the drastic decline in peak intensity levels off and become less significant in change with higher temperatures. Where as the oxide/GaN samples shows an extended range of PL intensity increase than that of the non-oxide GaN samples.

The increase in PL intensity after irradiation and annealing of the n-GaN and activated P-GaN can possibly be explained by trap filling and emptying. In n-type GaN before radiation or annealing the traps are filled but do not contribute to PL emission. Once the sample is radiated the trap becomes empty becoming a non-radiative center and takes a long time to relaxed back to its filled neutral state. This long time relaxation is shown in figure 7-1, which presents a PL scan of Sc₂O₃/MgCaO/GaN seven months after 900°C anneal. The plot shows that over a period of time the luminescence of the GaN is recoverable to a certain point. Although it shows recovery, it does not recover to the initial PL intensity of 1.29. For the activated P-GaN, the traps are initially empty and non-radiative due to the location of the fermi level within the sample. After irradiation these traps are filled with carriers and the PL increases. Once the trap has had ample
time to relax it should return to its empty non-radiative state thus resulting in lower PL intensity. The empty traps compete for carriers therefore reducing the amount of carriers available for radiative transitions. Based on behavior of p-GaN, trap level is estimated to be at \( \sim 1.655\text{eV} \) above valence band which is in agreement with literature reports using low energy proton radiation.

The roughening of the oxide/GaN interface suggests that operating temperatures of any device fabricated from the MgO and MgCaO oxides maybe be limited. As with the non-oxide GaN samples the irradiation of the oxide samples still significantly decreases the bandedge emission and increases the defect emission. The radiation also causes roughening of the oxide/semiconductor interface and roughening at the surface. After one year, the samples show some relaxation of long life-time traps that degrade the PL emission, as was shown with the non-oxide GaN. The effect of annealing on the oxide also causes samples to roughening at the surface and interface. Radiation of the samples makes them more susceptible to annealing damage because of the already present defects.

### 7.2 MOS Devices

Radiation and annealing of the MOS diodes does degrade the samples, just as it does with the PL and XRR in the gallium nitride materials. It also causes a forward flatband voltage shift in the CV curves, indicating higher fixed oxide charge and a decrease in IV breakdown. Radiation and annealing empties traps in both the bulk GaN and the oxide, resulting in higher Dit and an increase in diode leakage. An increase in diode current leakage causes a decrease in forward IV breakdown. With the CV curves the forward flatband voltage shifts indicate an increase in fixed oxide charge and damage to the dielectric. Based on the study, the oxides do not protect the GaN from bulk damage from the radiation or annealing. What has been observed with the oxides is that the Mg-
containing oxides showed better resistance than Sc$_2$O$_3$ to radiation and thermal annealing. As stated in earlier sections, the purpose of the alternate dielectrics was to investigate one that allowed for elevated operating temperatures and stability harsh environments.
Figure 7-1 Recovery of Oxide/GaN post 900°C after 7 months.
LIST OF REFERENCES


BIOGRAPHICAL SKETCH

Kimberly Karlotta Allums was born April 27, 1978, in Houston, Texas. After completing high school in May of 1996, she went on to attend Prairie View A&M University. Just four years later, on May 13, 2000, she received her Bachelor of Science degree in electrical engineering.

She decided to continue her education at the University of Florida after being awarded a GEM Fellowship and an NSF Fellowship (AGEP). In the fall of 2000 Kimberly began pursuing a Master of Science degree in materials science and engineering with a specialization in electronic materials. After receiving her master’s degree in May 2002, she continued her graduate studies to complete a PhD in the same major.

Kimberly was involved in sports and various organizations throughout her high school and collegiate years. She played volleyball for Prairie View A&M University for four years. She has also worked for the University of Florida Intramural Sports program as a basketball official and sports supervisor for the last 3 years. She is currently a member of Zeta Phi Beta Sorority Inc., Epsilon Gamma Iota, Inc. (a co-ed engineering fraternity), and University of Florida’s Black Graduate Student Organization.

Upon graduation, her future goals include working in the research and development industry, a professorship and eventually owning her own businesses.