Advancement of Gallium Nitride PIN Diode and Development of Novel 3D+planar Core-shell Microstructures for Betavoltaic Device Technology

by

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Advancement of Gallium Nitride PIN Diode and Development of Novel 3D+planar Core-shell Microstructures for Betavoltaic Device Technology

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Dedicated to my best friend
and dearest love, Katie.
As well as the friends and family
that bring purpose to my life.
Acknowledgments

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Abstract

A betavoltaic (BV) device converts the kinetic energy from β-electrons emitted by radioactive decay into electricity, with output power in the <1 mW regime. Al$_x$Ga$_{1-x}$N is promising for use as the converter material in a BV device due to its wide bandgap, superior radiation tolerance, chemical inertness, and physical hardness. With the emergence of micro- and nano-technology, many low power systems require self-sustained power sources where replacement is difficult or impossible in harsh environments, for which BV batteries are a prime candidate. BV batteries have been investigated for several decades; however, the technology still lacks in efficient energy conversion and limited power output. The work reported here has addressed some of the challenges associated with, and improved upon, the GaN-based beta-energy converter, along with engineering new methods for device qualification under high energy irradiation and radioisotope sources. Uniquely, a converter structure has been designed, simulated, and implemented with the growth of novel GaN 3D+planar core-shell microstructures.

High performing GaN PIN planar diodes were fabricated by tailoring the fabrication process for BV specific needs such as a “beta-transparent” p-contact, low forward leakage by a KOH wet-etch passivation treatment and implementing a large area mesa for greater electron absorption. Electron energy irradiation was performed to mimic radioisotope exposure using an electron flood gun (4-16 keV) and a custom in-operando TEM setup (62 – 200 keV). Beam voltage and beam current dependence is reported for several devices. For the electron flood gun irradiation, the highest efficiency of energy conversion at 7% is reported for GaN PIN exposed to 16 keV electron irradiation. Direct measurement of these GaN PIN diodes under exposure to solid metal
radioisotope and liquid radioisotope solution is also reported, for both $^{63}$Ni and $^{147}$Pm isotopes. Moreover, methodology for device testing in custom enclosures was developed and executed.

A combined 3D (planar+core-shell) PIN device has been proposed as the optimal design for GaN application as a BV battery. This optimized structure layout leads to enhanced conversion (depletion) region volume compared to that in a planar device under equilibrium conditions. Monte Carlo simulation indicates there is a 3.75x increase in the amount of power absorbed in the GaN layers ($P_{\text{GaN/cm²}}$) at approximately half the activity density for a 3D structure with 4 μm mesa height compared to planar designs with 10 μm $^{63}$Ni thickness with a 5.8x improvement in energy transfer efficiency ($\eta_{\text{src}}$). Metalorganic chemical vapor deposition (MOCVD) growth of the proposed combined 3D+planar GaN PIN was achieved in both a fin and pillar geometry. High aspect ratio n-GaN seeds with controlled facet stabilization were obtained by optimization of the MOCVD growth conditions. An optimized growth condition is achieved where GaN semipolar sidewalls are replaced by m-plane nonpolar sidewalls characterized by their 90° angle with respect to the c-plane (substrate), which was selected as the seed condition for the combined 3D+planar core-shell structure. The fin combined 3D+planar PIN showed bipolar diode behavior with a threshold voltage of ~3 V.
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Chapter 1 Introduction

1.1 Nuclear Batteries and Applications, Motivation

Upon the discovery of spontaneous radioactivity over a century ago, the realization to harness this energy propelled the field of nuclear batteries forward. Nuclear battery technology has shown a steady advancement over the past century with a multitude of types being developed including betavoltaic, alphavoltaic, thermoelectric, thermophotoelectric, direct charge collection, scintillation intermediate and thermionic. The basis for all nuclear batteries is the conversion of radioisotope decay energy into electricity, with the lifetime of the battery aligning with the half-life of the radioisotope being used. Thermoelectric, thermophotoelectric, and thermionic battery types rely on the decay energy for heat production while betavoltaic, alphavoltaic and scintillation intermediate rely on the decay energy for the excitation of carriers within the material. The power produced and voltage load for these battery types vary, for example the thermoelectric batteries are capable of >100 W operation, while betavoltaic batteries are capable of operating at <1 mW.

The radioisotope thermoelectric generator (RTG) has shown the most success. The National Aeronautics and Space Administration (NASA) has deployed over 46 RTGs on 31 space missions, with the first being launched into Earth orbit in 1961. This battery type relies on the Seebeck effect, where decay energy from a radioisotope is converted to heat inside of a material. This heat is converted to electricity as an electromotive force is formed between a temperature gradient of two different metals or semiconductors. The most commonly used radioactive source for this battery type is plutonium-238 (Pu-238) which emits alpha particles with an average energy of 5.6 MeV, along with some amount of gamma rays and neutron radiation. The Multi-Mission
Radioisotope Thermoelectric Generator (MMRTG) utilizes Pu-238 and is NASA's most advanced RTG, with a beginning-of-life specific power of 2.8 W/kg and system efficiency of 6% under a 30 V load voltage. The MMRTG emits 110 W electrical power which decreases nominally to 72 W after 17 years (end-of-design-life), along with 2,000 W of thermal power.\(^2\) The Mars Curiosity rover, Mars Perseverance rover, and eventual Dragonfly rotorcraft are all powered by the MMRTG system.

In contrast to the high energy RTG nuclear batteries that rely on the transfer of energy via heat, the lower energy betavoltaic (BV) battery directly converts the kinetic energy of the decay particle into current. With the emergence of micro- and nano- technology, many low power systems require self-sustained power sources, for which BV batteries are a prime candidate. For space missions, applications such as electric field detectors, microphones, magnetometer, and sensor-on-a-chip operate at <1 W, nearing suitability for BV cells.\(^3\) In the medical field, cardiac pacemakers are considered a possible market for BV batteries, with lithium ion batteries currently dominating this market.\(^1,5,6\) Low power lithium ion batteries used for this purpose tend to require replacement after 10-15 years, where surgery is necessary to access the battery causing additional discomfort to the patient. A BV cell, with proper radiation shielding, has the potential to power the pacemaker for the lifetime of the patient without any additional surgical procedure necessary to replace the battery. Micro- and nano- electromechanical systems (MEMS, NEMS) have increased in prominence over the past decade, some of which could benefit from a self-sustained power source with long lifetime. The footprint of such devices is becoming smaller and with it the power requirement is decreasing.\(^7\) However, the power supply is not following suit, as macro-scale battery energy decays relatively quickly and requires replacement which can be time consuming.
and costly. This calls for an integrated battery technology that can provide low levels of power for a long duration, for which the betavoltaic battery is an ideal choice. BV batteries have been investigated for several decades; however, the technology still lacks in efficient energy conversion and limited power output. Low bandgap semiconductor materials, such as Si, show a radiation-induced material degradation during operation and limited efficiency. In recent years, the wideband gap semiconductors (GaN, SiC, diamond) have shown continuous improvement due to epitaxial growth quality and processing techniques. Gallium Nitride (GaN) is promising for use as the converter material in a BV device due to its wide bandgap, superior radiation tolerance, chemical inertness, and physical hardness. The fundamentals of β-decay, operating principle of BV batteries, and a detailed look into the state-of-the-art in the field will be discussed in the next section.

The results reported in this thesis focus on the utilization of the (Al)GaN material system for the advancement of BV technology, with development of a novel 3D device layout and improvements made to the traditional 2D planar device. A thorough investigation of the challenges associated with this transition are also introduced, most specifically related to crystal growth. Extensive growth of novel 3D PIN core-shell GaN structures using metal organic chemical vapor deposition (MOCVD) has been performed. Planar and 3D devices have been grown, fabricated, and tested under monochromatic irradiation ranging from 3 keV – 200 keV, metal foil radioisotope, along with liquid-form radioisotope (\(^{63}\text{NiCl}, \ ^{147}\text{PmCl}\)). A more detailed outline of the results is provided at the end of the introductory chapter.
1.2 Beta Decay Fundamentals

The main benefit of betavoltaic batteries is the extremely high specific energy density which is in the range of $10 - 10000 \, W \cdot h/kg$. This is due to the energy stored in the beta-emitting material which is on the order of $10^8 \, J/gm$, that emits for many years. The rate at which a radioisotope decays is given by the radioactive decay law:

$$N = N_0 e^{-\lambda t}$$  \hspace{1cm} (1.1)

Where $N$ is the current number of radioactive atoms at time $t$, $N_0$ is the number of radioactive atoms at $t = 0$, and $\lambda$ is a radioactive decay constant. The time at which half of the nuclei decay is referred to as the half-life ($t_{1/2}$) of the material, an important characteristic of a given radioisotope.

$$\frac{N_0}{2} = N_0 e^{-\lambda t_{1/2}}$$  
$$t_{1/2} = \frac{\ln 2}{\lambda}$$  \hspace{1cm} (1.2)

Another important characteristic of a radioisotope is the activity, $A$. This is a measure of the number of nuclei decaying for a given unit time, measured in units of Becquerel (Bq) or Curie (Ci) where $1 \, Ci$ is equivalent to $3.7 \times 10^7 \, Bq$.

$$A = -\frac{dN}{dt} = \lambda N$$  \hspace{1cm} (1.3)

There are three types of beta decay: negative beta decay ($\beta^-$- particle) in which a neutron converts into a proton creating a high energy electron and antineutrino, positive beta decay ($\beta^+$-
particle) in which a proton converts into a neutron creating a positron and neutrino, and orbital electron capture (e) where a positron and electron convert to a neutron. The atomic number (Z) and mass number (N) are changed by one unit such that the mass number (A) remains constant. Unlike alpha decay, where the particle has a discrete energy, beta decay results in a continuous spectrum of energies up to the expected value based on nuclear mass difference, with a neutrino carrying away the missing energy. For negative beta decay, the reaction is given as:

$$A_N^Z \rightarrow A'_{N-1}^{Z+1} + e^- + \bar{\nu}$$

To calculate the beta decay spectrum, Fermi’s Theory of Beta Decay is utilized. Important considerations in the development of this theory were that the electron and neutrino are created in the process and do not exist prior to decay, the particles must be treated relativistically, and a spectrum of energies must result. From Fermi’s theory, a spectrum shape for momentum ($p$) and kinetic energy ($T_e$) can be defined, as:

$$N(p) = \frac{C}{c^2} p^2 [Q - \sqrt{p^2 c^2 + m_e^2 c^4 + m_e c^2}]$$

$$N(T_e) = \frac{C}{c^8} (T_e^2 + 2T_e m_e c^2)^{1/2} (Q - T_e)^2 (T_e + m_e c^2)$$

Deviations exist between the above theoretical spectrum and experimental measurement of the beta energy probability spectrum. Two additional terms are required, including the Fermi function, $F(Z', T_e)$, which accounts for the effect of the nuclear Coulomb field on the electron wave function, and the nuclear matrix element, $M_{fi}$, which accounts for effects of certain initial and final nuclear states.
Beta-emitting radioisotopes are either naturally occurring or can be man-made. Tritium ($^3$H) is a cosmogenic radioisotope, meaning that it is produced by the interaction of cosmic-rays and the Earth’s soil or atmosphere. This is the cheapest and most abundant beta-emitting isotope available, with a relatively low emission spectrum at a maximum of 18 keV beta kinetic energy. The other common beta-emitting sources are $^{63}$Ni and $^{147}$Pm which can be produced artificially by neutron capture or by use of a particle accelerator. Referring to Table 1, the characteristics of the common radioisotope sources are compared. The half-life of $^{63}$Ni at 92 years enables the longest operation time of the three sources, but the cost per Ci is substantially larger than $^3$H due to availability and cost of production. The $^{147}$Pm radioisotope has the shortest half-life but is the most energetic of the group, with an average $\beta$-energy of 62 keV and maximum of 225 keV. Figure 1-1 shows the characteristic emission probability spectrum for $\beta$-decay for $^3$H and $^{63}$Ni, which can be derived by eq. 1-6.

<table>
<thead>
<tr>
<th>Isotope</th>
<th>Average $\beta$ energy (keV)</th>
<th>Maximum $\beta$ energy (keV)</th>
<th>Half-life (years)</th>
<th>Approx. Cost (per Ci)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tritium $^3$H</td>
<td>5.7</td>
<td>18</td>
<td>12.3</td>
<td>$3.50</td>
</tr>
<tr>
<td>Nickel-$^{63}$Ni</td>
<td>16</td>
<td>67</td>
<td>92</td>
<td>$4000</td>
</tr>
<tr>
<td>Promethium-$^{147}$Pm</td>
<td>62</td>
<td>225</td>
<td>2.6</td>
<td>-</td>
</tr>
</tbody>
</table>
Figure 1-1: Beta-decay energy spectrum for (left) Tritium and (right) Nickel-63

1.3 Betavoltaic Batteries

1.3.1 Background

P. Rappaport at Radio Corporation of America (RCA) was the first to realize a BV battery design in 1953. The electron-voltaic effect was measured from the bombardment of germanium or silicon p-n junction with beta particles from a 50-millicurie Sr$^{80}$-Y$^{90}$ source. A conversion efficiency of 0.4% was obtained, with factors such as bulk and surface recombination, leakage, and energy loss from phonons leading to the low efficiency. Degradation of the material was observed due to the high kinetic energy of the beta particles emitted from the Sr$^{80}$-Y$^{90}$ source.

A BV battery can be in the form of a direct-conversion or indirect-conversion architecture. In both architectures, the converter material is typically a semiconductor PIN/PN or Schottky diode. A direct conversion device generates usable current through the conversion of the kinetic energy of a beta particle into electron-hole-pairs (EHPs). The internal electric field within the device separates the EHPs and accelerates holes towards the p-layer and electrons towards the n-
layer, when connected to a load this creates a flow of electricity. A single beta particle, in this case, can be responsible for generating thousands of EHPs along its path length as it traverses the material, until the energy is less than that required for EHP generation. In the case of indirect-conversion devices, there is an intermediary medium in the form of a radioluminescent material. The beta particle impinges onto the radioluminescent material and transfers its kinetic energy to a single photon with equivalent energy. The photon is then directed onto the converter material which can generate electricity. Both BV types are illustrated in Figure 1-2. The efficiency of the indirect conversion device is typically lower than that of the direct-conversion architecture given the additional conversion step. The indirect conversion type also requires a close matching of the emission wavelength of the radioluminescent material and the spectral response curve of the converter.13

![Figure 1-2: (left) Direct conversion BV battery connected to external load, (right) Indirect conversion BV battery with phosphor radioluminescent material](image)

**1.3.2 Device Fundamentals**

The operating principle of a BV cell is analogous to a photovoltaic (PV) cell, where incident radiation excites EHPs within the semiconductor bulk, as introduced in the previous
section. A $\beta$-induced short-circuit current ($I_{sc}$) is generated from the separation of EHPs which are formed through impact ionization within the semiconductor bulk. The total current in the device ($I$) can be written as the sum of the $\beta$-induced current ($I_{sc}$) and the diode current density ($I_D$) which is a voltage-dependent characteristic of the diode, shown in eq. 1-7. If considering an ideal diode and the dark saturation current ($I_o$), the open-circuit voltage ($V_{oc}$) can be determined by setting the current to zero, shown in eq. 1-8.

$$I = I_{sc} - I_D(V) = I_{sc} - I_o \left( \frac{qV}{kT} - 1 \right)$$  \hspace{1cm} \text{1-7}$$

$$I = 0 \rightarrow V_{oc} = \frac{kT}{q} \ln \left( \frac{I_{sc}}{I_o} \right)$$  \hspace{1cm} \text{1-8}$$

The dark saturation current, $I_o$, is a combination of both a diffusion component, $I_{diff}$, from the quasi-neutral regions of the device and the generation recombination current, $I_{g-r}$, as shown in eq. 1-9. Here $D_p$ and $D_n$ are the hole and electron diffusion coefficients, $L_p$ and $L_n$ are the hole and electron diffusion lengths, $p_{n0}$ and $n_{p0}$ are the hole and electron concentrations, respectively, $\tau_c$ is the carrier lifetime, $n_i$ the intrinsic carrier concentration, and $W_d$ is the depletion width. In a real device the effect of series and shunt resistance must also be considered due in part to defects within the material and contact resistance. Therefore, eq. 1-7 can be modified to consider terms for the non-ideal case, including the ideality factor ($n$), the effective shunt resistance ($R_{sh}$) and effective series resistance ($R_s$), shown in eq. 1-10.

$$I_o = I_{diff} + I_{g-r} = \left[ \frac{qD_p p_{n0}}{L_p} + \frac{qD_n n_{p0}}{L_n} \right] + \frac{q n_i W_d}{\tau_c}$$  \hspace{1cm} \text{1-9}$$
\[ I = I_{sc} - I_0 \left( e^{\frac{q(V+IR_s)}{nkT}} - 1 \right) - \frac{V + IR_s}{R_{sh}} \]

Figure 1-3: Equivalent circuit diagram for BV cell / solar cell

As pointed out by Olsen, L.C.,\(^\text{16}\) the series resistance can be relatively large for BVs due to the low \( I_{sc} \) inherit to the device (\( \mu \text{A/cm}^2 \)), in contrast to photovoltaics where the \( I_{sc} \) is much higher (\( \text{mA/cm}^2 \)). It is also necessary to maximize the shunt resistance to minimize the shunt conductance, similar to a photovoltaic. As seen in the circuit diagram in Figure 1-3, the shunt resistance (\( R_{sh} \)) is parallel to the diode, if low enough this will allow generated current to flow through resulting in power loss. This effect is especially severe for the low power operating regime for BV device. The maximum power point (MPP) of the device can be calculated by eq. 1-11, where the fill factor (FF) is a measure of the “squareness” of the IV curve.

\[ MPP = I_{sc}V_{oc} \cdot FF \]

An effort into improving the performance of BV batteries through the use of different material systems, device designs, and radioisotope sources has been undertaken since the first battery was demonstrated. The efficiency of the BV cell (\( \eta \)) is the product of the radioisotope efficiency (\( \eta_r \)) and converter (or semiconductor) efficiency (\( \eta_s \)). The efficiency of the radioisotope source is determined by the total number of betas available within the source to the betas that are
incident to the converter. The converter efficiency is defined by the ratio of total beta kinetic energy to the electrical energy produced in the form of EHPs. In theory, the efficiency of conversion can be improved by using a semiconductor converter of higher bandgap energy.\textsuperscript{16} Referring to Figure 1-4, converter efficiency reaches a maximum of approximately 30% for the wide-bandgap (Al)GaN material system, with bandgap ranging from 3.4 eV – 6.2 eV. This assumes that the semiconductor converter is an ideal homojunction. This bandgap ($E_g$) dependence of the theoretical limit in efficiency ($\eta_s$) arises from its relation with pair creation energy ($\varepsilon$) shown in eq. 1-12, where $\varepsilon$ is the EHP creation energy (also known as the effective ionization energy) and is the sum of the bandgap, thermalization-loss of the secondary electrons created ($E_K$), and optical phonon-loss of the original high energy particle ($E_R$) determined phenomenologically.\textsuperscript{17} This relation is shown in eq. 1-13. For the derivation of $E_K$, given in eq. 1-14, all electrons are assumed to be evenly distributed in k-space and thermalization occurs when the electron energy is less than the impact ionization threshold ($E_I$).\textsuperscript{16–18} The impact ionization threshold energy is widely approximated to be equivalent to $\frac{3}{2}E_g$, therefore leading to $E_K$ equaling $\frac{9}{5}E_g$. This model overall assumes simple parabolic bands with effective masses of electrons and holes being equal, and that the energy spectrum of excited electrons are uniform in probability in the Brillouin zone as previously mentioned.\textsuperscript{17} In 4H-SiC devices it has been shown that the EHP creation energy could be much less than that predicted by this model and other experimental measurements.\textsuperscript{18} The current multiplication ($M = I_{sc}/I_{bulk}$) within a 4H-SiC PN junction was measured using electron irradiation spanning 5 – 30 keV within an SEM. The multiplication increased linearly from 6-10 kV, and by finding the maximum slope the pair creation energy was measured to be $\varepsilon=5.05$ eV. Any uncertainties in the multiplication value due to loss within metal contacts were removed in
this study, as the measurement was made through irradiation of the bare semiconductor. The authors attribute this to be the reason for the difference in value compared to other experimental reports of pair creation energy (5.05 eV compared to 8.4 eV). In addition, Monte Carlo calculations of impact ionization in SiC have shown an ionization threshold energy ($E_T$) that is less than the bandgap energy, which can be linked to band-to-band tunneling.

\[ \eta_s \leq \frac{E_g}{\varepsilon} \]

\[ \varepsilon = E_g + \langle E_K \rangle + \langle E_R \rangle = \frac{14}{5} E_g + \langle E_R \rangle \]

\[ \langle E_K \rangle = 2 \times \frac{\int_0^{k_T} E_K \cdot 4\pi k^2 dk}{\int_0^{k_T} 4\pi k^2 dk} = 2 \times \frac{\int_0^{k_T} \frac{\hbar^2 k^2}{2m} \cdot 4\pi k^2 dk}{\int_0^{k_T} 4\pi k^2 dk} = \frac{9}{5} E_g \]
Along with higher theoretical conversion efficiency, the materials with high bandgap are more resistant to radiation damage. The higher atomic displacement energy for the wide band gap materials relative to lower bandgap materials means they are more resistant to point defect formation. The exact radiation response of a given material depends on the dose and energy of the radiation, along with the carrier concentration, dislocation density, and impurity levels within the material.

1.3.3 State-of-the-art

Several materials have been tested in a BV configuration including but not limited to, Si, SiC, diamond, ZnO, TiO$_2$, GaAs, and GaN. With the most mature material development and advanced processing capabilities available, silicon is the most prevalent in the early published work on BVs in the 1960s and continues to be studied today. Crystalline, amorphous, porous, and micro-channeled Si have all been tested with varying degrees of success.\textsuperscript{20-27} Historically, Si BVs have suffered from low efficiency and low power output, for both planar configurations and 3D configurations. They also suffer from temperature effect, where the resistance within a silicon device can vary by greater than 200 \% in the temperature range of 25 °C - 150 °C which is not suitable for harsh environment applications.\textsuperscript{28} When comparing performance of a Si p-n junction and Au/Si Schottky diode with 296 MBq $^{63}$Ni and 1538 MBq $^{3}$H radioactive source, the efficiency was <0.3\% for all combinations tested.\textsuperscript{26} The Au/Si Schottky diode shows higher $I_{sc}$ and lower $V_{oc}$ than the Si p-n diode, where the higher $I_{sc}$ in the Schottky diode is attributed to lower loss of $\beta$-particles in the thinner top-layer as compared to the p-n diode. Given that the Au/Si Schottky diode is a majority carrier device, it has a higher $I_{o}$, about 1-2x higher than that of the Si p-n diode.
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Referring to eq. 1-8, $V_{oc}$ would therefore be expected to be lower in the majority carrier device, which is backed by the experimental observation in this work. Hydrogenated amorphous Si PIN diode has also been studied. Here the $^3$H radioisotope is located within the i-layer of the material through direct bonding during synthesis, which in theory greatly increases the likelihood of capturing emitted β-particles. However, degradation of the I-V characteristics is observed due to the dangling bonds created from the $^3$H decay, which can be mitigated by decreasing the layer thickness of the tritiated i-layer. Additional studies for Si based-BV device prefer a 3D-layout over the traditional planar design, whether through electrochemically-etch porous Si or highly ordered 3D features formed by post-growth processing. A 3D macro-porous Si p-n diode has been shown to lead to an order of magnitude increase in efficiency (0.22%) over a planar p-n under exposure of tritium gas. This increased efficiency is likely due to the increased surface area available in the 3D pore. More recently, a high-aspect ratio (430 µm:17µm) 3D ridged Si PN diode has shown a 200 nW max power produced using a 29.5 mCi $^{147}$Pm source, with the $I_{sc}$ measuring near 77% of the theoretical maximum. Only partial filling of the ridges was achieved using the PmCl$_3$-6(H$_2$O) solution and a large increase in output power is expected, up to 3.8 mW/cm$^3$ by simulation, if full-filling of the ridges is achieved. The simulation work contained here and published by the author of this thesis was used as the basis for the prediction in the recent report on 3D ridged Si PN diode.

The wideband gap SiC BV has shown tremendous progress in recent years, followed closely by GaN and diamond. The 4H polymorph of SiC is the most commonly used in BVs due to the availability of high-quality substrates leading to low leakage current devices. The highest reported device efficiency of 18.4% has been achieved by Widetronix, Inc. with a 4H-SiC PN junction
irradiated by an external 14.97 mCi/cm\(^2\) titanium titride (TiH\(_3\)) foil.\(^{30}\) The \(V_{oc}\) and \(I_{sc}\) for this device were 2.09 V and 75.47 nA/cm\(^2\), respectively, with a fill factor of 0.86. The main mechanism for the lower than simulated maximum (23%) efficiency being in large part due to surface recombination, which is a main source of loss for wide bandgap BV devices. The authors suggest the use of a highly doped and thin PN junction is ideal to mitigate the effects of surface recombination.\(^{30}\) In previous work, this group was also able to achieve a 4H-SiC BV cell utilizing a similar PN planar structure and irradiated by a 4 mCi/cm\(^2\) \(^{63}\)Ni source. The power efficiency was 6%, with a \(V_{oc}\) and \(I_{sc}\) of 0.72 V and 16.8 nA/cm\(^2\). After a ten-day period, there was no degradation in the electrical output indicating that no radiation damage has occurred. The low efficiency was again attributed to surface recombination and also edge recombination. A surface passivation technique is proposed to improve the efficiency of the device. Simulation optimization for a \(^{147}\)Pm SiC PIN device has also been performed using Monte Carlo techniques.\(^{31}\) The conversion efficiency for the PIN cell is shown to be 3.74% with an i-layer thickness of 20 µm and doping density of \(5\times10^{14}\) cm\(^{-3}\). A graded N-layer is proposed and shown to increase the efficiency of to 4.58%.

Diamond possesses a large bandgap (5.5 eV) and is a notably radiation resistant material. Diamond Schottky diodes have shown success when used for \(\beta\)-particle energy conversion, specifically in a multi-planar stacked device layout.\(^{32,33}\) Using an ion-implantation assisted lift-off method, individual diamond Schottky cells can be thinned and stacked in parallel between 24% radioactive \(^{63}\)Ni-Ni foil. By connecting 200 individual cells an output power of 0.93 µW at 0.9V is obtained, which is expected to increase when using a higher enriched \(^{63}\)Ni-Ni foil.\(^{32}\) It is difficult to obtain n-type doping in diamond, which has held back the progress of diamond PN-diode based
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BV. The equilibrium solubility of dopants in diamond has been shown to be low. Phosphorus is the best candidate with a deep donor level (0.6 eV below the conduction band) which leads to limited conduction electrons at room temperature. Only recently has the first diamond PIN-diode BV been demonstrated, showing leading values in both efficiency and realized V_{oc} at 28% and 4.26 V, respectively. For the doping of the p-type and n-type layers, two separate microwave plasma-assisted chemical vapor deposition (MPCVD) systems are required. It is important to note however that these values were for irradiation of a monochromatic electron beam at 15 kV, which is known to lead to different electrical output characteristics than for full spectrum irradiation from a radioisotope.

1.4 III-Nitride BV

1.4.1 III-Nitride Background

The III-Nitride material system (Al_{x}In_{y}Ga_{1-x-y}N) shows great promise as the converter material within a BV cell, particularly gallium nitride (GaN) and its ternary alloy aluminum gallium nitride (Al_{x}Ga_{1-x}N), with direct band gap in the energy range of 3.4 eV – 6.4 eV. The ternary alloy indium gallium nitride (InGaN) is an important material within this system that has enabled light emitting diode (LED) for solid state lighting application, which has had a profound impact to modern life. Due to its lower band gap (1.7 eV – 3.4 eV) and complications in growing high-quality thick material with high n-type or p-type doping, InGaN has yet to be considered for BV application. The III-Nitrides system possesses many desirable material properties including a wide and tunable bandgap, physical and radiation hardiness, chemical inertness, high electric field breakdown and relatively high mobility. III-Nitrides are best known for use in photon detection, power electronics, high frequency switching applications, and other advanced optoelectronic
devices. A table comparing III-Nitride properties to the other common semiconductor materials is shown in Figure 1-5.

<table>
<thead>
<tr>
<th>Property</th>
<th>GaN AlGaN/GaN*</th>
<th>Si</th>
<th>4H-SiC</th>
<th>GaAs AlGaAs/InGaAs**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bandgap energy (eV)</td>
<td>3.4</td>
<td>1.1</td>
<td>3.26</td>
<td>1.43</td>
</tr>
<tr>
<td>Electric breakdown field (MV/cm)</td>
<td>3.3</td>
<td>0.3</td>
<td>2.0</td>
<td>0.4</td>
</tr>
<tr>
<td>Relative dielectric constant</td>
<td>9.0</td>
<td>11.8</td>
<td>10.0</td>
<td>12.8</td>
</tr>
<tr>
<td>Saturated (peak) drift velocity ($\times 10^7$ cm/s)</td>
<td>2.5 (2.7)</td>
<td>1.0 (1.0)</td>
<td>2.0 (2.0)</td>
<td>1.0 (2.1)</td>
</tr>
<tr>
<td>Electron mobility (cm$^2$/Vs)</td>
<td>900 &gt;2000*</td>
<td>1350</td>
<td>700</td>
<td>8500 10000**</td>
</tr>
<tr>
<td>Thermal conductivity (W/cm.K)</td>
<td>1.3</td>
<td>1.5</td>
<td>4.5</td>
<td>0.5</td>
</tr>
</tbody>
</table>

*Figure 1-5: Comparison of electrical properties for common semiconductor materials.*

The III-Nitrides are thermodynamically stable in the wurtzite hexagonal crystal structure, though it is also possible to grow GaN in the zinc-blende and rock salt structure. The basal plane (0001) is the most common growth orientation for GaN where the surface can be terminated with Ga, (0001) Ga-polarity, or N (000-1) N-polarity as shown in Figure 1-6. These orientations lack center of inversion symmetry and are typically grown on lattice mismatched substrate, possessing spontaneous and piezoelectric polarizations. These polarization effects can alter the predicted device behavior and need to be considered in the development process. The piezoelectric coefficients in III-Nitrides are near an order of magnitude larger than other III-V materials. In devices such as high electron mobility transistors (HEMTs) the strain induced by AlGaN/GaN heterostructure leads to a high carrier density 2D electron gas (2DEG) at the interface. This 2DEG allows for superior device performance such as high mobility, high switching speeds, and low on-
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Nonpolar planes are also present within the crystal structure, namely the a-plane (11-20) and m-plane (1-100), which can be seen in Figure 1-7. These nonpolar orientations lack internal polarization, therefore contain no polarization induced electric field. This is a promising attribute for LEDs, where the internal quantum efficiency (IQE) is improved when no such fields are present, and high degree of overlap of the electron and hole wavefunctions occurs minimizing the quantum confined stark effect (QCSE). When a nonpolar orientation is not being utilized, the QCSE can also be mitigated by reducing the strain-induced polarization by growing films with similar lattice constant. For example, by employing a p-InGaN layer as the hole injection layer, rather than p-GaN within an LED structure, the QCSE is reduced due to better lattice matching with the multi quantum well (MQW) layers which leads to an increase in the hole injection current.

![Figure 1-6: Wurtzite crystal structure of III-Nitride, oriented in the basal c-plane](https://www.st.com/content/st_com/en/about/innovation--technology/GaN.html)
1.4.2 State-of-the-art

There are reports demonstrating PIN and Schottky GaN-based BV,\textsuperscript{42-45} along with detailed simulation and optimization.\textsuperscript{29,46-50} When joined with a 5 mCi/cm$^2$ - $^{147}$Pm plate, a $V_{oc}$ of 1.07 V, $I_{sc}$ of 0.554 nA, and FF of 24\% was achieved for a GaN PIN converter.\textsuperscript{45} The poor performance is attributed to the low level of radioactive material deposition within the converter and lower quality epitaxial layer. For a 3 mCi/cm$^2$ $^{63}$Ni loaded GaN Schottky diode, a $V_{oc}$ of 0.1 V, and $I_{sc}$ of 1.2 nA/cm$^2$ was achieved.\textsuperscript{51} This low-level performance can be associated with the limited active region from the metal-semiconductor (Ni/Au - GaN) junction which has a barrier height of 0.64 eV.\textsuperscript{51} A 1 cm$^2$ GaN PIN device in close proximity to a $^{63}$Ni source (3.27 µW energy emitted), resulted in an output power of 0.52 nW.\textsuperscript{43} This appears to be the best reported output power for a GaN / $^{63}$Ni cell. In the absence of the availability of a radioisotope, a common method for
evaluating the BV performance of a given converter is measurement under monochromatic electron irradiation within a scanning electron microscope (SEM), or electron flood gun. This can simulate the energies and currents found within the β-decay spectrum for the common isotopes used. Reports of such evaluation of GaN converters are in the literature, including work presented in this thesis utilizing a novel transmission electron microscope (TEM) method for measurements in-operando. Khan et al. estimate output powers of 53 nW and 750 nW with overall efficiencies of 0.96% and 4.4% at the average energy emission of $^3$H (5.6 keV) and $^{63}$Ni (17 keV), respectively, for a GaN PIN device. Further improvement to the planar GaN PIN device used in that work is presented in this thesis, with an increase in the resulting output power and efficiency under identical irradiation conditions. Also in this thesis, a large-area planar GaN PIN (0.04 cm$^2$, 17.8 nA/cm$^2$ at −5 V) device is demonstrated with an MPP of 2.45 μW/cm$^2$ at 62 kV irradiation voltage and 5 nA/cm$^2$ input beam current. This MPP is increased up to 48.2 μW/cm$^2$ at 62 keV and 177 nA/cm$^2$ beam current.

1.4.3 Motivation for 3D Structure

Although there has been progress with GaN-based BV cells, there are many limitations when using the traditional planar material. A large leakage current is typically observed in such epitaxial films due to the high dislocation defects ($10^8 – 10^{10}$ cm$^{-3}$) present from growth on foreign substrate such as sapphire and Si. The defect density can be lessened by epitaxy on bulk GaN substrates ($10^5$ cm$^{-3}$) which would lead to improved leakage current, but the cost of such substrates is currently extremely high (>$3000 per 2-inch substrate). Although material quality is a major contributor, the low conversion efficiencies are in part due in large part to the lack of device design
optimization and processing optimization to a lesser degree. In the case of Schottky diodes, significant beta energy is absorbed and lost in the inactive metal layer. Similarly, for GaN PIN diodes a lack of layer thickness optimization can lead to a large “dead zone” in the semiconductor where no electric field is present. Both effects lead to a reduced capture efficiency of incident β-particles. In order to create an efficient nuclear battery, it is vital that the associated energy dispersion of emitted particles matches well with the thickness and density of the convertor. In particular, for a PIN junction-based device design, the bulk of energy absorption will need to occur in the depletion region to allow the capture of generated carriers. Outside of the depletion region, generated minority carriers rely on diffusion to reach the depletion layer to be swept by the electric field and collected as current. A low $\eta_s$ is commonly achieved due to generation of EHPs outside of the depletion region, which are not separated by the built-in potential and are lost to recombination. Also, low $\eta_r$ is commonly the result of the limited source area that is in contact with the converter. In planar configurations, the assumption is made that 50% of β-particles are incident to the opposite direction of the converter, as shown in Figure 1-8.

*Figure 1-8: Planar PN junction joined with beta-emitter. 50% of beta-particles because of multidirectional emission*
Implementation of a 3D microstructure configuration is expected to lead to an increased surface area in contact with the radioisotope, and hence to enhanced capture of \( \beta \)-particles and conversion efficiency. The limit of 50% incident betas that is found for the planar layout is removed as conformal deposition of the source around a 3D structure can occur. Combined with that, 3D structures grown using a bottom-up approach will result in significant reduction in surface and bulk defect density due to enhanced lateral growth and as-grown device surface.\textsuperscript{54} This would in turn result in further decrease in leakage current.\textsuperscript{55} PIN core-shell GaN structures have previously been reported for application in LEDs.\textsuperscript{56,57} A more detailed discussion of the gains from a 3D structure over a planar structure, along with simulation and experimental results, is presented elsewhere in this thesis.

1.5 Thesis Organization

Chapter 1 provides the reader with a foundational understanding of nuclear batteries with a focus on BV devices and their place in the market. The state-of-the-art in device design and material used for BV is also introduced while providing motivation for utilizing the III-Nitride system for the next generation of BV. Chapter 2 contains the simulation work for optimization of the depletion region of both planar and combined 3D+planar GaN structures utilizing physics based TCAD simulation. This is paired with Monte Carlo simulation to quantify the energy deposition in the material with depth dependence. The optimized combined 3D+planar structure layout and PIN-layer dimensions are derived from this work. Chapter 3 discusses the epitaxial growth method used to grow the III-Nitride layers and structures used in this thesis, along with providing information on the characterization techniques used throughout this work. Chapter 4
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presents the results of electron irradiation (4 keV – 200 keV) of GaN PIN diodes providing both a beam voltage and beam current dependence. The development and operation of a custom high-energy irradiation setup is also described. Chapter 5 deals with the direct coupling of GaN PIN devices with $^{63}$Ni and $^{147}$Pm radioactive material in both metal and liquid form in custom experimental environments. Alpha irradiation of GaN PIN and the analysis of resulting damage is also provided. Chapter 6 details the fundamentals of selective area growth (SAG) and the growth optimizations of novel core-shell 3D+planar microstructures. A full 3D+planar PIN structure is achieved in both a fin and pillar architecture. Chapter 7 goes on to discuss the device fabrication and electrical characterization of these structures, while pointing to the challenges to address for future iterations.

## 1.6 References


Chapter 1


Chapter 1


Chapter 1


Chapter 1


Chapter 2  Device Simulation and Optimization

Prior to the experimental demonstration of planar and 3D GaN PIN, simulations have been performed to optimize the device structure for use as a BV converter. Physics-based technology computer aided design (TCAD) simulations allow for tuning the PIN layer thicknesses for maximum depletion region volume. Monte Carlo simulations of β-particle trajectory and absorption can provide a means to quantify the amount of energy absorbed in the converter (GaN) material for a given radioisotope. A comparison between different dimensions in multiple structure configurations (planar vs. 3D) can be made to enhance the amount of energy absorbed and carriers collected. The combination of simulation methods provides a feedback loop with the experimental work to intentionally enhance BV performance.

Synopsys Sentaurus TCAD simulations were performed to fine-tune the device layer thicknesses and model the depletion region layout of the proposed device structure for application with a $^{63}$Ni metal source. Both planar PN and PIN GaN diodes were modeled to maximize the depletion region area. Monte Carlo N-particle Extended (MCNPX) and CASINO2 have been utilized to simulate the energy absorption profile of incident β-particles within planar and 3D PIN device layouts. The proposed optimal structure is a combined 3D core-shell PIN + planar PIN (hereafter referred to as combined 3D PIN) that was described in Chapter 1.4.3. Simulations of monoenergetic electron penetration using CASINO2 and a spectrum of electron energies using
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MCNPX are performed for this structure. When joining a radioisotope source to this combined 3D PIN structure, conformal deposition of $^{63}$Ni metal film onto the structure will be difficult due to the high aspect ratio of the structures which will require a considerably thick layer. The high-density metal layer will suffer from energy loss due to self-absorption, with the effect becoming more prominent as the thickness increases. A liquid solution form ($^{63}$NiCl$_2$) of the radioisotope has a lower degree of self-absorption and can achieve a higher internal activity than the metal form. In the liquid-solution the radioisotope can, in theory, completely fill the gaps between GaN mesas. As such, a $^{63}$NiCl$_2$ liquid form solution is modeled, with a density of 1.05 g/cc that is interfaced with the GaN surface (6.15 g/cc). The simulation results are summarized at the conclusion of the chapter.

2.1 Sentaurus TCAD

Synopsys Sentaurus TCAD is a suite of tools used for physics-based simulation of advanced semiconductor device structures. The main tools within the Sentaurus TCAD suite are Sentaurus Structure Editor/Mesh, Device, Workbench, and Visual. Within Sentaurus Structure Editor, the 2-D or 3-D device structure dimensions can be defined along with material type, doping profiles, meshing strategies, and contact placement. Default material properties (for example, GaN and Si) are provided by Sentaurus, it is possible to adjust the material properties using a user-defined parameter (.par) file. For example, the bandgap, density of states, thermal conductivity can all be customized. Once the structure is defined, Sentaurus Device takes the structure and meshing profile to compute the electrostatic potential, electron and hole concentrations and electron and hole currents present within the device based on physics models solved at each mesh point. Solving
at a discrete number of mesh points provides an approximation to a real device, so it is preferred that the meshing be laid out such that a higher resolution can be obtained within areas where a change in current density, electric field, and charge generation is expected. The device physics models for drift-diffusion, thermodynamic and hydrodynamic, if desired and are computed either under electrostatic or biased conditions. All device definitions can be scripted using the Scheme scripting language. Lastly, Sentaurus Workbench and Visual are GUIs that provide a means to interact with the simulation for analysis and visualization.

### 2.1.1 Electrostatic Model and Material Parameters

The Poisson equation, shown in eq. 2-1, describes the electrostatic potential within the device, where \( n \) and \( p \) are the free electron and hole concentrations, \( N_{D^+} \) and \( N_{A^-} \) are the ionized donors and acceptors, and \( \varepsilon \) is the isotropic semiconductor permittivity. The electron and hole continuity equations are given in eq. 2-2 and eq. 2-3. Upon initiating the simulation with a defined doping profile, eq. 2-1, eq. 2-2, and eq. 2-3 will be solved iteratively while maintaining self-consistency. The simulation will end once equilibrium convergence is reached.

\[
\nabla^2 \psi = -\frac{q}{\varepsilon} (p - n + N_{D^+} - N_{A^-})
\]

\[
\nabla \cdot \vec{J}_n = qR + q \frac{\delta n}{\delta t}
\]

\[
\nabla \cdot \vec{J}_p = -qR - q \frac{\delta p}{\delta t}
\]
The drift-diffusion model is utilized for carrier transport in the TCAD simulations reported here. The standard assumptions of full ionization of dopants and constant temperature are made. The electron and hole current densities are shown in eq. 2-4 and eq. 2-5 where $\mu_{n,p}$ are the electron and hole mobilities, $D_{n,p}$ are the electron and hole diffusivities and $\Phi_{n,p}$ are the electron and hole quasi-Fermi potentials, respectively. The currents can be expressed in terms of the quasi-Fermi potentials if the Einstein relation, shown in eq. 2-6, holds.

\[
\vec{J}_n = q \mu_n nE + qD_n \nabla n \equiv -qn \mu_n \nabla \Phi_n
\]

\[
2-4
\]

\[
\vec{J}_p = q \mu_p pE + qD_p \nabla p \equiv -qp \mu_p \nabla \Phi_p
\]

\[
2-5
\]

\[
D_n = \frac{kT}{q} \mu_n, D_p = \frac{kT}{q} \mu_p
\]

\[
2-6
\]

The spontaneous and piezoelectric polarization fields ($P_{SP}$, $P_{PE}$) present within the III-Nitride semiconductor need to be considered within the simulation. The polarization induced charges can be added to the Poisson equation. An additional modification to the Poisson equation can be made to include fixed/trapped charge ($\rho_{\text{trap}}$) that may result in additional doping, enhancing recombination, and leakage current.\(^1\) The below eq. 2-7 shows the modification to eq. 2-1 where $q_{PE}$ is the polarization induced charge density. This can be found by taking the derivative of the total polarization ($P_{\text{total}}$), where $P_{\text{total}}$ is a sum of both the spontaneous and piezoelectric components.
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\[ \nabla^2 \psi = -\frac{q}{\varepsilon} \left( p - n + N_{D^+} - N_{A^-} \right) + q_P - \rho_{\text{trap}}, \quad q_P = -\nabla \cdot \vec{P}_{\text{total}} \]

Table 2 shows the chosen default material parameters used for the TCAD simulations. Any deviation from the default values throughout the simulation set is specifically noted.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Symbol</th>
<th>GaN</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Bandgap</strong></td>
<td>( E_g )</td>
<td>3.47 eV</td>
</tr>
<tr>
<td><strong>CB DOS</strong></td>
<td>( N_c )</td>
<td>2.2 ( \times ) 10^{18} cm(^{-3} )</td>
</tr>
<tr>
<td><strong>VB DOS</strong></td>
<td>( N_v )</td>
<td>4.6 ( \times ) 10^{19} cm(^{-3} )</td>
</tr>
<tr>
<td><strong>Intrinsic carrier concentration</strong></td>
<td>( n_i )</td>
<td>4.1 ( \times ) 10^{-10} cm(^{-3} )</td>
</tr>
<tr>
<td><strong>Electron mobility</strong></td>
<td>( \mu_n )</td>
<td>1800 cm(^2)/Vs</td>
</tr>
<tr>
<td><strong>Hole mobility</strong></td>
<td>( \mu_p )</td>
<td>30 cm(^2)/Vs</td>
</tr>
<tr>
<td><strong>Electron diffusion coeff.</strong></td>
<td>( D_n )</td>
<td>39 cm(^2)/s</td>
</tr>
<tr>
<td><strong>Hole diffusion coeff.</strong></td>
<td>( D_p )</td>
<td>0.75 cm(^2)/s</td>
</tr>
<tr>
<td><strong>Dielectric const.</strong></td>
<td>( \varepsilon_r )</td>
<td>8.9</td>
</tr>
</tbody>
</table>

2.1.2 **Planar GaN and Combined 3D GaN PIN**

Physics-based Sentaurus TCAD simulations were performed to fine-tune the device layer thicknesses and model the depletion layer layout of the proposed combined 3D device structure. To begin, a comparison is made between planar GaN PN and PIN diodes to qualify the simulation output. Realistic estimated carrier densities of 5 \( \times \) 10^{17} cm\(^{-3} \) holes for the p-GaN layer, 1 \( \times \) 10^{18} cm\(^{-3} \)
3 electrons in the n-GaN layer and 1 x 10^{16} \text{cm}^{-3} of electrons in the “intrinsic” u-GaN layer were used for the TCAD simulation input. Note that the term u-GaN here refers to the fact that there is no intentional dopant introduced during growth. For a standard PN junction, the depletion width can be calculated from known dopant densities (N_A, N_D) and built-in potential (V_o). In order to calculate the V_o, the intrinsic carrier density (n_i) is calculated given the conduction and valance band density of states (N_C, N_V) for GaN at room temperature (300 K), taken from Table 2.

\[
n_i,\text{eff} = \sqrt{N_C \cdot N_V \cdot e^{-\frac{E_g}{2kT}} = 4.1 \times 10^{-10} \text{cm}^{-3}}
\]

\[
V_o = \frac{kT}{q} \ln \left( \frac{N_A N_D}{n_i^2} \right) = 3.3 \text{ V}
\]

\[
W_d = \sqrt{\frac{2\varepsilon V_o}{q} \left( \frac{1}{N_A} + \frac{1}{N_D} \right)} = 98.7 \text{ nm},
\]

with \(N_A x_p = N_D x_n \Rightarrow x_n = 32.9 \text{ nm}, x_p = 65.8 \text{ nm}\)

This is shown to match the TCAD simulation output, shown in Figure 2-1, which is a plot of the band diagram overlaid with the electrostatic potential. The depletion width (W_d) measures approximately ~100 nm, with a V_o measured to be ~3.3 V. The Fermi level is shown to be placed just below the conduction band minimum, indicating near-degeneracy, which is consistent with the given doping level and density of states available. For betavoltaic application, the PN diode will have a limited ability to capture EHPs given the small depletion volume, where capture relies heavily on the presence of electric field for carrier separation and drift.
Figure 2-1: Band diagram and built-in potential of GaN PN junction at equilibrium

Figure 2-2: Band diagram and built-in potential of GaN PIN junction at equilibrium, 500 nm u-GaN layer
Figure 2-3: Band diagram and built-in potential of GaN PIN junction at equilibrium, 1000 nm u-GaN layer

In order to increase the size of the depletion region, while maintaining the fixed dopant densities in the GaN PN diode, a PIN diode can be utilized. As the naming suggests, a PIN is similar to the PN diode with the inclusion of an intrinsically doped layer. In practice, the intrinsic layer is typically a highly resistive lightly n- or p-type doped material. For this work, a u-GaN layer is used with a background free electron density of $1 \times 10^{16}$ cm$^{-3}$, for simplification this PN-N diode will be referred to as PIN diode. Referring to Figure 2-2, it is seen that the potential drops across the full u-GaN layer, enhancing the depletion width to $>500$ nm, a 5x increase over the PN junction depletion width. From the Maxwell equation, $(\vec{E} = -\nabla V(y))$ the gradient of the potential equals the electric field, therefore if there is a change in the potential within y-dimension (in the case of this 1D simulation) an electric field will be present. This is confirmed in Figure 2-4, which
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shows the electric field distribution throughout the PIN diode. At equilibrium ($V_a = 0$), the full u-GaN region contains an electric field, i.e. it is fully depleted. The maximum electric field ($E_{\text{max}}$) is approximately $1.1 \times 10^5$ V/cm compared to approximately $6.6 \times 10^5$ V/cm in the case of the GaN PN diode. Increasing the u-GaN layer thickness to 1000 nm, referring to Figure 2-3 and Figure 2-4, a portion of the layer ($y = \sim 0.6 \ \mu m$ to $\sim 1.05 \ \mu m$) now contains $\Delta V = 0$, meaning no electric field is present. This is nonideal for betavoltaic application, as EHPs generated within this region will have a lowered probability to be collected. The collection of EHPs will rely on their diffusion into the depletion region where an electric field is present, otherwise they will recombine, and the energy will be lost. Regions such as this are sometimes referred to as “dead-zone” meaning the probability for collection of carriers from excitation that occurs within this region is low to negligible. For this reason, a u-GaN region of 500 nm in thickness is considered for a combined 3D PIN layout. Monte Carlo simulations described later in the thesis, show that this thickness is also well-suited for absorption of $^{63}$Ni $\beta$-emission continuum.
Figure 2-4: Electric field distribution in GaN PN and PIN diode, varied u-GaN layer thickness
As described previously in Chapter 1.4.3, a combined 3D PIN design will enhance the surface area contact with the radioisotope, lower average defect density in material, and increase collection probability. By combining a 3D-core-shell and planar design, larger devices can be made while also utilizing the full wafer footprint. In contrast, a standalone 3D-core-shell design will have electrical discontinuity between features and will require the deposition of an additional conductive material to join features. Here, a combined 3D PIN was simulated with the film layer thicknesses similar to the planar PIN simulated structure, with 800 nm n-GaN seed core, 500 nm u-GaN overgrown film, and 100 nm p-GaN overgrown film. Mesa width, spacing, and height are optimized for a variety of factors including experimental limitations such as growth interactions between adjacent pillar growth and minority carrier diffusion length, amongst others. The structure shape, doping profile, and electric field distribution is shown in Figure 2-5. The script code that was written to construct this structure and define the meshing coordinates is provided in Appendix B: Sentaurus Code. Figure 2-6 is a plot of the electric field magnitude along the horizontal width of a single core-shell structure. The depletion region coverage is seen to extend vertically along the n-GaN core providing a built-in electric field in the planar regions and 3D structures simultaneously. Both lateral and vertical electric fields contributed to the absolute electric field distribution shown in Figure 2-5. Approximately 50% of the total core-shell volume consists of the depletion region leading to increased probability of absorption compared to only planar PIN layout or only 3D. The thickness of the i-layer was optimized to match the approximate depletion width. The thickness of the p-GaN layer was reduced to increase the likelihood of capturing generated carriers near surface, based on the simulated energy absorption profile.
Figure 2-5: Sentaurus TCAD simulation at 0 V bias of 3D combined PIN. (left) structure shape and doping profile of PIN (right) Absolute electric field within the material.

Figure 2-6: Field distribution taken through the core-shell width. The field extends approximately 10 nm into the pGaN layer. The peaks in electric field at approximately 1.6 µm and 2.4 µm distance correspond to the n+-n- junction.

To better illustrate the likelihood of beta capture, Figure 2-7 shows an estimate for collection probability as a function of distance into the proposed pin core-shell structure. The collection probability is considered unity for carriers generated inside of the high field (depletion) region, while the collection probability is considered zero for carriers generated more than a diffusion length from the depletion region. The variable $L_h$ ($L_e$) represents minority carrier
diffusion length in n-GaN (p-GaN) material. The minority diffusion length is impacted by defects and impurities in the material. Referring again to Figure 2-7, the collection probability is zero near the center of the nGaN core since the generated carriers will recombine or will interact with a trap state prior to reaching the depletion region. It has been shown that minority hole / electron diffusion length is a direct function of dislocation density and doping level of the material. For a typical MOCVD grown GaN on sapphire with dislocation density >10^{10} cm^{-3} and Mg acceptor (Si donor) doping of 10^{19} cm^{-3} (10^{18} cm^{-3}) the electron (hole) minority diffusion length is ~200 nm (50 nm). For holes in n-type GaN this value can be markedly improved with a reduction in the dislocation density while maintaining [Si] concentration. By utilizing HVPE bulk GaN with dislocation density <10^{6} cm^{-3}, the hole minority diffusion length can be improved to ~230 nm. The minority carrier diffusion length in p-type GaN is heavily dependent on [Mg] level, but if considering [Mg] = 10^{19} cm^{-3}, the diffusion length can be improved to ~700 nm. It is worth noting that a reduction in the dislocation density is expected in the current design as both the i- and the p- layers are laterally overgrown layers. In such growth method, the bulk of the threading dislocations will not propagate laterally into the overgrown layer. This will further enhance the diffusion length of minority carriers and improve overall device efficiency by reducing carrier scattering.
2.2 CASINO Monte Carlo

2.2.1 Background

“monte CARlo Simulation of electroN trajectory in sOlids” (CASINO) is a Monte Carlo simulation software that employs a single-scattering algorithm, which is designed to model electron beam interactions in bulk and thin samples. The software is capable of handling vertical and horizontal planes (2D), which enables heterostructures, multilayers, and grain boundaries to be simulated. Regions can be defined, with a characteristic thickness, chemical composition, and weight density. The software will automatically calculate a weight density based on the atomic/weight fraction of each element; however, it is recommended to change the density based
on known literature values; GaN is 6.91 g/cc and Ni is 8.90 g/cc, Al₂O₃ is 2.98 g/cc. The user defined materials can be saved into a library for future use.

Physical models can be selected using the Simulation/Change Physical Models drop-down based on user preferences and nature of the work. The default parameters are used for the simulations described in this thesis. CASINO assumes a Gaussian electron beam; the width and penetration angle of the beam are defined by the user. Within the user defined width, 99.9% of the distribution is considered. The XY coordinates (X₀ and Y₀) for the incident electron beam are given by eq. 2-8. The distance between two successive collisions, L, is calculated by eq. 2-9, where the λₑl is defined alongside eq. 2-9. Cᵢ, Aᵢ, are the weight fraction and atomic weight of element i, and ρ is the density of the region (g/cc) being considered. The cross-section σᵢ (nm²) are taken from tabulated values. R is a random number between 0 and 1. The electron energy loss is tracked by a continuous loss function, known as the Bethe relation (eq. 2-10, eq. 2-11). Inelastic scattering processes such as plasmon excitation, phonon excitation, Bremsstrahlung generation and ionization are not directly considered in the simulation. The angle of elastic collision is determined using pre-calculated values of partial elastic cross-section and a single random number.

\[
X₀ = \frac{d\sqrt{\log(R₁)}}{2 \cdot 1.65} \cdot \cos(2\pi R₂), \quad Y₀ = \frac{d\sqrt{\log(R₁)}}{2 \cdot 1.65} \cdot \cos(2\pi R₃)
\]

\[
L = -\lambdaₑl \log(R₄) [nm], \quad \frac{1}{\lambdaₑl} = \rho N₀ \sum_{i=1}^{n} \frac{Cᵢσₑlᵢ}{Aᵢ}
\]

\[
E_{i+1} = E_i + \frac{dE}{dS}L
\]
\[
\frac{dE}{dS} = -7.85 \times 10^{-3} \rho \cdot \sum_{j=1}^{n} \frac{G_{j}Z_{j}}{F_{j}} \ln \left( 1.116 \left( \frac{E_{i}}{J_{j}} + k_{j} \right) \right) [\text{keV/nm}]
\]

2.2.2 Relative Energy Absorption in $^{63}\text{Ni}$ / GaN Pillar Layout

A series of simulations were performed to quantify the energy absorbed at a given incident electron energy, within the combined 3D GaN PIN layout. The simulations also serve to determine the relative electron energy loss within the isotope ($^{63}\text{Ni}$) source, dependent on the starting position, along with quantifying the energy that can reach an adjacent GaN pillar. Starting positions of 0 $\mu$m (GaN surface), 0.5 $\mu$m (into Ni), and 1.0 $\mu$m (into Ni) were simulated. A value of 50 eV cut-off energy was defined for the electron trajectory; $10^4$ electrons were simulated and with electron energy ranging from 2-67 keV in increments of 5 keV. This range of electron energies covers the spectrum of $^{63}\text{Ni}$ emission. The material parameters were defined with the GaN density set to 6.91 g/cc and Ni set to 8.90 g/cc. Figure 2-8 below shows an example of the simulation output which displays the electron path through the materials when the starting position of incidence is at the GaN surface. The trajectory was at $0^\circ$ incidence in the horizontal direction, as indicated in the left image in Figure 2-8, and the “pillar” is assumed to be infinitely tall, therefore energy lost from large deviations in trajectory orthogonal to the incident angle (out of pillar top or below pillar) is not considered. Only 1% of electrons are displayed, where the blue line path indicates full incident energy absorption within the material layers while the red line path indicates a backscattered electron i.e., partial amount of incident energy absorbed within the material, the rest is lost as the electron trajectory is terminated (energy absorbed outside of the material). The total relative energy absorbed for all simulated electrons can be found by integrating the energy loss curve within a specific region of the material.
Figure 2-8: (right) Example of simulation output (electron path) for starting position at GaN sidewall surface (left) for 17 keV and 30 keV electron energy.

Referring to Figure 2-9, all electron energy is absorbed within the first GaN pillar for starting energy of 2-22 keV. For a starting energy of 22-42 keV, a certain number of electrons will penetrate beyond the first GaN pillar and deposit energy within the adjacent Ni layer shown by the red curve, ranging from 0 – 40% of the total energy available. For a starting energy >42 keV, a finite amount of absorption will occur in the adjacent GaN pillar and beyond. However, for these higher energy electrons the bulk of the absorption is occurring in the Ni layer beyond the starting GaN pillar which is nonideal as this energy is lost and will not contribute to device output.
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Figure 2-9: Energy absorption between layers for emission at GaN sidewall surface

Figure 2-10: Energy absorption between layers for emission 0.5 microns into Ni-layer

Figure 2-11: Energy absorption between layers for emission 1.0 microns into Ni-layer
Referring to Figure 2-10, with a starting position of 0.5 µm (into Ni) and starting electron energy of 2-12 keV all energy is absorbed in the Ni layer. This effect is known as self-absorption, where some percentage of the electron (β) loses energy within the radioisotope it originated from. For starting energy ranging from 27-47 keV, the dominant absorption occurs in GaN rather than...
the Ni. For a starting position of 1.0 \( \mu \text{m} \) (into Ni), as shown in Figure 2-11, the self-absorption worsens with electron energy up to 22 keV losing all energy within the starting Ni layer. Figure 2-12 shows a bar graph representation of the ratio of energy absorbed in GaN:Ni at each starting position, considering all layers present within the simulation. For example, the energy absorbed in the first GaN pillar and second GaN pillar and summed to result in the total value absorbed in GaN. The plots show the significant effect of self-absorption as the electron starting position begins within the isotope itself. Assuming an isotropic radioisotope film, only a small fraction of electrons will originate from near surface – with minimal attenuation of kinetic energy prior to reaching the GaN converter material. This shows a clear need to either lower the thickness of this radioisotope layer or revert to a radioisotope compound that has lower material density. This is addressed in the next section with a transition to \( ^{63}\text{NiCl}_2 \) liquid form solution which has a resulting density of 1.05 g/cc.

There are several limitations when using the CASINO software for BV simulations, the main limitation is the joint inability to simulate multiple electron energies simultaneously and multiple emission directions simultaneously. Only a single starting electron energy can be simulated at a single angle of incidence in any given simulation run. Although this serves as a reasonable starting point, it does not match the reality of \( \beta \)-emission which is a probability spectrum of energies and directionality.

2.3 Monte Carlo N-Particle Extended (MCNPX)

2.3.1 Background

MCNPX is a general-purpose Monte Carlo code that can be used to model neutron, photon, and electron (or coupled) transport \(^{10}\). The code uses an extensive collection of scatter cross-
sectional data and is able to simulate the transportation of these particles with energy from 1 keV to 100 MeV in materials. MCNPX code best mimics a real source by modeling isotropic/full spectrum emission of radiation. The likelihood of specific emission energy is dictated by a probability spectrum defined by the user. As has been reported, a large error in the penetration depth and peak energy absorption will result for cases where simplification is made to consider only a mono-directional path of the incident particles or average energy of emission. The differences associated with various modeling software and method has been well studied. To illustrate this, a brief comparison was made here between monodirectional/monoenergetic beta penetration (CASINO2 and MCNPX), and isotropic/full spectrum beta penetration (MCNPX only). Table 3 shows the values, in microns, for approximate energy deposition range in GaN. The depth quoted for the MCNPX and CASINO software is for the point at which 60% or 90% of the total energy is absorbed. An important observation is that the CASINO and MCNPX penetration is nearly identical for the single energy electrons. This makes sense as the models that are tracking the electron trajectory are the same, as indicated in the software manuals. The main distinction, as previously noted, is the capability of MCNPX to track many different electrons with different starting coordinates and angles of incidence, where CASINO cannot. Note in Table 3 the large deviation in the 90% energy absorbed value when considering the full emission spectrum of a given isotope compared to using just its’ average energy value. For example, $^{63}$Ni has an average $\beta$-emission energy of 17 keV which leads to 0.7 µm depth for which 90% of energy is absorbed, while when considering the full spectrum ranging from 1 – 67 keV the depth is 2.5 µm. The MCNPX isotropic/full spectrum model was chosen for all parametric studies of the combined 3D device, as it is the most realistic case and should reduce errors in device optimization. The
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associated energy deposition trend for $^{63}\text{NiCl}_2$ in GaN is shown below in Figure 2-13. It is confirmed that a peak in energy absorption occurs near-surface, around 100 nm into the GaN. This is due to the highly probable, low energy electrons that are now being considered from the emission source. For this reason, it is necessary to minimize the thickness of the surface layer to enable generation of the largest portion of carriers near/in the depletion region of the device.

Table 3: Approximate energy deposition range in GaN using different source parameters (in microns).

<table>
<thead>
<tr>
<th>Energy Spectrum</th>
<th>MCNPX (60% total)</th>
<th>MCNPX (90% total)</th>
<th>CASINO (60% total)</th>
<th>CASINO (90% total)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 keV</td>
<td>0.1</td>
<td>0.16</td>
<td>0.1</td>
<td>0.14</td>
</tr>
<tr>
<td>17 keV</td>
<td>0.5</td>
<td>0.7</td>
<td>0.45</td>
<td>0.68</td>
</tr>
<tr>
<td>63 keV</td>
<td>4.5</td>
<td>7.5</td>
<td>4.9</td>
<td>7.2</td>
</tr>
<tr>
<td>240 keV</td>
<td>36.6</td>
<td>60.8</td>
<td>47.5</td>
<td>69.0</td>
</tr>
<tr>
<td>$^3\text{H}$</td>
<td>0.1</td>
<td>0.3</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$^{63}\text{Ni}$</td>
<td>1.0</td>
<td>2.5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$^{147}\text{Pm}$</td>
<td>9.5</td>
<td>18.6</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 2-13: Energy deposition profile of $^{63}\text{NiCl}_2$ source into GaN material using MCNPX isotropic/full spectrum model.
The goal of the following simulations is to compare the coupling of isotope layers to the planar and 3D designs. Parametric studies of isotope layer thickness on planar design were investigated. For the 3D design, parametric studies of GaN mesa width and height (blocks 71-74 in Figure 2-14), and isotope gap width (blocks 61-63, 83-89 in Figure 2-14) were evaluated. The blocks are not to scale in the schematic. The variations were then evaluated for figures of merit that include \( \frac{C_i}{cm^2} \), \( \frac{P_{GaN}}{cm^2} \) (where \( P_{GaN} \) is defined as energy deposited per second in the GaN material) and \( \eta_{src} \) (defined here as power deposited in GaN vs nuclear power available from isotope, or \( P_{GaN}/P_{nuc} \)).

![Figure 2-14: The planar design (left) and 3D+planar design (right) are modeled on 100 µm x 100 µm GaN base. The yellow regions denote radioisotope volumes of 1.05 g/cc, while the grey and blue volumes are GaN transducer of density 6.15 g/cc.](image)

A \(^{63}\text{NiCl}_2\) liquid form solution is modeled, with a density of 1.05 g/cc that is interfaced with the GaN surface (6.15 g/cc). The liquid solution is chosen because the liquid fills the gaps between GaN mesas. The liquid solution also shows promise due to lower material density and therefore lower self-absorption. Liquid form \(^{63}\text{Ni}\) has been used with phosphors.\(^{17}\) The mesa structures can efficiently be filled with \(^{63}\text{Ni}\) activity after GaN wafer production. The process of
coating the wafer with liquid solution increases manufacturability of devices for future cost efficiency, as depositing solid metal radioisotope is expensive and not always conformal.

### 2.3.2 Planar GaN with $^{63}\text{NiCl}_2$

The energy deposition of liquid form $^{63}\text{NiCl}_2$ was calculated and shown in Figure 2-15 for a planar GaN layer. As an example, the 15 μm thick layer of $^{63}\text{NiCl}_2$ deposits a low amount of energy in the air above, as the absorption in air 0.00129 g/cm$^3$ is much less than that of the GaN wafer below. The energy deposition range in the GaN due to incident $^{63}\text{NiCl}_2$ beta spectrum is shown to fall off to 10% of its original value within 2 μm and 1% of the original energy deposition within 4 μm. These ranges are consistent with previously calculated results of $^{63}\text{Ni}$ electron range in GaN.$^{16}$ The 2 μm range for 90% energy deposition is compatible with scalar values for NIST CSDA scalar calculations when three characteristics (maximum energy, average energy, and energy that contains the most flux) of the $^{63}\text{Ni}$ beta are included.$^{18}$

![Figure 2-15: (left) Energy deposition (MeV/cc) in planar GaN from $^{63}\text{NiCl}_2$ layer (middle layer) with air layer on top. (right) A cut of the energy deposition through the layer stack, values are shown. The boundary edges are clearly visible between layers.](image)
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In Figure 2-16 the maximum power generated inside of the GaN material is realized at an isotope layer thickness of ~10 μm, while the power generated in the isotope layer itself continuously increases (because of the increase in volume and Ci/cm² generated). The efficiency of the source material in transferring energy into GaN continues to decrease as the isotope layer thickness increases (Figure 2-16). This is expected due to self-attenuation in the isotope medium. It is also expected that for the lower density of the source medium the medium can be thicker before power gain is saturated (gas<liquid<solid).

![Figure 2-16](image)

*Figure 2-16: (left) The efficiency continues to decrease as the isotope layer thickness increases. (right) The transfer of energy to the GaN energy converter peaks at 10 μm isotope layer thickness, where $\eta_{src}$ is ~5.5%.*

### 2.3.3 Combined 3D GaN with $^{63}$NiCl₂

The mesa width of the combined 3D GaN device was varied from 2 μm to 4 μm. Given the average range of beta penetration in GaN from the $^{63}$Ni source, the larger 4 μm width does not add power performance or energy source efficiency performance. The 2 μm width has the highest $\eta_{src}$
for all isotope gap spacing. This can be viewed in Figure 2-17 and Figure 2-18. As the gap spacing increases between mesas, the power continues to increase because the isotope source material is added to the surface (Ci/cm²) of the combined device. However, the energy transfer efficiency is reduced as the gap spacing creates more self-absorption. Therefore, the smaller the gap spacing on the 2 µm mesa width, the higher the conversion efficiency will be. Figure 2-19 shows a schematic version of the simulation output for the absorption as a function of gap spacing with constant mesa width of 4 µm.

Figure 2-17: (left) The 2 µm mesa width shows higher power generated for all values of mesa separation (63Ni Gap Size) compared to 4 µm mesa width. (right) Source activity per wafer area shows similar trend
Figure 2-18: Efficiency for 2 μm mesa width is higher for all values of mesa separation (63Ni Gap Size), with the highest efficiency achieved at 2 μm gap (2 μm mesa).

Figure 2-19: Example of MCNPX simulation output (energy deposited) for 4 μm mesa width with changing variable isotope filled gap spacing (2-15 μm), i.e. 4x2 refers to (mesa)x(spacing).

Mesa height was also varied from 4 μm to 8 μm. As is seen in Figure 2-20, a doubling of the mesa height only increases the energy deposited in the GaN 3D pillar by a factor of ~1.5x. This is consistent with the change in surface area, as the base surface area between the mesas does not
change during the mesa variation – only sidewall area. The energy transfer efficiency reduces greatly for the 8 μm mesa height for all isotope gap spacing because more isotope activity is being used and self-attenuated (Figure 2-21).

Figure 2-20: (left) Doubling the mesa height results in ~1.5x power gain, but also requires higher source activity (right) Source activity per wafer area

Figure 2-21: The efficiency lowers as the mesa separation ($^{63}$Ni Gap Size) is increased, independent of mesa height.
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An important distinction when changing structure dimensions is not only the power generated but the change in total activity per surface area (Ci/cm$^2$). In terms of its economics, it is not useful to have a structure that has an increased activity per surface area without substantial increase in power. Table 4 shows peak values for power generated and the associated activity density for the planar, and combined 3D structure with 4 μm mesa height, and 8 μm mesa height. Each value quoted is with an isotope gap spacing of 10 μm and mesa width of 2 μm.

By moving to 3D+planar device design with 4 μm mesa height the simulation shows a 3.75x increase in power generated per wafer surface area at roughly half the activity density, compared to the planar device. As the mesa height is increased, so is the activity density with a large increase in power generated. Comparing the 8 μm mesa height to the planar structure, there is a 5.65x increase in power generated at only 1.07x the activity. Depending on the application a larger aspect ratio structure may be preferred.

Table 4: Comparison of power generated per sq. cm compared to the activity cost per sq. cm

<table>
<thead>
<tr>
<th>Structure</th>
<th>$P_{\text{GaN}}$ (μW)/cm$^2$</th>
<th>Ci / cm$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Planar</td>
<td>2.0</td>
<td>0.42</td>
</tr>
<tr>
<td>4 μm mesa height</td>
<td>7.5</td>
<td>0.23</td>
</tr>
<tr>
<td>8 μm mesa height</td>
<td>11.3</td>
<td>0.45</td>
</tr>
</tbody>
</table>

2.4 Summary

A combined 3D (planar+core-shell) PIN device has been proposed as the optimal design for GaN application as a BV battery. This optimized structure layout leads to enhanced depletion
region volume compared to that in a planar device under equilibrium conditions. MCNPX code was used to simulate a more realistic full spectrum isotopically emitting beta source to couple with a 3D combined device structure. $^{63}$NiCl$_2$ material has greatly decreased material density compared to Ni metal film which limits self-absorption and can allow a higher thickness to be used efficiently. The material also has the advantage of coming in liquid form to easily spread and cover 3D structures conformally. A parametric study of mesa width, mesa height, and isotope gap spacing was performed with a comparison made to a planar structure. It is shown that both the power generated and efficiency of energy transfer decline with an increase in mesa width. When increasing mesa height from 4 $\mu$m to 8 $\mu$m there will be a trade-off where power generated will increase at the cost of efficiency and isotope activity density. There is a 3.75x increase in $P_{\text{GaN}}$/cm$^2$ at approximately half the activity density for the 4 $\mu$m mesa height structure compared to planar designs optimum at 10 $\mu$m $^{63}$Ni thickness with 5.82x improvement in $\eta_{\text{src}}$. The 8 $\mu$m mesa height structure shows a 5.56x increase in power generated at only 1.07x the activity.

The optimum device dimensions of 2 $\mu$m pillar/mesa width with p-GaN(100nm), u-GaN(500nm), and n-GaN (800nm), 4 $\mu$m mesa height, with 2 $\mu$m mesa separation is chosen based on simulation, deposition, and fabrication considerations. Realistic doping levels of $5 \times 10^{17}$ cm$^{-3}$ holes, $1 \times 10^{18}$ cm$^{-3}$ electrons were used for the simulation input leading to optimum depletion coverage in the core-shell of ~50% total volume. The 3D combined p-i-n design shows promise to increase efficiency for GaN based BV batteries through increase in surface area, reduction of material defects, and enhanced depletion layout. This methodology can be used to enhance the device dimension optimization process for next generation 3D GaN BV structures coupled with any isotope source to increase overall device performance.
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2.5 References


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Chapter 3 Epitaxial Growth and Film

Characterization Methods

3.1 Metalorganic chemical vapor deposition (MOCVD)

Metalorganic chemical vapor deposition (MOCVD), also known as organometallic vapor phase epitaxy (OMVPE) or metalorganic vapor phase epitaxy (MOVPE), is a chemical vapor deposition (CVD) method in which gaseous precursors are injected into a reactor at high temperature which then react on the surface of a substrate to form a crystalline solid. The “metalorganic” comes from the use of chemical precursors in the form of metal atoms attached to methyl groups. The relatively low cost, repeatability, and high-volume production using the MOCVD growth process has led to the commercialization of LED technology worldwide. A complete fundamental understanding of MOCVD growth is still being developed, given the complexity of the chemical reactions during the deposition process. Therefore, most of the current understanding of III-Nitride crystal growth has been developed empirically. The theoretical component is typically divided into thermodynamics and kinetics. Growth regimes can be separated into kinetic limited, mass transport limited, and desorption limited. Commonly, mass transport limited regime is considered the most dominant process, in which the diffusion of the reactant to the surface is the limiting variable to growth rate. The basics of the MOCVD process will be discussed in this chapter as it relates to the growth of III-Nitride thin films. The added
complexity of selective area growth (SAG) or selective area epitaxy (SAE) in which growth takes place only on specified portions of the surface will be discussed in detail in Chapter 6.

3.1.1 Thermodynamics and Kinetics

MOCVD is described as a nonequilibrium process, given that gases (vapor phase) enter the deposition system in a state of supersaturation and are not in equilibrium with the growing solid due to kinetic factors. The growth process can be thought of as the solid phase and vapor phase being separated by a boundary layer. The chemical potential of the vapor, later defined, are drastically reduced across the boundary layer and a local equilibrium is achieved at the solid and vapor interface.¹ To understand the equilibrium at this interface, beginning with the basics of thermodynamics, the Gibbs free energy per mole, \( G \), is defined as eq. 3-1, where \( H \) is the enthalpy, \( S \) the entropy, and \( T \) the temperature. The enthalpy, \( H \), can be defined as the sum of the internal energy, \( E \), and product of pressure (\( P \)) and volume (\( V \)) of a system. The Gibbs free energy is the maximum amount of work that can be performed within a closed thermodynamic system. A decrease in \( G \) corresponds to the work done by the system. When the Gibbs free energy per mole is minimized, the system is said to be in equilibrium. For a two-phase system such as the one found in MOVPE, any movement of material between these phases should result in no change in the total Gibbs free energy. This derivative of Gibbs free energy with respect to moles of material is known as the chemical potential (\( \mu_i \)) with the pressure and temperature remaining constant. The equilibrium condition is therefore defined as eq. 3-2.

\[
G = H + TS, \quad H = E + PV
\]

\[
\left( \frac{\delta G}{\delta n_i} \right)_{\text{solid}} - \left( \frac{\delta G}{\delta n_j} \right)_{\text{vapor}} = \mu_i^{\text{solid}} - \mu_j^{\text{vapor}} = 0
\]

³-1

³-2
For a chemical reaction in equilibrium a general formula is expressed in eq. 3-3, with the total change in Gibbs free energy shown in eq. 3-4. The Gibbs free energy of an individual species can be represented by eq. 3-5 with $G_i^o$ being the free energy of a given species at a reference condition, R the ideal gas constant, and $a_i$ the activity. The activity of a gas is equivalent to its partial pressure, $p_i$.

$$aA + bB \rightleftharpoons cC$$

$$\Delta G = cG_C - aG_A - bG_B$$

$$G_i = G_i^o + RT \ln a_i$$

For the chemical reaction in eq. 3-3, the change in Gibbs free energy can then be written as eq. 3-6. For the equilibrium case we consider this to equal zero, from this the equilibrium constant term ($K$) is defined, shown in eq. 3-7. Recalling the relationship between Gibbs free energy and chemical potential, eq. 3-6 can then be rewritten as eq. 3-8 which defines the thermodynamic driving force of the system to return to an equilibrium state.

$$\Delta G = \Delta G^o + RT \ln \left( \frac{a_C^c}{a_A^a a_B^b} \right)$$

$$0 = \Delta G^o + RT \ln \left( \frac{a_C^{eq}}{a_A^{eq(a)} a_B^{eq(b)}} \right) \rightarrow -\Delta G^o = RT \ln(K)$$

$$\Delta \mu = \mu_C^o - \mu_A^o - \mu_B^o - RT \ln \left( \frac{a_C^c}{a_A^a a_B^b} \right)$$
Using the above, the thermodynamics of GaN solid crystal growth by MOCVD can be defined. The common precursors employed are tri-methyl gallium (TMGa) for the Ga source, and ammonia (NH$_3$) for the N source. If assumed that the pyrolysis of each precursor is unity at the growth temperature (commonly >1000°C) the chemical reaction can be expressed as eq. 3-9. The chemical potential driving force for the formation of GaN is then defined by eq. 3-10, where the activity is replaced by the partial pressures given that the species are all in a gaseous state. This defines a maximum possible growth rate for a given system.

$$Ga(g) + NH_3(g) \Leftrightarrow GaN(s) + \frac{3}{2} H_2(g)$$  \hspace{1cm} 3-9

$$\Delta \mu = \mu_{Ga}^0 + \mu_{H_2}^0 - \mu_{Ga}^0 - \mu_{NH_3}^0 - RT \ln \left( \frac{\frac{3}{2}p_{H_2}}{p_{Ga}p_{NH_3}} \right)$$  \hspace{1cm} 3-10

In real-world application there are many competing effects that alter the achieved growth rate. For one the NH$_3$ source has a limited cracking efficiency at growth temperature, meaning incomplete pyrolysis of available input source. There is also the need for very high partial pressure of N above the GaN solid to ensure it incorporates and to limit the formation of a Ga liquid. The high temperatures needed for high crystalline GaN formation leads to increased volatility of the N species resulting in vacancies within the material. These vacancies can be filled by unwanted/unintentional impurities, such as O, which will act as n-type dopant leading to high background electron carrier concentration in “intrinsic” GaN films. There is also an abundance of vapor-phase reactions that take place which alter the expected source reaching the growing surface.
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Thermodynamics describes the energy within the system during the initial, equilibrium and final states but does not provide information on the time required to achieve equilibrium, the steps involved, or the rates at which various processes are occurring. The steps involved in MOCVD are schematically depicted in Figure 3-1, which is common for most all CVD processes. Precursors are injected into heated chamber and diffuse towards a substrate surface. Gas phase reactions may occur during vapor transport, altering the available precursor to the solid surface and composition of species present in the chamber. Once transported through the boundary layer adsorption to the film surface occurs. The species can now diffuse, nucleate, and/or desorb from the film back into the boundary layer. In an ideal case, for GaN growth, Ga and N adatoms will nucleate into a solid crystalline form free from impurity and defects. For growth of III-V semiconductors, there are a number of complex vapor-phase reactions that take place between the species that result from pyrolysis. An overview of such reactions for GaN is shown in Figure 3-2, where two main pathways exist 1) the adduct formation path and 2) the TMGa pyrolysis path. This alludes to the complexity of tracking the kinetics of a given species. In addition, the flow dynamics and temperature variation within a reactor are highly complex and difficult to predict without intensive computer simulation.
3.1.2 Origin of defects in III-Nitride by MOCVD and mitigation techniques

A remarkable trait of the III-Nitride material system is the ability to perform in a multitude of device applications while having a substantial amount of dislocation defects present in the thin film from heteroepitaxial growth. The lattice mismatch and thermal mismatch between GaN and the common non-native substrate materials (sapphire, SiC, Si) leads to a high density of extended defects in the range of $10^8 - 10^{10} \text{ cm}^{-2}$. The most common type of defect being the threading
dislocation (TD) which can extend through the full thickness of the epitaxial film and may act as nonradiative recombination and scattering centers which will affect the carrier diffusion length and mobility. As an example, enhanced internal quantum efficiency was observed for an InGaN/GaN-based LED with a reduction in TD-induced nonradiative recombination centers by growing on patterned sapphire substrates.³

Sapphire is the most commonly used substrate material due to its low cost, stability at high temperature, and technological maturity for GaN growth. Exhibiting a hexagonal crystal structure, the common (0001) basal plane of GaN aligns with the (0001) of sapphire. The lattice mismatch between the materials is calculated to be >30%, however the small cell of Al atoms is oriented 30° away from the larger sapphire unit cell leading to a calculated mismatch of 16% shown in eq. 3-11.⁴ This also means that the a-plane (11-20) of GaN and m-plane (10-10) of sapphire are parallel to each other, and vice-versa. This is an important consideration when making alignments to the substrate for regrowth masks in SAG, and/or device fabrication relative to the substrate wafer flat.

\[
\frac{\sqrt{3}a_{\text{GaN}} - a_{\text{sapphire}}}{a_{\text{sapphire}}} = 0.16
\]

3-11

Figure 3-3: Orientation relationship between (0001) c-plane GaN to sapphire (0001) c-plane
Due to this lattice mismatch, a multi-step growth is required to achieve a smooth GaN film, which is detailed in Section 3.3.1 on the growth of n-type GaN thin films (a.k.a. GaN template). In addition to planar growth optimizations, several unique MOCVD growth methods have been used or developed to reduce the number of defects in GaN. These methods include epitaxial lateral overgrowth (ELO), pendeo-epitaxy (PE), and growth on patterned, or on native substrates. The ELO and PE methods rely on dislocation reduction through bending and termination in a 3D format with growth anisotropy (lateral-dominant vs vertical basal plane). Utilizing a ELO growth method for GaN on sapphire, it has been shown that a reduction in average dislocation density to $\sim 10^6$ cm$^{-3}$ can be achieved while maintaining a smooth top c-plane surface. This value is for the average dislocation density for the film which includes both the ELO openings and winged regions (above the regrowth mask). In the winged regions the material is nearly defect free, with most all of the ELO opening regions containing the dislocation density similar to the underlying film. A detailed description of the growth dynamics of these techniques will be provided in Chapter 6, where the similar SAG/SAE method is employed for the growth of 3D+planar GaN microstructures and is a crucial component of this thesis work.

### 3.2 Film Characterization Methods

To evaluate the initial and post-irradiation properties of the III-Nitride planar and 3D BV structures presented in this thesis, several surface and bulk semiconductor characterization methods are utilized. A brief description of the fundamentals of operation for the techniques are provided below.
3.2.1 Atomic force microscopy (AFM)

AFM is a surface measurement technique which uses a scanning probe tip to measure parameters (roughness, topography, surface potential, local conductivity) of a material surface. In the common tapping mode setup, a laser beam and detector track the deflection of the probe tip as it oscillates/taps the material surface to provide information on roughness and topography. This mode is used for all measurements of surface roughness provided in this thesis. This mode is preferred over the conventional contact mode as it increases tip longevity and leads to high resolution imaging. The resolution of the surface roughness measurement is on the order of angstroms. In a conductive AFM mode of operation (C-AFM), both the topography and local nanoscale conductivity of a material is measured which can provide insight into leakage pathways and doping nonuniformities. For C-AFM, a bias voltage is applied between the sample and conductive probe tip leading to a current (or tunneling current) between them while maintaining a constant tip force. Figure 3-4 provides a schematic of the two distinct modes of operation (tapping mode AFM, C-AFM).

![Figure 3-4: AFM components in (left) contact tapping mode configuration (image credit to 6) and (right) conductive AFM measurement setup (image credit to 7)]
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3.2.2 Hall effect measurement

The Hall effect measurement can provide values for carrier concentration, resistivity, mobility, and confirmation of carrier type. An Accent Hall Measurement tool was used for all data collected in this work. The basic physical principle behind the Hall effect is the Lorentz force, provided in eq. 3-12 showing the force acting on a carrier when in the presence of both an electric and magnetic field. Referring to Figure 3-5 and assuming the carriers can only flow in the x-direction, the net force along the y- and z-direction is zero. This leads to the relationship between the applied magnetic field and electric field in the y-direction as shown in eq. 3-14.

\[ \vec{F} = q(\vec{E} + \vec{v} \times \vec{B}) \]

3-12

![Figure 3-5: Schematic of fields and forces acting on carriers in a Hall measurement configuration (image credit to \textsuperscript{8})](image)

\[ \vec{F} = q\vec{E} + q \begin{bmatrix} \hat{e}_x & \hat{e}_y & \hat{e}_z \\ v_x & 0 & 0 \\ 0 & 0 & B_z \end{bmatrix} = qE_x\hat{e}_x + q(E_y - v_xB_z)\hat{e}_y + qE_z\hat{e}_z \]

3-13

\[ F_y = q(E_y - v_xB_z) = 0 \quad \rightarrow \quad E_y = v_xB_z = \frac{J_x}{qp}B_z \text{ or } -\frac{J_x}{qn}B_z \]

3-14
This is referred to as the Hall field, with a Hall coefficient defined as the Hall field divided by the applied current density and magnetic field. The Hall coefficient can also be calculated by measuring the current, $I_x$, and voltage, $V_H$. The relationship between the Hall coefficient and the carrier density and mobility is provided in eq. 3-15 and 3-16, respectively, assuming a uniformly doped layer.

$$R_H = \frac{1}{qp} = -\frac{1}{qn} = \frac{V_H}{I_x B_z W}$$

$$\mu_p = \frac{R_H}{\rho}, \mu_n = -\frac{R_H}{\rho}$$

3.2.3 Electron microscopy

A scanning electron microscope (SEM) is a characterization tool that allows for high magnification/resolution imaging of a sample through the use of a focused electron beam. The tool used for all electron imaging in this work is a JEOL LEO1550 SEM. The tool consists of an electron source, lens column for focusing, scanning coils for raster, and detector for output signal collection, as shown in the schematic in Figure 3-6. Incident electrons from the source are directed to the sample surface at which point they generate electrons or photons which can be detected pixel by pixel to form an image of the sample geometry. Features within the image are distinguished by a relative contrast that arises from differences in intensity of the output signal. The output signal can be in the form of secondary electrons, backscatter electrons, x-rays, and other photon energies; all of which are generated and originate from various depths within the sample. This means that
different electron energies can be used to probe specific characteristics of the sample. Specialized detectors are used for each type of output radiation signal.

Figure 3-6: Schematic of SEM column showing the main components of the system. (image credit to ⁹)
The sample can be oriented perpendicular to the electron source for top-down imaging or tilted relative to the source for out-of-plane imaging for 3D topography and cross-section imaging. For the characterization of 3D structures in this work, a significant amount of cross-sectional imaging was performed where the sample was cleaved along the a- or m-plane and the cleaved face placed parallel to the beam. When in this layout, multiple layers (for example, PIN layers) can be viewed simultaneously. Of specific usefulness of this method to this work is that the dopant type and density play a role in secondary emission intensity. It has been shown that the secondary electron emission intensity from p-GaN is highest compared to n-type and u-GaN at low electron beam voltages, showing the technique’s efficacy for imaging dopant distributions. Numerous explanations have been provided for dopant contrast, with the escape depth parameter showing a major influence on contrast. This parameter is influenced heavily by the level of surface band
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bending, which is present in both p-GaN and n-GaN due to varied levels of surface states. An upward surface band bending of ~0.7 eV in n-GaN leads to a depletion of electrons from the surface resulting in lower contrast, while a downward surface band bending of ~1.1 eV in p-GaN leads to an enhanced electron transport to the surface increasing contrast.\textsuperscript{10}

![Figure 3-8: Band diagram for n-GaN and p-GaN (left) showing surface band bending and cross-sectional SEM image of PIN planar layers (right). (image credit to \textsuperscript{10})](image)

3.2.4 Photoluminescence (PL) and electroluminescence (EL)

Photoluminescence (PL) is a spectroscopy technique in which incident light is used to probe the electronic structure of a semiconductor. Upon introducing a focused light source with energy greater than the bandgap (hν > E\textsubscript{G}) photoexcitation will occur creating hot carriers. A portion of these carriers will relax and release a photon of equivalent energy, known as radiative recombination, which are collected by a monochromator. While carriers can also recombine by nonradiative transitions which cannot be detected or quantified by this method. Radiative recombination can come in the form of band-to-band, free excitonic, bound excitonic, free electron - neutral acceptor/donor, and neutral donor – neutral acceptor.\textsuperscript{9} The resulting energy spectrum will provide insight into the impurities and defects present within the light excitation depth of the
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semiconductor. Nonradiative recombination processes will also occur in the form of phonons / heat. Electroluminescence (EL) has the same fundamental principle as PL except in this case the excess carriers are provided through electrical injection rather than photoexcitation. The characterization system described below can also be used for EL, through the use of *in situ* biasing by electrical feedthroughs into the cryogenic system.

For PL on GaN thin films, a 325 nm HeCd laser is used with an excitation depth on the order of ~100 nm in GaN. The excitation spot size can be adjusted from approximately 2 mm to 400 µm. For EL of GaN, a Keithley 6430 source-meter is used for electrical biasing. The resulting luminescence for each characterization is directed to an Oriel Cornerstone monochromator where the signal is wavelength-resolved and amplified by a PMT biased at 800 V (unless otherwise stated). Temperature-dependent PL and EL can be obtained down to approximately 18 K, by using a closed-cycle cryogenic vacuum system and He compressor. A liquid N₂ cold trap is placed between the cryogenic housing and the process vacuum pump. An image of the optical bench where the system is located is shown below in Figure 3-9.
Figure 3-9: Photoluminescence setup and optics. The cryogenic housing can be seen in the bottom-right corner of the image.

3.3 MOCVD of Planar GaN PIN

3.3.1 n-GaN template growth

A Veeco D180 vertical cold walled MOCVD reactor is used for all crystal growth presented here. A ~3.7 µm thick n-GaN template layer is the base for all subsequent PIN device epitaxy, both in planar and 3D format. The template layer was grown heteroepitaxially on c-plane sapphire (Al₂O₃) substrate of 430 µm thickness. In the case of heteroepitaxy on sapphire, the growth is performed per the following sequence: 1) a high temperature anneal of the sapphire (1070 °C) in H₂ environment for surface preparation, 2) low temperature (560 °C) GaN buffer layer growth at high V/III (15000), 3) high temperature (1040 °C) 3D island formation at reduced V/III (10000), 4) recovery layer to transition from 3D coalescence to layer-by-layer growth at reduced V/III (3800), and 5) layer-by-layer growth mode by a further reduction of V/III (2100) – the Si n-type dopant is introduced. The precursor sources are tri-methyl gallium (TMGa) for the
Ga source, ammonia (NH$_3$) for N source, and silane (SiH$_4$) for the n-type Si dopant. In situ optical measurement is performed by a k-space ICE tool, which enables tracking of the film reflectance, curvature, and temperature during growth. An example of the reflectance data for a standard GaN template growth is shown in Figure 3-10, the growth mode is noted starting with buffer layer growth in region “a”. The reflectance is used to track the quality of the thin film during the growth process, along with providing the ability to quantify the thickness of the thin film by the number of interference fringes that result during the growth. This feature can be seen in region “d” of Figure 3-10. Figure 3-11 shows AFM scans of the n-GaN epitaxially surface, showing low surface RMS roughness from flat (left) to round (right) of a 2-inch wafer and presence of atomic steps. For this specific film, the Hall effect measurement indicates a carrier concentration of 2.0x10$^{18}$ cm$^{-3}$ and mobility of 257 cm$^2$/Vs. Additional growths of n-GaN templates on sapphire used for device epitaxy presented in this thesis have similar morphological and electrical characteristics to those shown here.

Figure 3-10: Example of reflectivity data for GaN template growth which allows for control of thickness and in-situ quality monitoring.$^{11}$
3.3.2 PIN device epitaxy

As will be described in Chapter 4, several variations in relative layer thicknesses for GaN PIN epitaxy were tested for BV performance. While the relative growth time of the PIN layers is altered, the order and reactor conditions remain the same for each. Prior to PIN epitaxy on existing n-GaN template wafers, an n-GaN interface-buried layer is overgrown. This interface-buried layer reduces the effect that interfacial impurity induced charge will have on the PIN device layer. This interfacial impurity can be present following a growth interruption, where an interruption is defined by temporarily ceasing growth and removing the wafer from the growth chamber into the load lock or ambient. Shallow dopants such as O and Si and deep level C acceptor can collect at regrowth interfaces and effect PN device leakage when grown directly adjacent.\textsuperscript{12,13} Following the interface-buried layer, the PIN was grown continuously without interruption in the following order, n-GaN / u-GaN / p-GaN. The u-GaN layer is grown under identical conditions to the n-GaN layer without the supply of the SiH\textsubscript{4} dopant precursor. The p-type dopant precursor source is Bis(cyclopentadienyl)magnesium (Cp\textsubscript{2}Mg). This thin film layer has been previously optimized.
with a chamber pressure, temperature, and \( \text{Cp}_2\text{Mg} \) precursor flow of 300 Torr, 980 °C, and 120 sccm, respectively. Each layer was calibrated in separate growth runs prior to continuous PIN device epitaxy; the range of standard Hall measurement data is listed in Table 5. The planar PIN films used in this thesis fall within this range of electrical properties.

Table 5: Standard Hall effect measurement values for doped/undoped GaN layers grown by MOCVD.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Carrier Concentration (cm(^{-3}))</th>
<th>Mobility (cm(^2)/Vs)</th>
<th>Resistivity (ohm-cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-GaN</td>
<td>(1-2 \times 10^{18})</td>
<td>250-300</td>
<td>~0.01</td>
</tr>
<tr>
<td>u-GaN</td>
<td>(3-4 \times 10^{16})</td>
<td>550-600</td>
<td>~0.4</td>
</tr>
<tr>
<td>p-GaN</td>
<td>(2-3 \times 10^{17})</td>
<td>5-10</td>
<td>~7</td>
</tr>
</tbody>
</table>

Figure 3-12: AFM of standard u-GaN surface from a sample grown in close proximity to s0520, expect u-GaN layer in PIN growth to be of similar quality. Smooth step-flow growth present at 2 \(\mu\)m x 2 \(\mu\)m and 5 \(\mu\)m x 5 \(\mu\)m scan area.
Figure 3-13: AFM of the p-GaN surface of s0520 GaN PIN. Smooth step-flow growth present at 2 µm x 2 µm and 5 µm x 5 µm scan area. Undulation in surface topology due to stress caused by Mg dopant.

Figure 3-12 and Figure 3-13 are AFM images of the u-GaN and p-GaN layer, respectively. Note the increase in surface undulation and step bunching for the p-GaN surface. P-type doping in GaN, and other wide bandgap materials, has historically been difficult to achieve. Mg is a relatively deep dopant at ~200 meV however, it forms molecular complexes with H₂ during growth which remain at room temperature—a large advantage in the use of Mg as p-dopant. These complexes passivate the Mg dopant resulting in a highly resistive film. A post-growth activation annealing, in the form of a thermal, microwave or electron beam source, is required to break the Mg:H₂ complexes leading to p-type conductivity in the film. The maximum density of free holes at room temperature has been shown to be ~10¹⁸ cm⁻³ with Mg concentration of ~3x10¹⁹ cm⁻³. However, a 1% activation at low 10¹⁷ cm⁻³ is most common. These values are for Ga-polar / c-plane oriented growth, there is a difference in the incorporation dynamics for Mg in GaN along different crystallographic fronts. For N-polar GaN growth, an enhancement in Mg incorporation along the
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semi-polar facets has been observed by atom-probe tomography. Similar concepts, as it relates to the doping of 3D PIN core-shell microstructures will be addressed in this work.

3.4 References


6 Https://Www.Witec.de/Techniques/Afm/ (n.d.).


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Chapter 4 Electron Beam Response

Enhancement of GaN PIN Diodes by Improvement in Device Processing

4.1 Device Fabrication

4.1.1 Design considerations for planar GaN BV

4.1.1.1 Beta-transparent p-contact

For device applications that rely on light extraction (LEDs) or radiation absorption (solar cells, BV, photodetectors), the selection of the top contact location, thickness, and material is critical. In contrast, for standard III-Nitride HEMT, PIN / PN device layout for power electronics, a top p-contact is placed over the full device mesa. The characteristic thickness and location of this contact is of minimal concern for such applications, with the sole objective being to lower the contact resistivity. In addition to the top electrode metallization, inclusion of an oxide passivating layer or nitride field-plate layer can enhance switching performance, increase maximum breakdown voltage, and decrease field spikes within the electric field profile.\textsuperscript{1,2} The thickness of such features can be on the order of several microns. For LEDs, it is standard to use a thin current-spreading contact layer for the p-contact, called the “transparent” contact. The transparent layer allows for enhanced light extraction out from the MQW region, where a thick electrode would attenuate the light intensity significantly. It is important for the contact material to maximize transmittance in the visible wavelength region while maintaining an ohmic relation with the
semiconductor, for which thin Ni/Au, thin Pt, and ITO have been used. Without the transparent contact it is difficult to achieve uniform current spreading across the full device area given the high resistivity found in the p-GaN top layer. These concepts are similar to requirements for GaN BVs, where a thin top contact is required for minimal attenuation of the incident $\beta$ kinetic energy, and a current spreading layer is required to capture a larger percentage of EHPs generated across the full device area. Though there is no contact metal that can achieve true transparency to $\beta$ irradiation, the contact can be made thin enough to minimally attenuate the incident kinetic energy. For all devices discussed in this thesis, a thin p-contact was utilized in addition to a thick outer ring p-contact for probing. The material(thickness) used was Ni(4nm)/Au(4nm) and Ni(50nm)/Au(50nm) for the thin and thick p-contact, respectively. This contact was shown to absorb <3% of incident electron energies (at 16 keV) and is a standard thickness for the transparent layer used in MQW LEDs showing good ohmic behavior to p-GaN.

4.1.1.2 Surface defect passivation by wet chemical etching

Inductively coupled plasma (ICP) - reactive ion etching (RIE) is necessary for top-down mesa isolation of individual device structures. Due to the predominately physical nature of this process at high power (typically $>300$ W) the GaN surface (and in some cases regions deeper into the film) is damaged. This can appear macroscopically in the form of increased surface roughness and microscopically as the formation of dangling bonds and N vacancies. The presence of such damage has been shown to degrade the electrical and optical performance of the device, typically in the form of increased leakage current through the exposed/etched GaN surfaces. The severity of this effect can be limited through an optimization of the etch conditions (ICP/RIE power, gas chemistry, relative flows) for a given film. Furthermore, through the use of a post-etch dielectric
passivation or wet-chemical treatment the surface properties can be restored and performance
enhanced.\textsuperscript{5,7,8} For the devices presented in this thesis, a passivation treatment was used for all
device structures exposed to dry-etch damage. A 20 wt% potassium hydroxide (KOH):H\textsubscript{2}O
solution has been used as a wet-etch passivation treatment for planar diodes following mesa
isolation. In GaN, KOH is a polarity-selective etchant where the c-plane epitaxial plane is left
unaffected by exposure to the chemical. This selectivity is shown to originate from the surface
bonding configuration of nitrogen, where Ga-polar orientation (c+ plane, [0001]) is inert to KOH
etch and the N-polar orientation (c- plane, [000-1]) shows strong continuous etching.\textsuperscript{9} A proposed
mechanism of the plane-dependent etching of GaN by KOH attributes the degree of etching to
planar density (number of atoms / Å\textsuperscript{2}) and the number of dangling bonds on the nitrogen atom.\textsuperscript{8}
The authors is that work propose that the product of these quantities provides an etching barrier
index (EBI), which ranks the crystallographic planes in the following order for difficulty in etching
(hardest -> most prone to etching): c+ \rightarrow a \rightarrow m \rightarrow -c \rightarrow [10-1-1] \rightarrow r-plane. When dealing with
a 3D device that has multiple crystallographic planes present, such a treatment is not used due to
the strong morphological change that will occur. Referring to Figure 4-1, an as-ICP etched GaN
mesa was subjected to a 2 wt% KOH-containing solution, AZ400K, at 60 °C for 6 hrs. The relative
etching between crystal planes is observed, with the semi-polar planes etching quickly resulting in
micro-faceting towards the nonpolar sidewalls. The c-plane mesa surface remains unaffected.
Consecutive wet etchings with 20 wt% KOH:H₂O solution at 60 °C on an ICP-etched GaN PN mesa was performed to test the degree of improvement in leakage current versus etch duration. The ICP etching of the GaN PN mesas was performed under standard conditions (BCl₃/Cl₂ etch chemistry, 300 W ICP, 100 W RIE). After each successive etch, the I-V characteristics of a single 0.04 cm² diode were measured and compared to the initial state. Referring to Figure 4-2, over an order of magnitude improvement in leakage current at -5 V is observed after 15 minutes. As etch time is increased to 30 min, further improvement is negligible.
4.1.1.3 Large area device

To maximize power output of a BV, access to large area in a single device is highly desired. However, as GaN device area increases there is typically an accompanying increase in the diode leakage current due to increased number of bulk crystalline defects, presence of randomly located small pits or nano pipe defects through the as-grown thin film and increased total etched surface area during the mesa isolation process step. This can be detrimental to BV performance where the device operates under low injection currents that can be on the same order of magnitude as the dark leakage current. The goal towards achieving a high performing device is to ensure the leakage current is much lower than the injected/generated current from the β-source ($J_d < J_{gen}$). In order to study the performance of devices with varied electrical properties and area under electron
injection, diodes with varied areas were fabricated. The diodes with largest area were made to be 0.04 cm$^2$ square and 0.0314 cm$^2$ circular.

### 4.1.2 Fabrication process flow

Planar PIN GaN layers were grown by MOCVD in a Veeco D180 reactor as described in the previous chapter, both on sapphire and bulk GaN substrate. Three different PIN layer stacks were grown, with the associated sample ID and layer dimensions shown in Table 6, and layer schematic shown in Figure 4-4. The layer dimensions / nominal carrier concentration are 1) 80 nm p-type GaN / $1 \times 10^{17}$ cm$^{-3}$, 500 nm u-GaN / $3 \times 10^{16}$ cm$^{-3}$, on n-type GaN template / $1 \times 10^{18}$ cm$^{-3}$, 2) 200 nm p-type GaN / $1 \times 10^{17}$ cm$^{-3}$, 500 nm u-GaN / $3 \times 10^{16}$ cm$^{-3}$, on n-type GaN template / $1 \times 10^{18}$ cm$^{-3}$, and 3) 100 nm p-type GaN / $1 \times 10^{17}$ cm$^{-3}$, 700 nm u-GaN / $3 \times 10^{16}$ cm$^{-3}$, on n-type GaN template / $1 \times 10^{18}$ cm$^{-3}$.

**Table 6: Sample ID and PIN layer dimensions, all grown on n-GaN template.**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Substrate</th>
<th>p-GaN</th>
<th>u-GaN (i)</th>
</tr>
</thead>
<tbody>
<tr>
<td>s0520b</td>
<td>Sapphire</td>
<td>80 nm</td>
<td>500 nm</td>
</tr>
<tr>
<td>s0520c</td>
<td>Sapphire</td>
<td>200 nm</td>
<td>500 nm</td>
</tr>
<tr>
<td>s1507a, s1507b</td>
<td>Sapphire</td>
<td>80 nm</td>
<td>500 nm</td>
</tr>
<tr>
<td>S1419b</td>
<td>Bulk GaN</td>
<td>100 nm</td>
<td>700 nm</td>
</tr>
</tbody>
</table>
The fabrication process flow is schematically depicted in Figure 4-3. Optical lithography was performed using AZ5214 photoresist to open mesa areas. Deposition of 100 nm Ni metal etch mask for mesa isolation etch was completed using a Varian 980 electron beam evaporator. Mesa isolation was achieved by inductively coupled plasma (ICP) etch in a Trion ICP-RIE system, with the etch concluding at 200 nm depth into the n-GaN template layer (~1 µm total depth, depending on PIN thickness). The Ni etch mask was removed by wet chemical etch (Ni etchant TFB). After mesa formation, the sample was treated with a 20% KOH:H₂O wet etch to passivate the etched surfaces unless otherwise specified. Device mesa area ranged from 7.84x10⁻⁴ cm² to 0.04 cm², for square and circular geometries. N-type ohmic contact alloy of Ti(25nm)/Al(220nm)/Ni(60nm)/Au(50nm) was deposited by electron beam evaporation. The n-type contact was annealed at 800 ºC for 30 sec in an N₂ inert environment using a rapid thermal annealing (RTA) system. The P-type contact of Ni(4nm)/Au(4nm) and Ni(50nm)/Au(50nm) for the thin and thick regions, respectively, was deposited by electron beam evaporation in separate runs. The p-contact was annealed at 450 ºC for 5 min in O₂ environment using an RTA system.
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For all steps requiring lithography, an organic rinse of acetone, IPA, and DI was used to clean samples, and the resist was removed by heated Microposit Remover 1165 at 65 °C and O₂ plasma Asher. Figure 4-5 shows optical micrographs of fabricated planar GaN diodes with (left) lower dimensional devices with conformal n-contact and (middle) larger 0.0314 cm² device with spaced n-contact (n-contact pad not visible in image). Figure 4-5 also shows the distinction between the mesa surface-covering (thin) p-contact and the ring (thick) p-contact meant for probing previously mentioned. Samples were cleaved and packaged into a 12-lead printed circuit board (PCB) adhered by silver paint placed on the sapphire substrate (Figure 4-5, right). Al wire-bonds were used to attach the selected devices to the PCB board’s Au contact pads. Bulk wire-leads were then connected to the large PCB pads and attached to the source meter and/or parameter analyzer for electrical characterization.

*Figure 4-4: Planar GaN PIN schematic showing the relative layer dimensions for the three iterations of PIN, not to scale*
4.1.3 Dark I-V characteristics

4.1.3.1 Deviations from ideal characteristics

As mentioned in Chapter 1.3.2, numerous effects such as parasitic resistances can lead to PN or PIN I-V behavior that is nonideal. These parasitic resistances are separated into series resistance caused by high contact resistance or high resistance in the intrinsic regions, and parallel resistance which is caused by any unwanted current channel that is parallel to the diode junction.\(^\text{11}\) The unwanted current channels are likely caused by defects in the bulk or at the surface. The eq. 4-1 shows the relative regimes for calculating the parallel and series resistance, respectively, for a given diode. The parallel resistance, or shunt resistance, will always be higher than the series resistance in the region \(V \approx 0\) and therefore the series resistance can be ignored. Similarly, the parallel resistance can be ignored in the region \(V \gg V_{th}\) where the depletion region has collapsed, and high forward conduction occurs. A custom python script (Appendix II) was written to calculate the parasitic resistances values for several I-V measurements instantaneously, without any manipulation of the data by the user.

\(90\)
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\[ R_{sh} = \frac{dV}{dI} @ V \approx 0, \quad R_s = \frac{dV}{dI} @ V \gg V_{th} \]

4-1

It is also common to observe sub-threshold turn-on voltage in a real diode. This subthreshold turn-on can occur due to an additional lower energy barrier (such as a Schottky diode from non-ohmic contacts) in parallel with the PN or PIN diode, and from generation-recombination within the depletion region. Figure 4-6 shows the nonideal I-V behavior for an arbitrary diode, partitioned by the voltage regime (region a,b,c,d,e) where a given effect alters the current conduction.\(^\text{12}\) Generation-recombination is not considered in the Shockley equation and causes an excess current in both the forward and reverse bias regimes, dominating at low voltages.\(^\text{11}\) The current due to generation, \(J_{ge}\) within the depletion region of a PN junction is given by eq. 4-2, where \(\tau_{ge}\) is the generation lifetime and \(U\) is the generation rate. This current is proportional to the depletion width at any given temperature, which will change with the applied bias. Therefore, for abrupt junctions, \(J_{ge}\) is proportional to \(V\) as shown in eq. 4-3.

\[ J_{ge} = \int_0^{W_d} q|U| dx \approx q|U|W_d \approx \frac{q n_i W_d}{\tau_{ge}} \]

4-2

\[ J_{ge} \propto (\psi_{bi} + V)^{\frac{1}{2}} \]

4-3

The current in reverse bias for a PN junction can then be approximated by the sum of the diffusion component and generation current, shown in eq. 4-4, which refer to the “region e” in Figure 4-6. This shows that for semiconductors with large \(n_i\) the diffusion term will dominate.
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Under forward bias most of the generation-recombination in the depletion region is recombination, so a recombination current, $J_{re}$, is added to the diffusion current shown in eq. 4-5. This forward current corresponds to region a-b in Figure 4-6.

$$J_R = J_D + J_{ge} = q \frac{D_p n_i^2}{\tau_p N_D} + \frac{q n_i W_d}{\tau_{ge}}$$

4-4

$$J_F = J_D + J_{re} = q \frac{D_p n_i^2}{\tau_p N_D} \exp \left( \frac{qV}{kT} \right) + \sqrt{\frac{\pi}{2}} \frac{kT n_i}{\tau_p \xi_o} \exp \left( \frac{qV}{nkT} \right)$$

4-5

Figure 4-6: Example of nonideal I-V compared to ideal I-V for arbitrary diode with a) generation-recombination current b) diffusion current c) high-injection d) series resistance e) reverse leakage due to generation-recombination. (Image credit to Sze Chapter 2.3, Page 97)
4.1.3.2 Planar GaN PIN Dark I-V

Details on the device geometry, area, leakage current density \( (J_d) \) at -5 V, \( R_{sh} \) and \( R_s \) is shown in Table 7-10, for the planar GaN PIN devices tested here. Comparing s0520b and s0520c, which differ in p-GaN layer thickness and fabricated device area, in Table 7 and Table 8 respectively, an order of magnitude difference emerges between both \( R_{sh} \) and \( R_s \). These values were calculated according to eq. 4-1. When comparing s0520b and s1507a which have the same p-GaN layer thickness but different device areas, the \( R_{sh} \) is comparable. This implies that the p-GaN thickness may play a vital role in the resulting \( R_{sh} \). The \( R_s \) however seems to scale with device area, independent of the sample PIN layer thickness. For sample s0520b, \( R_s \) is on the order of \( 10^{-2} \) \( \Omega \)cm\(^2\) for device area <0.0075 cm\(^2\) and on the order of \( 10^0 \) \( \Omega \)cm\(^2\) for device area >0.0314 cm\(^2\). As mentioned previously, it is optimal to maximize the \( R_{sh} \) when using a device for BV energy conversion. The \( R_{sh} \) runs parallel to the diode and if low in magnitude the generated current will flow through this pathway resulting in power loss. The \( R_s \) does not have as large of an impact given the voltage regime where power is being generated, typically \( V_{oc} < 2.5 \) V. The \( J_d \) is another important characteristic to consider from the dark I-V. Since the generated current from \( \beta \)-irradiation is extremely low, it is a requirement that the \( J_d \) be lower than that of the generated current, as mentioned previously. Note that the value quoted in the Table 7- Table 10 is at -5 V bias, and the operating regime for BV is in the 4\(^{th}\) quadrant (0 V – \( V_{oc} \)), but a low \( J_d \) at -5 V is a good indication for a significantly lower value at ~0 V as the value scales with applied voltage. Figure 4-7 shows a typical I-V trace for the diodes, and the trend in \( J_d \) as a function of voltage can be seen.
Figure 4-7: Dark I-V of s0520c Device S4, typical I-V trace for GaN PIN studied here (absolute log scale).

Table 7: s0520b DIE2

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>I_d @ -5V (A)</th>
<th>J_d @ -5V (mA/cm²)</th>
<th>R_sh (Ωcm²)</th>
<th>R_s (Ωcm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>Square / 0.00144 cm²</td>
<td>-2.8x10⁻¹²</td>
<td>-1.0x10⁻⁵</td>
<td>3.7x10⁷</td>
<td>0.038</td>
</tr>
<tr>
<td>11</td>
<td>Square / 0.00336 cm²</td>
<td>-1.0x10⁻¹⁰</td>
<td>-3.0x10⁻⁶</td>
<td>4.3x10⁷</td>
<td>0.035</td>
</tr>
<tr>
<td>17</td>
<td>Circle / 0.00754 cm²</td>
<td>-2.8x10⁻⁸</td>
<td>-3.7x10⁻²</td>
<td>9.4x10⁷</td>
<td>0.085</td>
</tr>
<tr>
<td>19</td>
<td>Square / 0.00144 cm²</td>
<td>-6.4x10⁻⁹</td>
<td>-4.4x10⁻²</td>
<td>5.8x10⁷</td>
<td>0.021</td>
</tr>
<tr>
<td>24</td>
<td>Square / 0.00608 cm²</td>
<td>-5.6x10⁻⁹</td>
<td>-9.3x10⁻⁴</td>
<td>4.5x10⁷</td>
<td>0.078</td>
</tr>
<tr>
<td>32</td>
<td>Circle / 0.00264 cm²</td>
<td>-9.5x10⁻⁸</td>
<td>-3.6x10⁻²</td>
<td>4.7x10⁷</td>
<td>0.031</td>
</tr>
</tbody>
</table>
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**Table 8: s0520c DIE1**

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>( I_d ) @-5V (A)</th>
<th>( J_d ) @-5V (mA/cm(^2))</th>
<th>( R_{sh} ) ((\Omega)cm(^2))</th>
<th>( R_s ) ((\Omega)cm(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>Square / 0.04 cm(^2)</td>
<td>-1.9x10(^{-2})</td>
<td>-0.48</td>
<td>4.2x10(^5)</td>
<td>2.3</td>
</tr>
<tr>
<td>S4</td>
<td>Square / 0.04 cm(^2)</td>
<td>-2.3x10(^{-7})</td>
<td>-5.7x10(^{-3})</td>
<td>5.6x10(^6)</td>
<td>2.2</td>
</tr>
<tr>
<td>C7</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-1.0x10(^{-6})</td>
<td>-3.3x10(^{-2})</td>
<td>6.9x10(^5)</td>
<td>1.8</td>
</tr>
<tr>
<td>C3</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-3.1x10(^{-5})</td>
<td>-0.996</td>
<td>1.4x10(^5)</td>
<td>2.1</td>
</tr>
</tbody>
</table>

Comparing s1507a and s1507b, the distinction between the two samples is a KOH wet etch passivation following the mesa isolation dry etch. The wet etch passivation shows an improvement to both the \( J_d \) and \( R_{sh} \) by at least an order of magnitude on average. But the values are still in a reasonable range for obtaining power from an electron irradiation-induced generation current.

**Table 9: s1507a (no passivation)**

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>( I_d ) @-5V (A)</th>
<th>( J_d ) @-5V (mA/cm(^2))</th>
<th>( R_{sh} ) ((\Omega)cm(^2))</th>
<th>( R_s ) ((\Omega)cm(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>C3</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-1.5x10(^{-8})</td>
<td>-4.7x10(^{-4})</td>
<td>3.2x10(^{7})</td>
<td>8.3</td>
</tr>
<tr>
<td>C4</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-6.0x10(^{-10})</td>
<td>-1.9x10(^{-5})</td>
<td>4.4x10(^{7})</td>
<td>6.6</td>
</tr>
<tr>
<td>C7</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-2.7x10(^{-9})</td>
<td>-8.6x10(^{-5})</td>
<td>5.5x10(^{7})</td>
<td>3.2</td>
</tr>
<tr>
<td>C10</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-3.0x10(^{-7})</td>
<td>9.5x10(^{-3})</td>
<td>5.6x10(^{7})</td>
<td>2.8</td>
</tr>
</tbody>
</table>

**Table 10: s1507b (KOH passivation)**

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>( I_d ) @-5V (A)</th>
<th>( J_d ) @-5V (mA/cm(^2))</th>
<th>( R_{sh} ) ((\Omega)cm(^2))</th>
<th>( R_s ) ((\Omega)cm(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>Square / 0.04 cm(^2)</td>
<td>-7.9x10(^{-10})</td>
<td>2.0x10(^{5})</td>
<td>7.1x10(^{8})</td>
<td>2.4</td>
</tr>
<tr>
<td>S5</td>
<td>Square / 0.04 cm(^2)</td>
<td>-1.3x10(^{-10})</td>
<td>-3.3x10(^{6})</td>
<td>1.6x10(^{9})</td>
<td>1.5</td>
</tr>
<tr>
<td>C9</td>
<td>Circle / 0.0314 cm(^2)</td>
<td>-1.0x10(^{-9})</td>
<td>-2.5x10(^{5})</td>
<td>4.6x10(^{8})</td>
<td>2.9</td>
</tr>
</tbody>
</table>
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4.2 Electron flood-gun irradiation

4.2.1 System description

A Kimball Physics EMG-12 electron flood-gun was used for electron irradiation in the energy range of 0-16 keV. An electron beam is formed by thermionic emission from a tungsten filament. The electrons are incident to a sample chamber at 2 x 10^{-6} Torr, which is equipped with a Faraday cup for measurement of beam current. The beam current can be set as high as 1 mA but can become unstable at such high currents. The beam current is most stable, and representative of real isotope activity, in the range of 0.1 - 1 nA. Beam diameter can be adjusted between 0.5 mm to 10 mm. Custom steal apertures can be placed in the beamline over a device to isolate the exposure area to <= 0.5 mm. I-V measurements were captured in situ by electrical feedthroughs and a HP 4155B parameter analyzer.

The more-probable β-decay energies for both the \(^3\)H and \(^{63}\)Ni radioisotope are within the range of energies the flood-gun can produce. This irradiation study is a means to mimic joining the devices with a radioisotope source, by quantifying the device response under electron energies similar to those emitted by the radioisotope. Maximum power produced (MPP) and power conversion efficiency (\(\eta_{\text{conv}}\)) are used as the figures of merit for the performance of the device at a given electron irradiation energy. This along with tracking the short circuit current density (\(J_{\text{sc}}\)) and the open circuit voltage (\(V_{\text{oc}}\)) of the device, provides a means for qualifying and comparing the applicability of devices for use as a BV converter.
4.2.2 Electron beam energy dependence: Maximum power produced (MPP) and efficiency ($\eta_s$)

Device #24 (s0520b) was selected due to its’ larger size and low leakage characteristics for irradiation studies using the electron flood gun, as described above. The device was irradiated at electron beam energies of 4-16 kV, with a target radioactivity of 2.5 Ci/cm$^2$ achieved by setting the beam current to 18.5 pA and using a 400 $\mu$m stainless steel aperture to isolate the irradiation to the device area.

As the electron energy is increased, therefore the increased input power, the device shows a gradual improvement in $\eta_{\text{conv}}$. Based on the measured carrier concentrations, the depletion region is modeled to be contained within the 500 nm u-GaN layer. For carriers generated within this region they are collected with a high probability. If carriers are generated in regions outside of the depletion region, such as the p-GaN surface layer and n-GaN template layer, there is a lower
probability that they will be captured which is based on the diffusion length/direction of the generated minority carrier. The finite probability can be estimated using the relation \( \tanh(W/L) \), where \( W \) is the distance from the depletion region and \( L \) is the diffusion length of the minority carrier. Electrons with lower incident energies dissipate most of their energy within the p-GaN surface layer. While those of higher energy dissipate the bulk of their energy in the depletion region (u-GaN), contributing to pair creation and high collection efficiency. At 16 keV beam energy (the average energy of the \(^{63}\text{Ni}\) beta decay) and 315 nW input power, the device shows 21.9 nW MPP leading to 6.96% \( \eta_{\text{conv}} \) maximum. Table 11 shows the measured values for open circuit voltage (\( V_{\text{oc}} \)), short circuit current density (\( J_{\text{sc}} \)), and the calculated values of MPP and \( \eta_{\text{conv}} \). The efficiency of conversion at 16 keV (the average for \(^{63}\text{Ni}\) source) is the highest value measured and reported for a GaN PIN device.

\[\text{Table 11: s0520b Device 24: Electrical output characteristics for flood gun irradiation (through } \Phi=400 \mu\text{m aperture)}\]

<table>
<thead>
<tr>
<th>( E ) (keV)</th>
<th>Input Power (nW)</th>
<th>( J_{\text{sc}} ) (( \mu\text{A/cm}^2 ))</th>
<th>( V_{\text{oc}} ) (V)</th>
<th>MPP (nW)</th>
<th>( \eta_{\text{conv}} ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>79</td>
<td>0.43</td>
<td>1.32</td>
<td>0.19</td>
<td>0.235</td>
</tr>
<tr>
<td>5.7</td>
<td>104</td>
<td>1.68</td>
<td>1.48</td>
<td>0.82</td>
<td>0.791</td>
</tr>
<tr>
<td>8</td>
<td>148</td>
<td>3.90</td>
<td>1.76</td>
<td>3.69</td>
<td>2.49</td>
</tr>
<tr>
<td>10</td>
<td>182</td>
<td>5.71</td>
<td>1.90</td>
<td>6.94</td>
<td>3.81</td>
</tr>
<tr>
<td>12</td>
<td>217</td>
<td>8.24</td>
<td>1.98</td>
<td>10.8</td>
<td>4.99</td>
</tr>
<tr>
<td>14</td>
<td>257</td>
<td>9.87</td>
<td>2.02</td>
<td>14.0</td>
<td>5.44</td>
</tr>
<tr>
<td>16</td>
<td>315</td>
<td>14.5</td>
<td>2.07</td>
<td>21.9</td>
<td>6.96</td>
</tr>
</tbody>
</table>
Figure 4-9: s0520b Device 24, IV-dependence on incident electron energy, showing power generation.

Device S4 and S3 (s0520c) were also tested. A Ø =1 mm stainless steel aperture was centered over the device to isolate the beam. With a beam current of 117 pA an activity density of 2.5 Ci/cm² was achieved to match the previous experiment. The beam energy was scaled from 4 – 16 keV similar to the previous experiment. Device S4 and S3 differed in dark I-V characteristics,
therefore it is anticipated that the performance under electron irradiation will also differ given the difference in starting $J_d$ and $R_{sh}$.

Device S4 shows a limited power response at the lower irradiation energies (4 keV, 5.7 keV) and begins measurable power production at energies > 8 keV. Recall that this sample has a 200 nm p-GaN layer (compared to the 80 nm p-GaN layer of s0520b), therefore most of the kinetic energy of the incident electrons with starting energies of 4 keV and 5.7 keV is dissipated prior to entering the collection (depletion) region. An MPP of 60.4 nW and 57.3 nW is achieved at an irradiation beam energy of 16 keV and 14 keV, respectively. The maximum $\eta_{conv}$ is 3.83% under irradiation of 14 keV beam energy. An interesting feature that appears is a turn-around of the $V_{oc}$ at 16 keV to below that of 14 kV, but the $I_{sc}$ is larger – leading to a slightly higher power produced, shown in Figure 4-11 and Figure 4-12. The turn-around of the $V_{oc}$ correlates quantitatively with a decrease in the $\eta_{conv}$ to 3.54%.

Given that the aperture diameter in this experiment was ½ the diameter of the mesa, and centered in the middle of the mesa, the aperture window was shifted towards the edge of the device in order to test whether the response to irradiation would be the same for this area. The results of which can be reviewed in Table 13. Compared to the readings with the aperture centered on the mesa, the $V_{oc}$, MPP, and $\eta_{conv}$ are measurably lower when the aperture is shifted towards the edge. This is likely due to a number of incident electrons impinging on the thick outer ring p-contact rather than the thin mesa p-contact. For those electrons, a significant amount of kinetic energy would be lost prior to penetrating the GaN diode which would lead to a measured decrease in the power output.
Comparing the different p-GaN layer thickness of s0520c, Device S4 (200 nm thick p-GaN layer) to s0520b, Device 24 (80 nm p-GaN layer), the efficiency of collection at 16 keV and 2.5 mCi/cm² increases from 3.54% to 6.96%, an improvement of ~2x. Though it should be noted that additional factors other than the p-GaN layer thickness could contribute to the increase, such as the dark current and parasitic resistances of the diode. The open circuit voltage at 16 keV for the two samples varies from 1.29 V (s0520c) to 2.07 V (s0520b).

Table 12: s0520c, Device S4, (through Ø=1000 µm aperture)

<table>
<thead>
<tr>
<th>E (keV)</th>
<th>Input Power (nW)</th>
<th>$J_{sc}$ (µA/cm²)</th>
<th>$V_{oc}$ (V)</th>
<th>MPP (nW)</th>
<th>η_{conv} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>428</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5.7</td>
<td>610</td>
<td>0.043</td>
<td>0.04</td>
<td>~0</td>
<td>~0</td>
</tr>
<tr>
<td>8</td>
<td>856</td>
<td>0.94</td>
<td>0.66</td>
<td>1</td>
<td>0.16</td>
</tr>
<tr>
<td>10</td>
<td>1070</td>
<td>3.65</td>
<td>1.14</td>
<td>16.1</td>
<td>1.50</td>
</tr>
<tr>
<td>12</td>
<td>1284</td>
<td>6.98</td>
<td>1.26</td>
<td>36.4</td>
<td>2.83</td>
</tr>
<tr>
<td>14</td>
<td>1498</td>
<td>10.61</td>
<td>1.29</td>
<td>57.3</td>
<td>3.83</td>
</tr>
<tr>
<td>16</td>
<td>1712</td>
<td>11.73</td>
<td>1.25</td>
<td>60.4</td>
<td>3.54</td>
</tr>
</tbody>
</table>

Table 13: s0520c, Device S4, aperture shifted towards mesa edge, (through Ø=1000 µm aperture)

<table>
<thead>
<tr>
<th>E (keV)</th>
<th>Input Power (nW)</th>
<th>$J_{sc}$ (µA/cm²)</th>
<th>$V_{oc}$ (V)</th>
<th>MPP (nW)</th>
<th>η_{conv} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>428</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5.7</td>
<td>610</td>
<td>0.039</td>
<td>0.06</td>
<td>~0</td>
<td>~0</td>
</tr>
<tr>
<td>8</td>
<td>856</td>
<td>0.96</td>
<td>0.64</td>
<td>1.8</td>
<td>0.21</td>
</tr>
<tr>
<td>10</td>
<td>1070</td>
<td>3.50</td>
<td>0.86</td>
<td>10.2</td>
<td>0.95</td>
</tr>
<tr>
<td>12</td>
<td>1284</td>
<td>6.85</td>
<td>0.96</td>
<td>27.5</td>
<td>2.14</td>
</tr>
<tr>
<td>14</td>
<td>1498</td>
<td>9.20</td>
<td>0.97</td>
<td>37.2</td>
<td>2.48</td>
</tr>
<tr>
<td>16</td>
<td>1712</td>
<td>12.91</td>
<td>1.02</td>
<td>54.7</td>
<td>3.20</td>
</tr>
</tbody>
</table>
Figure 4-11: s0520c Device S4, IV-dependence on incident electron energy, showing power generation.

Figure 4-12: s0520c Device S4, power output curves for electron energy (4 keV – 16 keV)
Figure 4-13: s0520c Device S4, Aperture shifted towards mesa edge, IV-dependence on incident electron energy

Figure 4-14: s0520c Device S4, Aperture shifted towards mesa edge, power output curves for electron energy (4 keV – 16 keV)

Device S3 does not perform as well as Device S4, with lower MPP and lower $\eta_{\text{conv}}$ for all irradiation energies as shown in Table 14. This is likely attributed to the lower local material quality of this device mesa, as indicated by the higher $I_d$ and lower $R_{th}$ shown in...
Table 8. The higher leakage indicates a higher density of dislocation or impurity that contribute to current flow when the diode should be in reverse blocking mode. The lower $R_{sh}$ could also lead to a possible alternative pathway for current flow which is opposite in direction to the $I_{gen}$ which would lower the measured MPP and $\eta_{eff}$.

Table 14: s0520c, Device S3, \( \text{through } \varnothing=1000 \mu m \text{ aperture} \)

<table>
<thead>
<tr>
<th>$E$ (keV)</th>
<th>Input Power (nW)</th>
<th>$J_{sc}$ ($\mu A/cm^2$)</th>
<th>$V_{oc}$ (V)</th>
<th>MPP (nW)</th>
<th>$\eta_{conv}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.7</td>
<td>610</td>
<td>0</td>
<td>0.02</td>
<td>~0</td>
<td>~0</td>
</tr>
<tr>
<td>8</td>
<td>856</td>
<td>0.89</td>
<td>0.28</td>
<td>0.7</td>
<td>0.08</td>
</tr>
<tr>
<td>10</td>
<td>1070</td>
<td>2.98</td>
<td>0.47</td>
<td>4.5</td>
<td>0.42</td>
</tr>
<tr>
<td>12</td>
<td>1284</td>
<td>5.59</td>
<td>0.60</td>
<td>12.4</td>
<td>0.97</td>
</tr>
<tr>
<td>14</td>
<td>1498</td>
<td>7.25</td>
<td>0.64</td>
<td>17.8</td>
<td>1.19</td>
</tr>
<tr>
<td>16</td>
<td>1712</td>
<td>8.95</td>
<td>0.66</td>
<td>22.2</td>
<td>1.30</td>
</tr>
</tbody>
</table>

Figure 4-15: s0520c Device S3, IV-dependence on incident electron energy, showing power generation.
By comparing the dark I-V characteristics for Device S4 and S3, shown in Figure 4-17, the reasons for the improved performance in S4 can be understood. As noted in Table 15, S4 outperforms S3 under all of the metrics tracked, with a two order of magnitude lower $J_d @ -5V$ and nearly one order of magnitude improved $R_{sh}$. The $R_s$ is comparable but as stated previously is not as important given the voltage regime under operating conditions. The highlighted region in Figure 4-17 defines the voltage regime where the voltaic effect takes place. The increased $I_d$ for S3 for all voltages within this regime leads to compensation of the generated current ($I_{gen}$) during irradiation that ultimately reduces the power production capability of the device.

Table 15: Electrical properties of Device S3 and S4

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>$I_d @ -5V$ (A)</th>
<th>$J_d @ -5V$ (mA/cm$^2$)</th>
<th>$R_{oh}$ (Ωcm$^2$)</th>
<th>$R_s$ (Ωcm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>Square / 0.04 cm$^2$</td>
<td>-1.9x10$^{-5}$</td>
<td>-0.48</td>
<td>4.2x10$^5$</td>
<td>2.3</td>
</tr>
<tr>
<td>S4</td>
<td>Square / 0.04 cm$^2$</td>
<td>-2.3x10$^{-7}$</td>
<td>-5.7x10$^{-3}$</td>
<td>5.6x10$^6$</td>
<td>2.2</td>
</tr>
</tbody>
</table>
4.2.3 Electron beam current dependence

Device S3 (s0520c) underwent irradiation at varied beam current of 24 pA, 117 pA, and 234 pA. The below Figure 4-18 shows the I-V dependence on input beam current. The $I_{sc}$ displays a linear dependence with beam current within this regime, where the $V_{oc}$ is nonlinear. The values of $I_{sc}$ and $V_{oc}$ track well with the expected logarithmic relation provided in Chapter 1, where $V_{oc} \propto \ln(I_{sc})$. Therefore, as the $I_{sc}$ continues to increase (with an increase in the injected current from the electron beam) the $V_{oc}$ will follow a logarithmic trend towards a voltage threshold. The corresponding power curve for these experiments is shown in Figure 4-19.
Figure 4-18: Beam current dependence of Device S4 for 23, 117, and 234 pA

Figure 4-19: Device S3 power output for varied beam current

4.3 High energy irradiation in TEM: novel in operando testing capability

Common methods for investigating device characteristics of direct conversion devices under irradiation for use in a BV configuration include in situ testing under an electron flood gun or in a
scanning electron microscope (SEM). The achievable electron accelerating voltages in these settings is typically limited to <30 kV. Bao et al. reported using a linear accelerator as the electron source to achieve high beam currents and voltages for the investigation of a radiation hardened-Si solar cell as a BV converter. These methods of testing differ from the emission of a radioisotope source in that the electrons incident on the device are monoenergetic, whereas with a real isotope source, a spectrum of electron energies is emitted at all angles. Although the common low-energy radioisotope sources ($^3$H, $^{63}$Ni) are statistically favored to emit electron energies <62 keV, $^{147}$Pm and $^{90}$Sr emit a greater proportion of beta electrons above this level. As such, it is important to understand and quantify device characteristics under irradiation by these higher energies over a period of time. High energy electrons can produce point defects in GaN in the form of Ga and N vacancies negatively impacting its electrical characteristics by the introduction of trap states. Early experimental results for the probability of defect creation utilizing the Rutherford cross-section model indicated that atomic displacement of the Ga atom by electron bombardment occurs at a threshold energy of 440 keV in AlGaN/GaN light emitting diodes (LEDs), which corresponds to a displacement energy of $E_d = 19 \pm 2 \, eV$. More recent molecular dynamics simulations indicate that the displacement energy is likely much higher for both Ga and N at $E_d = 73.2 \, eV$, $E_d = 32.4 \, eV$ respectively. The value for displacement energy is also highly dependent on the plane-orientation of the GaN film. It is known that dose, energy, dislocation density, impurity concentration, and carrier concentration will all have some effect on the characteristics of GaN in response to radiation. In order to select the best design for the next-generation of GaN-based betavoltaic devices it is important to experimentally demonstrate device response under the full energy and activity range of common isotope sources.
4.3.1 TEM system modifications

A JEOL 2010 TEM system with a tungsten (W) filament was used in this study. The standard specimen stage was bypassed within the TEM column, and the beam was brought to focus within the viewing chamber near the final imaging plane. A custom aluminum flange fitting was engineered to hold the packaged device in place while enabling vacuum feedthroughs into the viewing chamber for in situ electrical characterization of the device under irradiation, shown in Figure 4-20. The device package is mounted within a nonconductive G10 fiberglass arm using plastic screws. A Keithley 6430 sub-fA remote source meter paired with Model 6430 Remote Preamp is used to measure the current-voltage (I-V) characteristics of a given device. A Python script was used for remote-control of the source meter and automation of the data capture and storage (Appendix B).

Measurement of the beam current magnitude incident on the device is made via an ammeter in contact with the phosphor screen which is shown in Figure 4-21. This phosphor screen also allows for visualization of the beam size and uniformity. A reference circle of 500 µm diameter at the center of the screen allows for an approximation of the beam area.

![Figure 4-20: (left) Custom aluminum flange feedthrough with G10 fiberglass arm for package mounting and (right) photograph into the viewing chamber with the mounted device in the beamline.](image-url)
Figure 4-21: The phosphor screen that is used as both a beam blank and a means to measure the beam current incident to the device (note: the beam size is greatly reduced from here to match the device mesa area at ~2mm diameter/width).

4.3.2 Experimental parameters

A systematic study of irradiation conditions was performed with a beam of approximately equal area to the device area of 0.04 cm$^2$ for all of the tests, unless otherwise noted. Under each of the conditions described hereafter, the I-V characteristics of the DUT was captured using the methods of above. The beam accelerating voltage was increased from 62 kV–200 kV in increments of 20 kV. At each voltage setpoint, the focus and position of the beam were adjusted over the DUT, an example of the beam repositioning is shown in Figure 4-22. Following the first cross-over of the electron beam, the condenser lens can be altered to select an appropriate spot size which determines the overall diameter and current of the beam. In addition, a selection of physical aperture within the column can modulate the beam current incident to the sample. For this series of irradiations, the input beam current density was held constant at approximately 5 nA/cm$^2$ by tuning the condenser lens aperture and filament emission current appropriately at each accelerating voltage. A series of irradiations with varied beam input current density were also performed, ranging from approximately 4 nA/cm$^2$ to 500 nA/cm$^2$. The beam voltage was held constant at either 62 kV or 200 kV. An extended duration irradiation was performed at 200 kV, 500 nA/cm$^2$ for 1 hr, leading to a target total dose of approximately $1 \times 10^{16}$ cm$^{-2}$. This dose is approximately
equivalent to a 10 mCi radioisotope source for a duration of 1 yr. The I-V characteristics of the DUT were obtained at 10 min intervals to observe possible degradation of the device characteristics as the irradiation proceeded, specifically degradation in power generation.

![Image of a dummy device under test (DUT) illuminated by 10 mA injection current (blue), which sits adjacent to the 62-kV electron beam irradiating the GaN surface (green). This allows for locating the device to adjust the beam position.](image1)

**Figure 4-22:** (left) Image of a dummy device under test (DUT) illuminated by 10 mA injection current (blue), which sits adjacent to the 62-kV electron beam irradiating the GaN surface (green). This allows for locating the device to adjust the beam position. (right)

In addition, Monte Carlo simulations using CASINO2 software were performed to simulate the energy deposition profile within the material layers as a function of beam voltage. This allows for an estimation of the energy absorbed within the active region of the device compared to the nonactive layers, where the active region is defined as the distance from the surface through the depletion width within the i-region of the PIN. The simulated value for energy absorbed can be used to estimate the relative change in produced power, for a given electron beam energy. For these simulations, the material is considered ideal and free from impurities and defects which would alter the electron trajectory. For calculation of the energy absorbed along the depth of the simulated structure, a voltage-dependent value for the number of equally spaced in-plane sections (Δx) was used to maintain near 10 nm resolution along the depth of the sample, with 1 x 10^6 electrons being simulated at each beam voltage.
4.3.3 Results and Discussion

The experimental maximum power produced (MPP) as a function of the beam accelerating voltage is shown in Figure 4-23. The power output of the device is calculated by the product of measured device current and voltage during irradiation in the $0 \text{ V} - V_{oc} \text{ V}$ voltage range.\(^1\) The maximum value in this range is the MPP. The fill factor (FF) of the device is calculated by:

$$ FF = \frac{MPP}{I_{sc}V_{oc}} $$

Where $I_{sc}$ is the short-circuit current, and $V_{oc}$ is the open-circuit voltage. Here, the $FF$ was calculated at each of the beam voltage setpoints, leading to an average $FF_{avg} = 0.56 \pm 0.1$. The MPP is maximum at a beam voltage of 62 kV, with a value of 2.45 $\mu$W/cm\(^2\). This MPP continuously lowers as the beam accelerating voltage is increased up to 140 kV where a slight rise in the MPP occurs until again lowering at 200 kV to a minimum density of 0.45 $\mu$W/cm\(^2\) as shown in Figure 4-23. The slight rise in MPP may be associated with variation in the beam size and current as the beam energy is altered, and possible increased backscattering under the new beam condition. For voltages beyond 140 kV, maintaining the 5 nA/cm\(^2\) current density setting was shown to be challenging due to un-optimal operation of the electron source filament for lower emission currents. Dark current measurements after each irradiation energy shows no degradation in the electrical characteristics. The measured decrease in MPP with increasing beam voltage is consistent with the results of the CASINO2 Monte Carlo simulations, performed under the conditions described previously. Table 16 shows the percentage of energy absorbed within the active region of the device relative to the total energy absorbed for each beam energy simulated.
In order to deduce the change in produced power based on the change in percentage of energy absorbed in the active region, % energy absorbed in the 80 – 180 kV were weighted against the lowest beam energy at 62 kV. From this the simulated MPP was calculated from the simulated change in energy absorbed. The experimental MPP was calculated between each beam energy and the 62 kV baseline. The experimental MPP and the simulated MPP are plotted in Figure 4-24. The simulated MPP trends closely with the measured values for beam voltages below 140 kV. The difference in their values likely derives from the assumption of ideal material in the simulation. Such an assumption omits any interaction between incoming electrons and traps or impurities within the GaN layers, and thereby leads to a larger simulated interaction volume than exists in the real device. This may contribute to the overall lower MPP for the simulation results than the experimental at any given beam energy.

![Figure 4-23: Measured MPP as a function of beam accelerating voltage. The slight rise in MPP post-140 kV is attributed to variation in the beam current, beam area, and instability under high voltage / low current operation.](image)
Table 16: CASINO2 simulation results, calculating MPP based on energy absorbed in active region

<table>
<thead>
<tr>
<th>Energy (kV)</th>
<th>% total energy absorbed in active layer</th>
<th>Experimental MPP (nW)</th>
<th>Simulated MPP (nW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>62</td>
<td>6.1</td>
<td>97.8</td>
<td>97.8</td>
</tr>
<tr>
<td>80</td>
<td>3.2</td>
<td>62.8</td>
<td>50.9</td>
</tr>
<tr>
<td>100</td>
<td>1.8</td>
<td>47.3</td>
<td>29.2</td>
</tr>
<tr>
<td>120</td>
<td>1.2</td>
<td>37.6</td>
<td>19.5</td>
</tr>
<tr>
<td>140</td>
<td>0.9</td>
<td>21.3</td>
<td>14.4</td>
</tr>
<tr>
<td>160</td>
<td>0.7</td>
<td>23.6</td>
<td>11.2</td>
</tr>
<tr>
<td>180</td>
<td>0.6</td>
<td>25.1</td>
<td>9.3</td>
</tr>
</tbody>
</table>

Figure 4-24: Comparison of the change in MPP (ΔMPP) for the experimental data and the CASINO Monte Carlo simulation of energy absorbed. The value for MPP at 62 kV is used as the baseline to normalize the simulated data.

The MPP as a function of beam current is shown in Figure 4-25. A linear increase in the power as a function of input beam current density ($J_{in}$) is observed for both the 62 kV and 200 kV accelerating voltage setpoints, with different rate of increase for each of the voltages. The MPP is shown to increase up to 48.2 µW/cm² with increased beam current density to 177 nA/cm² at 62 kV.
accelerating voltage. The MPP for the 62 kV beam voltage is higher for all input beam current values when compared to the 200 kV beam voltage. The difference in the slope for the two cases is related to the efficiency of energy conversion. The energy conversion rate is markedly higher for the 62 kV electrons which dissipate proportionally more energy in the active layers than a 200 kV electron. For irradiation by 200 kV electrons EHPs are generated and lost well below the active layers, even into the sapphire substrate resulting in a low conversion efficiency and limited power output. The $I_{sc}$ and $V_{oc}$ are plotted against one another in Figure 4-26 and fitted using the following relationship:

$$V_{oc} = \frac{n k T}{q} \ln \left(\frac{I_{sc}}{I_o}\right)$$

Where $I_o$ is the saturation current of the device and is calculated as a fitting parameter for $I_{sc} \gg I_o$ and $T$ is 300 K. Using these values, the ideality factor of the device is measured to be $n = 2.97 \pm 0.12$. This value is in good agreement with previous reports for high performance GaN PIN devices on sapphire substrate.
Figure 4-25: Input beam current density dependence on measured MPP for 62 kV and 200 kV accelerating voltage. The y-intercept is fixed at $MPP(x=0) = J_{\text{in}}(x=0) = 0$ for both of the linear fits.

Figure 4-26: Relationship between $V_{oc}$ and $I_{sc}$ for the beam currents tested at 62 kV.

In order to simulate the long-term radiation response of the GaN PIN device, an irradiation dose test was performed. For this measurement, the electron source was set to 200 kV with a target input current density of 500 nA/cm$^2$ for 1 hr. This current density leads to a target dose of $10^{16}$ cm$^{-2}$.
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2, which is equivalent to exposure from a 10 mCi source for a period of 1 yr. The selected electron energy of 200 keV is near the maximum for a $^{147}$Pm source (225 keV). Table 17 shows the values for $I_{sc}$, $V_{oc}$, and MPP at selected time steps throughout the irradiation. $I_{sc}$, $V_{oc}$, and MPP each show a downward trend as time proceeds from $t = 0$ to $t = 60$ min, with the $I_{sc}$ decreasing from $3.75 \times 10^{-7}$ A to $2.22 \times 10^{-7}$ A, the $V_{oc}$ from 2.16 V to 2.09 V, and the MPP from $4.96 \times 10^{-7}$ W to $2.78 \times 10^{-7}$ W. MPP is plotted as a function of time in Figure 4-27. The decrease appears to be due to a slight decrease in the input beam current throughout the irradiation and not a change in device performance. To confirm this, dark current measurement was made after 1 hr of irradiation which shows negligible change in leakage characteristics and forward resistance. As such, the irradiation conditions appear to be below the threshold for significant lattice defect generation, consistent with previous reports. Other degradation mechanisms such as contact damage, Al wire-bond damage are also not expected to have occurred, given the sustained dark current characteristics and inspection under SEM.

Table 17: Device characteristics during dose test at 200 kV

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>$I_{sc}$ (A)</th>
<th>$V_{oc}$ (V)</th>
<th>MPP (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>-3.75E-07</td>
<td>2.16</td>
<td>4.96E-07</td>
</tr>
<tr>
<td>5</td>
<td>-3.16E-07</td>
<td>2.14</td>
<td>4.10E-07</td>
</tr>
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<td>2.12</td>
<td>3.52E-07</td>
</tr>
<tr>
<td>20</td>
<td>-2.30E-07</td>
<td>2.10</td>
<td>2.93E-07</td>
</tr>
<tr>
<td>30</td>
<td>-2.56E-07</td>
<td>2.11</td>
<td>3.26E-07</td>
</tr>
<tr>
<td>40</td>
<td>-2.01E-07</td>
<td>2.08</td>
<td>2.50E-07</td>
</tr>
<tr>
<td>50</td>
<td>-3.40E-07</td>
<td>2.15</td>
<td>4.47E-07</td>
</tr>
<tr>
<td>60</td>
<td>-2.22E-07</td>
<td>2.09</td>
<td>2.78E-07</td>
</tr>
</tbody>
</table>
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![Experimental MPP measured during the irradiation dose test. The decrease observed in MPP throughout the experiment is attributed to the filament emission current variability described above.](image)

**Figure 4-27:** Experimental MPP measured during the irradiation dose test. The decrease observed in MPP throughout the experiment is attributed to the filament emission current variability described above.

4.3.4 Summary

*In operando* characterization of a direct conversion GaN PIN device for BV application using a high energy (62-200 kV) electron source has been demonstrated here. The large-area planar GaN PIN (0.04 cm$^2$, 17.8 nA/cm$^2$ at -5 V) shows a decrease in maximum power produced (MPP) from 2.45 µW/cm$^2$ to 0.45 µW/cm$^2$ at an approximate input current density of 5 nA/cm$^2$ for increasing beam voltages from 62 kV to 200 kV. In addition, the MPP shows enhancement with increased beam current density up to 48.2 µW/cm$^2$ at 177 nA/cm$^2$ at 62 kV voltage set point. A dose test was performed to a target of $10^{16}$ cm$^2$ which led to no observable change in the dark current characteristics of the device, indicating no observable radiation induced degradation of the device. This unique characterization capability allows for non-destructive sample preparation and fast evaluation of new device designs and structures for the next generation of BV devices coupled with high energy sources.

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4.4 References


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(2013).


Chapter 5 Radioisotope Response and Alpha

Irradiation Induced Damage in GaN PIN Diodes

5.1 \textsuperscript{63}Ni and \textsuperscript{147}Pm Metal Foil Source

Following the investigation of device response under electron beam injection, direct measurement of GaN PIN diodes under exposure to solid metal radioisotope was performed at Army Research Lab (ARL). Both \textsuperscript{63}Ni and \textsuperscript{147}Pm isotopes were deposited by chemical vapor deposition (CVD) onto a Ni metal substrate, with an activity of 15 mCi and 100 mCi, respectively, at the time of production (April 2018 and March 2018, respectively). Measurements were performed in March of 2021. Methodology for device testing in a custom enclosure was developed and executed.

5.1.1 Experimental Setup and Initial Tests

The sources are in a radiation approved lab space at ARL with a Geiger counter adjacent to the sources for radiation leak detection. The lab is monitored by a certified radiation safety officer. The radioactive films are housed in a protective container (“pill box”) with a small opening at its front which acts as the entry way to insert a device under the radioisotope film. Figure 5-1 shows the housing for the two radioactive films available, \textsuperscript{147}Pm and \textsuperscript{63}Ni. The entry way can be seen at the front of each circular container. The higher energy \textsuperscript{147}Pm requires additional shielding surrounding the enclosure in the form of stone blocks pictured on the left in Figure 5-1. The \textsuperscript{63}Ni
and $^{147}$Pm films are estimated to provide an activity of 7.5 mCi and 22.5 mCi to the devices under test (DUT), with a film size of length = 10 mm (square) and diameter = 10 mm (circle), respectively. These activity values account for a presumed 50% loss of $\beta$-particles that emit in the $+z$ direction compared to the $-z$ direction where the DUT is placed, and also the natural loss of activity since production given the source half-life. A metal cage surrounds each pill box and is fixed to the table to ensure that the pill box will not be unintentionally knocked, also shown in Figure 5-1. Custom mounting hardware (referred to as “boat”) was designed to hold the device package and to bring it underneath the radioactive metal film, this is shown below in Figure 5-2. The boat allows for control of the x-y-z position of the DUT. As seen in Figure 5-2 (right) the device package sits on a platform that has an adjustable screw for z-height control bringing the device closer/further from the radioactive film as desired. This boat fixture and adjustable platform was designed using computer aided design (CAD). The boat was then 3D printed and the platform was milled from PVC using machining tools at SUNY Polytechnic Institute. As shown in Figure 5-2 (left), the boat contains grooves that allow the wire-leads to exit the pill box once the boat enters the pill box, allowing in-operando testing of the DUT. A Keithley 6830 source meter and custom python scripting are utilized for I-V measurement under irradiation.
Figure 5-1: Housing for radioactive films, Pm-147 (left) and Ni-63 (right)

Figure 5-2: Custom designed "boat" to hold device package underneath radioactive films
Figure 5-3: $^{63}$Ni experimental enclosure with the sled containing the boat (and device). The labels indicate the variable $X_{\text{dist}}$ which was the distance from the drawer opening to the end of the sled. This was used to determine the optimal position for each device to maximize $I_{\text{sc}}$ during the radioisotope measurements.

Prior to studying the response of a GaN PIN when in proximity to the radioactive foils, it was necessary to determine the x-y position and z-height of the package in the boat that leads to maximum power production i.e., locating the optimal proximity to the source for experimentation. This was done by making small adjustments to the x-y-z position while tracking the $I_{\text{sc}}$ ($V_a=0$) using a source meter, separately for the $^{147}$Pm and $^{63}$Ni pill boxes. Referring to Figure 5-4, the $I_{\text{sc}}$ as a function of boat position relative to the entry way was tracked for device pad 10 / s1507a under the $^{147}$Pm foil. To keep track of the x-position of the DUT a variable $X_{\text{dist}}$ was introduced and measured, which is the distance from the drawer opening to the end of the sled (defined in Figure 5-3). As an example, with the sled all the way into the enclosure opening, $X_{\text{dist}}=0$ and with the sled pulled all the way out of the enclosure door (as shown in Figure 5-3) the $X_{\text{dist}}=$maximum. For the $^{147}$Pm foil enclosure, a maximum $I_{\text{sc}}$ is measured at a position of $X_{\text{dist}} = \sim 12 \text{ mm} - 19 \text{ mm}$
which correlates well with the diameter of the foil, shown in Figure 5-4. The same method was used for the $^{63}$Ni foil, with the results shown in Figure 5-5. The smaller size of the film tracks with the maximum $I_{sc}$ at an $X_{dist} = \sim 11$ mm – 13 mm. This technique was used for all devices tested prior to collecting experimental data, to ensure maximized power output. Figure 5-6 shows how the position can greatly affect the I-V characteristics of the DUT, with the top-most trend indicating the closest proximity to the foil.

![position of S1507a pad 10](image)

*Figure 5-4: Pm-147 exposure of s1507a Pad 10: Tracking $I_{sc}$ as a function of $X_{dist}$ which is defined in Figure 5-3.*
Figure 5-5: Ni-63 exposure of s1507a Pad 10: Tracking Isc as a function of X_{dist} which is defined in Figure 5-3.

Figure 5-6: s1507a Pad 10: under Pm-147 exposure at various distances from foil center to show the large differences in power output based on location of the device in the enclosure

5.1.2 Results and Discussion

Devices with varied dark I-V characteristics were tested under each radioisotope film. The device area and relevant dark I-V characteristics are listed in Table 18. The optimum location
relative to the radioisotope film was located for each, as described above, and the I-V trace was taken manually from 0 V – V_{oc}. The I-V trace was then converted to MPP by the relation P=IV, the results of which are shown in Figure 5-7 and Figure 5-8, below.

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/ Area</th>
<th>I_d @ -5V (A)</th>
<th>J_d @ -5V (mA/cm^2)</th>
<th>R_sh (Ωcm^2)</th>
<th>R_s (Ωcm^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>Square / 0.04 cm^2</td>
<td>-7.9x10^{-10}</td>
<td>2.0x10^{-5}</td>
<td>7.1x10^{8}</td>
<td>2.4</td>
</tr>
<tr>
<td>C3</td>
<td>Circle / 0.0314 cm^2</td>
<td>-1.5x10^{-8}</td>
<td>-4.7x10^{-4}</td>
<td>3.2x10^{7}</td>
<td>8.3</td>
</tr>
<tr>
<td>C4</td>
<td>Circle / 0.0314 cm^2</td>
<td>-6.0x10^{-10}</td>
<td>-1.9x10^{-5}</td>
<td>4.4x10^{7}</td>
<td>6.6</td>
</tr>
<tr>
<td>C7</td>
<td>Circle / 0.0314 cm^2</td>
<td>-2.7x10^{-9}</td>
<td>-8.6x10^{-3}</td>
<td>5.5x10^{7}</td>
<td>3.2</td>
</tr>
<tr>
<td>C10</td>
<td>Circle / 0.0314 cm^2</td>
<td>-3.0x10^{-7}</td>
<td>9.5x10^{-3}</td>
<td>5.6x10^{7}</td>
<td>2.8</td>
</tr>
</tbody>
</table>

As can be seen in Figure 5-7 and Figure 5-8, Device S3 performed the best compared to the other devices in terms of MPP. Referring to Table 18, this improved performance is likely due to Device S3 having an order of magnitude higher R_{sh} and one of the lowest J_d @ -5 V, compared to the other devices. A high shunt resistance is vital for BV application since this will limit power loss through parallel current pathways in the diode (typically in the form of defects). A low J_d is vital as this leakage results in current flow in the opposite direction to the generated current, negatively impacting achievable power. Device C4 had comparable J_d to Device S3 but greater than one order of magnitude lower R_{sh}, which may be the reason for the comparatively lower power produced. A clear trend emerges overall that the devices with the lowest J_d have the highest value for MPP, for exposure to both $^{63}$Ni and $^{147}$Pm foil. The shape of the trend in MPP can be attributed to the R_{sh} where the “squareness” of the power curve correlates to a higher value in R_{sh}. When the
Chapter 5

curve is more rounded, such as Device C4 (red) under $^{147}$Pm, this would typically refer to a lower value of $R_{sh}$. The squareness or roundness of the I-V profile is commonly referred to as the fill-factor (FF), which was introduced in Chapter 4. Note that even for the best performing Device S3, the overall magnitude of the peak in MPP is relatively low, at 0.74 nW/cm$^2$ for exposure to $^{63}$Ni and 2.27 nW/cm$^2$ for exposure to $^{147}$Pm. This is due in part to the limited irradiated area and low source activity.

Given the source activity of 7.5 mCi and 22.5 mCi for $^{63}$Ni and $^{147}$Pm, this corresponds to a total power of approximately $7.55 \times 10^{-7}$ W and $8.26 \times 10^{-6}$ W, respectively. For a $^{63}$Ni source area of 100 mm$^2$ and $^{147}$Pm source area of 78.5 mm$^2$, respectively, this equates to a power density of $7.55 \times 10^{-9}$ W/mm$^2$ and $1.05 \times 10^{-7}$ W/mm$^2$. The amount then exposed to the device mesa (assuming uniform emission magnitude and direction across the width of the metal film radioisotopes and that the isotope is completely parallel to the mesa surface plane), is $3.02 \times 10^{-8}$ W and $4.21 \times 10^{-7}$ W, respectively. The fill factor (FF) defined in Chapter 4 is calculated to be 0.38 and 0.44, respectively. The efficiency of energy conversion for the GaN PIN is then calculated to be 0.098% and 0.022%, given the relation shown in eq. 5-1.

$$\eta = \frac{V_{oc}I_{sc}FF}{P_{input}}$$

5-1

This value for efficiency is low for several possible reasons. The device surface for which the particles are absorbed is not uniform, there are losses within both the thin contact and thick outer p-contact. This is especially true for the lower energy electrons emitted from the $^{63}$Ni film, where most of the kinetic energy is dissipated prior to reaching the active region. The opposite is true for the $^{147}$Pm irradiation, where most of the particles are absorbed well below the depletion
region, and the energy is lost. This deep penetration/loss is the most impactful factor for the efficiency being ~4.5x more for $^{63}\text{Ni}$ than for $^{147}\text{Pm}$. Backscatter of electrons from the metal contact and near the GaN surface is also not considered in the power input calculation. However, for electrons backscattering from the GaN layer, the metal layer can act as a second backscatter event for electrons to reenter the GaN. In addition, the calculation for input power of the radioisotope is using an average particle energy (17 keV for Ni and 62 keV for Pm radioisotope), therefore the true power radiated is likely lower than the estimate given the predominance of lower energy particles that are being emitted.

![Figure 5-7: MPP trend of each device under exposure to Ni-63 foil](image)

*Figure 5-7: MPP trend of each device under exposure to Ni-63 foil*
A more precise determination of the input power would include the spectrum of $\beta$-electron energies rather than assuming all are emitted at $E_{\text{avg}}$. A normalized function for electron distribution is obtained and the relative weight percent of several energies towards the overall activity is calculated. Table 20 shows the relative values for determination of the input power for $^{63}\text{Ni}$ when considering several electron energies. Using this new value for input power in eq. 5-1, the efficiency is then calculated as 0.107% which is an improvement over the original calculation.
\[ \begin{array}{|c|c|c|c|c|c|} \hline E \text{ (keV)} & \% \text{ Prob} & \text{weight \% of total} & \text{Power} f(E) & \text{Power by wt\%} & \text{Total Input Power} \\ \hline 3.70 & 9.10E-01 & 1.61E-01 & 1.64E-07 & 2.64E-08 & 6.89E-07 \\ 5.20 & 8.70E-01 & 1.54E-01 & 2.31E-07 & 3.55E-08 & \\ 8.90 & 7.90E-01 & 1.40E-01 & 3.95E-07 & 5.52E-08 & \\ 12.90 & 6.90E-01 & 1.22E-01 & 5.73E-07 & 6.99E-08 & \\ 16.28 & 6.20E-01 & 1.10E-01 & 7.23E-07 & 7.92E-08 & \\ 21.28 & 5.20E-01 & 9.19E-02 & 9.45E-07 & 8.69E-08 & \\ 25.43 & 4.40E-01 & 7.78E-02 & 1.13E-06 & 8.78E-08 & \\ 31.07 & 3.50E-01 & 6.19E-02 & 1.38E-06 & 8.54E-08 & \\ 40.20 & 2.10E-01 & 3.71E-02 & 1.78E-06 & 6.63E-08 & \\ 44.00 & 1.60E-01 & 2.83E-02 & 1.95E-06 & 5.53E-08 & \\ 52.30 & 8.00E-02 & 1.41E-02 & 2.32E-06 & 3.28E-08 & \\ 62.00 & 1.70E-02 & 3.01E-03 & 2.75E-06 & 8.27E-09 & \\ \hline \end{array} \]

5.2 \(^{63}\text{NiCl}_2\), \(^{147}\text{PmCl}\), and Pu Liquid Source

Several powder-form radioisotopes were held in a liquid solution to be used as the BV power source, including \(^{63}\text{NiCl}_2\), \(^{147}\text{PmCl}\), and Pu-based solution. The benefits of using a liquid source in comparison to a metal film source were described in Chapter 1 and 2. Testing of GaN PIN devices under liquid isotope irradiation was performed at Oak Ridge National Lab (ORNL) in the Fall of 2019. A limited number of experiments were performed given limited time allotted for the visit and resource constraints.

5.2.1 \(^{63}\text{NiCl}_2\) and Pu testing: Developing methodology

A sample detailed in the previous chapter (s0520c) was used for liquid radioisotope testing at ORNL. 3D printed reservoirs were placed overtop of Device S4, S3, and C3. The reservoirs were adhered to the sample using an epoxy, the opening diameter was <1 mm for each due to difficulties in the 3D printing process. Device S4 and S3 were loaded with \(^{63}\text{NiCl}_2\) solution and Device C3 was loaded with Plutonium (Pu). The Pu deposition was intended to observe and track possible
radiation induced damage within the device under high energy / heavy particle bombardment. Pu emits \( >5 \) MeV \( \alpha \)-particles as it undergoes its primary decay. A heating procedure of 4 minutes at 100 °C was developed after each incremental liquid dispensing. This was used to evaporate the solvent and to leave behind the radioactive solid crystal on the device surface. Electrical testing was carried out using a Keithley 6430 sub-femtoampere source meter and custom python script for sweeping voltage from 0 to 4 V. Dark current was measured prior to dispensing the radioactive solutions. Figure 5-9 shows the packaged sample with epoxied reservoirs and the electrical leads.

Device S4 showed minimal response to 10 µL and 30 µL of \(^{63}\text{NiCl}_2\) solution as shown in Figure 5-10a. During handling after 50 µL of dispensed solution, a wire-bond appears to have broken creating an open circuit. Due to the regulation of radiation-exposed materials at ORNL, this device is not allowed to leave the hood, as such repairing the device was not a possibility. Device S3 did not show any power out for up to 100 µL of solution as shown in Figure 5-10b. This may be due to a number of factors including limited active area open to the radioactive material given the geometry of the reservoirs, a greater leakage current than the higher performing Device S4, a large portion of the radioactive crystal adhering to the sidewall of the plastic reservoir therefore being unavailable to the device, and possible blocking of the device surface by the epoxy used to adhere the reservoir to the sample that may have spread out underneath the reservoir. These are all engineering challenges that require attention in future work. Device C3 was coupled with up to 200 µL of Pu and tested for degradation. The below Figure 5-10c shows the IV trace for 10 µL, 30 µL, and 200 µL of dispensed and evaporated Pu solution. No obvious trend emerges in the data obtained as a function of dispensed volume. The author does not rule out the possibility of device and/or wire damage during general handling of the package during dispensing.
of 200 µL, multiple scans were made to track whether or not the device was degrading due to the high energy α-particles. After approximately 40 minutes, no degradation was observed as shown in Figure 5-10d.

Figure 5-9: Photograph of the packaged sample, along with zoomed in views of each device and reservoir.
Figure 5-10: a) IV of Device S4 with dispensed $^{63}\text{NiCl}_2$ showing slight increase in $V_{oc}$ as volume increased to 30 µL b) IV of Device S3 with dispensed $^{63}\text{NiCl}_2$ showing no response up to 100 µL c) IV of Device C3 with Pu, resulting in uncorrelatable current relationship and d) Consecutive sweeps over 40 minutes of Device C3 with 200 µL of Pu, no change is detected

5.2.2 $^{147}\text{PmCl}$ and $^{63}\text{NiCl}_2$ source testing: Engineering improvements to source coupling methodology

Testing of planar GaN p-i-n, planar 4H-SiC p-n, and textured 4H-SiC p-n with $^{63}\text{NiCl}_2$ and $^{147}\text{PmCl}$ radioisotope was carried out at ORNL. Source material was provided by ORNL. The 4H-SiC was provided by Widetronix Inc. The $^{63}\text{NiCl}_2$ and $^{147}\text{PmCl}$ radioisotope were contained within a 5:1 ethanol:methonal solution, separately, that was placed within the device reservoir and baked at 50 °C to promote evaporation of the solvent. The planar GaN p-i-n device has an area of 0.04
cm$^2$ and the SiC p-n devices has an area of 0.203 cm$^2$. Custom reservoirs designed and 3D-printed with solvent resistant plastic as an improvement to the previous generation of testing-as outlined in Section 5.2.1. Figure 5-11 shows the CAD render for the designs considered. The most effective design was chosen to be the deep-angled-cone structure (center of Figure 5-11). This will allow for the liquid isotope to be safely dispensed onto the device with limited loss to the sidewalls of the reservoir, as was the case in the previous attempts. This was adhered to the sample wafer using solvent-resistance adhesive (3M 467MP), with aluminum foil contact pads extending underneath the structure for wire-bonding. The reservoir contained two active devices for testing. The prepared sample is shown in Figure 5-12, with one of the two devices being confirmed to operate upon arrival at ORNL, the adjacent device appeared to have been damaged during transport (broken wire-bond creating an “open”). Due to limited availability of radioisotope, a limited number of tests are performed.

Figure 5-11: Several reservoir designs were considered to contain the liquid radioisotope. Rectangular openings and cone-like openings with short (5 mm) and tall (10 mm) sidewall. Along with structures that contain an additional “lip” for the aluminum contact pad
Figure 5-12: (left) sample with aluminum foil contact pads. These are used so that the wirebond from the device pad to the package does not have to go over or under the reservoir and risk breaking or shorting, respectively. (center) the reservoir is placed down over the foil pads encapsulating two active devices. (right) one confirmed active device upon arrival at ORNL.

5.2.2.1 Output Characteristics and Longevity Test

Within the radiation hood at ORNL, 50 µL of \(^{147}\)PmCl was placed within the reservoir using a micro-pipet and was baked as described previously. An I-V sweep pre-deposition shows I-V characteristics indicative of an operational diode, with the post-deposition measurement showing resistor behavior, as seen in Figure 5-13. An additional 27 µL of \(^{147}\)PmCl (77 µL total) was added and the measurement repeated. The I-V sweep again showed resistor behavior with no turn-on of the device. It appears likely that the device was damaged during the deposition by the pipet, and resulting in conduction through the radioisotope itself. Investigation of the failure mechanism cannot occur since the package must be kept in the radiation hood and can only be handled by the ORNL staff.
Figure 5-13: I-V plot of the planar GaN p-i-n device within the radiation hood at ORNL. The color legend indicates the amount of radioisotope present for a given measurement, with brown being the dark current measurement.

It is clear that there needs to be an adjustment to the design of the package to deal with the unique challenges of a liquid radioisotope deposition method and testing (at ORNL). Multiple wire bonds per contact will introduce redundancy in case of a single wire bond failing. Using an Au ball-bonder (thermal) instead of an Al wire-bond (ultrasonic weld) will also be beneficial. In addition, it may be preferable to place both the p- and n- contact outside of the reservoir enclosure to remove the need for an aluminum (or similar) contact extension (as was shown in Figure 5-12). The team at ARL has worked on the design of a new lithography mask series to attend to these needs, and has developed reservoir formation by using a Nanoscribe Photonic Professional GT two-photon polymerization 3-D printer, which is of higher resolution than the 3D-printed reservoirs and can be placed directly around a single device.

For the planar and textured SiC devices, deposition of $^{63}\text{NiCl}_2$ was performed using the same methods outlined previously. The planar device showed a maximum power produced (MPP) of 0.71 nW and 1179 nW at 100 µL and 200 µL, respectively. The textured SiC device showed 1758
nW and 7901 nW at 100 µL and 200 µL, respectively. These results are unrelated to the GaN BV work presented in this thesis, therefore a full report on these results can be found under ARL Technical Report #ARL-TR-8910.² This work was done in collaboration with Drs. Marc Litz and Randy Tompkins from Army Research Lab (ARL) and Brenda Smith from Oak Ridge National Lab (ORNL).

5.3 Alpha Irradiation of GaN PIN Diodes

5.3.1 Background

There is a continued interest to understand the effects of ionizing radiation on a multitude of semiconductor materials and device architectures. For nuclear batteries, an understanding of the role the radioisotope source irradiation has on defect generation and degradation of the devices electrical output is vital to ensure it remains a useful power source. For other semiconductor microelectronics that are exposed to the harsh radiation conditions found in space, it is similarly necessary to study operation under such conditions to ensure longevity and prevent premature failure where replacement is difficult to nearly impossible. Beyond low-earth orbit (LEO), solar particle events (SPE) and galactic cosmic radiation (GCR) become the dominant ionizing radiation source. The GCR in particular consists of 98% nucleons stripped of their electrons, with 87% being protons, 12% alpha particles, and 1% high-Z nuclei all with energies up to several GeV.³ Alpha particles consist of two protons and two neutrons in a tightly bound configuration. Given the prevalence of proton and alpha particles outside of LEO, studies of device response under such irradiation are of particular interest.
Previous studies have explored irradiation effects of (Al)GaN-based high electron mobility transistor (HEMT) and polarization doped field effect transistor (POLFET) under both high energy alpha and proton flux, in the range of 2 – 18 MeV. There has been limited work looking at the effects of alpha irradiation on GaN PIN device structure. Alpha particles present more of a threat to damage a device as compared to protons given the higher mass and larger size, therefore larger collision cross-section. For AlGaN/GaN HEMTs, it has been shown that a significant reduction in the drain current, gate leakage and transconductance results after irradiation of 2 MeV alpha particles at $10^{13}$ and $10^{14}$ cm$^{-2}$ dose. The degradation is attributed to bulk deep-level defects in the GaN layer and dopant removal in the barrier layer. Another work shows the degradation of AlGaN/GaN HEMT with in situ SiN$_x$ cap layers under 18 MeV alpha irradiation. At a frequency of 100 kHz the saturation drain current was reduced by 32% and 41% following $10^{12}$ and $10^{13}$ cm$^{-2}$ dose. A carrier removal rate was calculated by using the carrier density pre- and post- alpha irradiation and was found to be in the range of 2062–2175 cm$^{-1}$. The carrier concentration was calculated from capacitance-voltage (C-V) measurement. A possible field of application for BV is a low power source for spacecraft components, it is therefore relevant to study effects on the GaN PIN device under irradiation conditions similar to those found in the space environment.

Here, the effect of 4.5 MeV alpha ($\alpha$) beam irradiation on a GaN/sapphire PIN and GaN/GaN PIN diode are studied. Post-irradiation characterization including SEM, XRD, and PL is performed to identify the characteristics of radiation damage and to propose a mechanism for any altered device-level behavior.
5.3.2 Experimental Setup and Beam Conditions

A linear accelerator at Aberdeen Proving Ground (APG) at ARL produces a 4.5 MeV He\(^{2+}\) \(\alpha\)-particle beam with tunable current setpoint. Figure 5-14 shows the system with a DIP mounted in a vacuum chamber in beam line. The purpose of such irradiation is to compare the damage resistance (or lack thereof) of the homoepitaxially grown PIN versus heteroepitaxially grown. The main physical distinction between the two films is the dislocation density, in the range of \(\sim 10^9\) cm\(^{-3}\) and \(\sim 10^5\) cm\(^{-3}\) for hetero- versus homoepitaxial, respectively. However, the dark I-V characteristics of the device are vastly different, as can be seen in Table 21. It is not completely clear why the fabricated devices differ greatly in dark characteristics. Moreover, it is expected that the bulk GaN device should perform better in general.

For each irradiation, the \(\alpha\)-particle beam was tuned such that the area was within the mesa area of the DUT, \(~1\) mm diameter beam for Device C4 and \(~0.19\) mm diameter beam for Device 16 (the beam could not be made smaller than the DUT area). Table 22 below shows the relevant data for beam condition and activity for the completed irradiations. Given the difference in the device area, the beam current was selected to achieve a similar activity density, to make for a comparative study. Each device was irradiated for \(~90\) minutes with I-V measurement made in regular intervals.
Figure 5-14: Alpha beam linear accelerator (left) with a device package mounted in the vacuum chamber in the beam line (right)

Table 21: Devices irradiated by the alpha beam

<table>
<thead>
<tr>
<th>Device #</th>
<th>Mesa Shape/Area</th>
<th>$I_d @ -5V$ (A)</th>
<th>$J_d @ -5V$ (mA/cm$^2$)</th>
<th>$R_{\text{sh}}$ (Ωcm$^2$)</th>
<th>$R_s$ (Ωcm$^2$)</th>
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</thead>
<tbody>
<tr>
<td>C4</td>
<td>Circle / 0.0314 cm$^2$</td>
<td>-6.0x10$^{-10}$</td>
<td>-1.9x10$^{-5}$</td>
<td>4.4x10$^7$</td>
<td>6.6</td>
</tr>
<tr>
<td>16</td>
<td>Circle / 2.84x10$^{-4}$ cm$^2$</td>
<td>-5.77x10$^{-6}$</td>
<td>-2.0x10$^1$</td>
<td>5.60x10$^2$</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Table 22: Beam conditions (4.5 MeV) during irradiation

<table>
<thead>
<tr>
<th>Device #</th>
<th>Beam current (nA)</th>
<th>Ion Flux (He$^{2+}$/sec)</th>
<th>Activity (mCi)</th>
<th>Activity density (mCi/mm$^2$)</th>
<th>Beam area (mm$^2$)</th>
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</thead>
<tbody>
<tr>
<td>C4</td>
<td>10</td>
<td>3.13x10$^{10}$</td>
<td>8.45x10$^2$</td>
<td>1.08x10$^3$</td>
<td>0.785</td>
</tr>
<tr>
<td>16</td>
<td>1</td>
<td>3.13x10$^9$</td>
<td>8.45x10$^1$</td>
<td>2.98x10$^3$</td>
<td>0.0284</td>
</tr>
</tbody>
</table>
Chapter 5

5.3.3 Results and Discussion

The result for the irradiations is summarized in Figure 5-15. The forward current (FWDCurr) is defined as the value of current at +5 V, with a compliance of 50 mA. The $V_{oc}$ is defined as the voltage for which net current flow is zero. The $I_{sc}$ has been defined previously and is also plotted here. The data contained within these plots were captured in situ during irradiation and directly following irradiation, with the x-axis indicating scan numbers (scan #) throughout the 90-minute exposure for each. The plots are sectioned out into pre-irradiation (blue), irradiation (green), and post-irradiation (red). Device 16 (GaN/GaN PIN) shows a drastic and irreversible change in the FWDCurr, which indicates damage to the device that has altered the resistance of the diode under forward bias. Device C4 shows only a slight reduction in the FWDCurr, which restores to its' original value post-irradiation. The $I_{sc}$ and $V_{oc}$ of Device 16 also decrease by a significant amount during the irradiation. The irreversibility of this reduction cannot be determined without re-irradiating the device under the same condition and re-measuring the variables at the start of the irradiation. Device C4 also shows a reduction in $I_{sc}$ and $V_{oc}$. The decrease in $I_{sc}$ is similar to Device 16, at approximately an order of magnitude, however the decrease in $V_{oc}$ is much less from 2.1 V to 2.0 V, compared to ~ 1.8 V to 1.0 V for Device 16. The decrease in $V_{oc}$ for Device 16 correlates with the measured reduction in FWDCurr and is likely also due to the irreversible damage incurred from the $\alpha$-particle bombardment.
Post-irradiation the samples were removed from the vacuum chamber while intermittent I-V measurements continued, this is the “Dark-IV post” (red) portion of the plots in Figure 5-15. For Device C4, it takes a few minutes for the FWDCurr value to return to its starting value following the irradiation, between scan# 20-22. The opposite occurs for Device 16, where a continued decrease is observed. Figure 5-16 compares the full I-V traces pre- and post- irradiation for each device. Device C4 shows a near identical trace, while Device 16 shows a dramatically
altered current profile. As plotted previously, the decrease in forward current is observed along with a reduced threshold voltage ($V_{th}$) to $< 1 \text{ V}$.

\textit{Figure 5-16: Pre- (blue) and Post- (orange) irradiation I-V trace for Device C4 (left) and Device 16 (right)}
Given the differences in the in-situ data for each irradiated GaN PIN device, it was important to characterize and compare the post-irradiated devices thoroughly to understand the type and mechanism of damage (or lack thereof). Both devices were characterized by scanning electron microscope (SEM), photoluminescence (PL), x-ray diffraction (XRD), and capacitance-voltage (C-V). The SEM characterization on Device C4 indicates that the irradiated region has undergone a change in bulk and/or surface resistivity, as the secondary electron intensity is vastly different than the unirradiated regions. This indicates either 1) the electron recombination is higher in this region due to defects resulting in reduced emission to the detector and/or 2) electrons are allowed to collect and “charge” the surface resulting in limited emission. This can be seen in Figure 5-17, top, in the form of a region with darker contrast. For comparison, an unirradiated device was also imaged by SEM and is shown Figure 5-17, bottom. This feature is also visible to the eye as a brown color on the device, shown in Figure 5-17, left. Device 16 (GaN/GaN PIN) was also imaged in SEM, shown in Figure 5-18. Although damage has been detected in the I-V characteristics, there does not appear to be any darkening contrast relative to other areas of the sample, like that of Device C4. It is worth reminding here that the full device mesa of Device 16 was exposed to the beam given the smaller device area, whereas with Device C4 the beam was contained within the mesa itself.
Figure 5-17: SEM of Device C4 post-alpha irradiation. (top) The dark contrast indicates the region that was bombarded and likely is attributed to a change in resistivity in the film. (bottom) an unirradiated device for comparison.

Figure 5-18: SEM of Device 16 (left) and an unirradiated device (right) for reference. No apparent contrast difference between the two devices.
To better quantify the properties of this region, the device was subjected to PL measurement. PL spectra were obtained at room temperature (RT) within several regions surrounding the irradiated/browned region, shown in Figure 5-19. The absorption depth of the 325 nm laser is approximately 100 nm in GaN, so this measurement is mostly of the p-GaN surface layer. The off-mesa region (purple), on-mesa / no beam (black), on-mesa / beam-center (blue), and on-mesa / beam-edge (red). There are three distinct emission peaks of interest, the near band edge (NBE) at ~3.44 eV, the blue luminescence (BL) at ~2.88 eV and the yellow luminescence (YL) peak at ~2.25 eV. Typically, the BL peak will only appear in p-GaN material where the Mg dopant is present and is a reasonable indicator for a p-type material. Referring to the on-mesa / no beam area (black) the BL emission is present which agrees with the expectation that there is a p-GaN surface layer present for the PIN device. When scanning the region where the alpha beam was present (blue, red), the BL emission is suppressed and no longer observed. This may indicate that the Mg dopants in this region have been displaced or Ga-N- vacancies are present which act as traps for carriers. Interestingly, the beam-exposed regions (blue, red) now closely resemble the n-GaN off-mesa spectrum (purple) with dominant NBE and YL peaks. A comparison was made between the on mesa / no beam (black) region on Device C4 with an unirradiated device mesa, this is shown in Figure 5-20. The RT-PL emission for this region on Device C4 closely resembles that for the unirradiated mesa surface, which indicates that the region has been completely unaffected by the α-beam that was positioned directly adjacent. This indicates the conclusion that, although the majority of Device C4 mesa is damaged from the α-beam, there are still regions free from damage (where the beam did not irradiate) that allow nominal current conduction. Hence this may be the reason for the post-irradiation I-V characteristics returning to the original values, as
shown in Figure 5-16. Device 16 shows a similar RT-PL spectrum, where the BL emission is suppressed within the irradiated region (the full mesa area in this case). Referring to Figure 5-21, a strong BL emission peak is present in an adjacent device mesa that has been untouched by the α-beam.

Figure 5-19: PL spectra for various regions surrounding the irradiated portion on Device C4
Figure 5-20: Comparing the unirradiated region of Device C4 with an unirradiated device mesa. The emission peaks present in the RT-PL are identical.

Figure 5-21: Comparing unirradiated device with the Device 16 irradiated mesa, RT-PL.
To assess any bulk crystalline alterations such as lattice expansion or deformation, XRD was performed. Figure 5-22 shows the symmetric-RC and 2θ-ω scans of Device C4. The data indicates there is minimal differences in the profile for all the positions tested as tabulated in Table 23. However, for Device 16 there is a clear change in both the symmetric-RC and 2θ-ω as compared to an unirradiated device / area. Referring to the red line in Figure 5-23, the irradiated area differs slightly in shape and has a reduced FWHM compared to the other positions. For the 2θ-ω the shape changes drastically as compared to the other positions, with a satellite peak that protrudes around 150 arcsec which may indicate a partial deformation in the lattice. Note that Table 24 is incompletely filled, this is due to the numerous peaks present that result in a single gaussian peak fit being unattainable.

*Table 23: XRD results for Device C4, legend provided below*

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<th>ID</th>
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<th>FWHM</th>
</tr>
</thead>
<tbody>
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<td>_01</td>
<td>16.90</td>
<td>0.0884</td>
</tr>
<tr>
<td>_02</td>
<td></td>
<td></td>
</tr>
<tr>
<td>_03</td>
<td>16.90</td>
<td>0.0865</td>
</tr>
<tr>
<td>_04</td>
<td>2.60</td>
<td>141.32</td>
</tr>
<tr>
<td>_05</td>
<td>5.93</td>
<td>140.62</td>
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<td>16.90</td>
<td>0.0868</td>
</tr>
<tr>
<td>_09</td>
<td>-0.97</td>
<td>141.25</td>
</tr>
</tbody>
</table>

- s1507_01 -- non-irradiated mesa, symmetric RC
- s1507_02 -- non-irradiated mesa, asymmetric RC
- s1507_03 -- irradiated mesa (brown area), symmetric RC
- s1507_04 -- irradiated mesa (brown area), symmetric omega-2theta
- s1507_05 -- irradiated mesa (non-brown area), symmetric omega-2theta
- s1507_06 -- off mesa (near irradiated mesa), symmetric omega-2theta
- s1507_07 -- off mesa (near irradiated mesa), symmetric RC
- s1507_08 -- irradiated mesa (non-brown area), symmetric RC
- s1507_09 - non-irradiated mesa, symmetric omega-2theta
Figure 5-22: XRD results for Device C4 (GaN/Sapphire PIN) with symmetric rocking curve (left) and 2theta-omega (right)

Table 24: XRD results for Device 16, legend provided below

<table>
<thead>
<tr>
<th>ID</th>
<th>Peak Center</th>
<th>FWHM</th>
</tr>
</thead>
<tbody>
<tr>
<td>.01</td>
<td>17.21</td>
<td>0.025</td>
</tr>
<tr>
<td>.02</td>
<td>17.22</td>
<td>0.032</td>
</tr>
<tr>
<td>.03</td>
<td>17.22</td>
<td>0.031</td>
</tr>
</tbody>
</table>

s1419_01 – irradiated mesa, symmetric omega-2theta
s1419_02 – irradiated mesa, symmetric RC
s1419_03 – nonirradiated mesa, symmetric omega-2theta
s1419_04 – nonirradiated mesa, symmetric RC
s1419_05 – off mesa, symmetric omega-2theta
s1419_06 -- off mesa, RC
5.3.4 Summary

Heteroepitaxial and homoepitaxial GaN PIN devices were subjected to focused 4.5 MeV α-beam irradiation with similar input current. The GaN/GaN PIN (Device 16) shows a permanent degradation in forward current because of the irradiation, while the GaN/sapphire PIN (Device C4) restores forward current once the irradiation ceases. The GaN/sapphire PIN shows a discoloration within the irradiated area, which upon PL measurement, correlates to a disappearance of the blue luminescence (BL) emission peak at ~2.88 eV. The BL is related to the Mg-dopant atom in GaN which may suggest displacement of the Mg dopant from the acceptor position and/or presence of increased compensating N-vacancy density from the alpha bombardment. The GaN/GaN PIN also shows disappearance of the BL following irradiation, and also a large reduction in the YL emission. It is possible that the restoration in forward current is a result of conduction through the regions of the device mesa that were not directly irradiated with the beam (non-
discolored regions). The PL in this region indicates no change in the emission peaks when compared to the non-irradiated mesa. Though the GaN/GaN PIN does not show a similar visible discoloration, the PL spectrum is identical in characteristic to the GaN/sapphire PIN. For the GaN/GaN PIN the entirety of the mesa area was irradiated given the smaller device size and limitation in size of the α-beam. The XRD spectrum for the GaN/GaN PIN post-irradiation shows a satellite peak that is indicative of lattice deformation. The FWHM of the GaN/sapphire PIN is large (due to higher dislocation density) and any lattice deformation may be hidden. Given the large differences in pre-irradiation dark I-V characteristics between the two devices, it is difficult to compare the differences in post-irradiation I-V in direct association to the specific substrate. To allow for such comparison the relative device area to irradiation area and the starting dark I-V characteristic need to be controlled.

5.4 References


Chapter 6 Core-shell 3D + Planar PIN by Selective Area Growth

6.1 Selective Area Growth (SAG) MOCVD

6.1.1 Overview and Fundamentals

Selective area growth (SAG), also known by selective area epitaxy (SAE), is a method employed in CVD for growth of a given semiconductor within isolated regions of a substrate. This is achieved by covering the substrate with a masking material (typically SiOₓ, SiNx, or W) then etching away regions of the mask to isolate certain areas and allow selective precursor absorption and growth within opened “windows”. A key attribute of the mask material is that it inhibits surface nucleation and undergoes limited decomposition under growth conditions. The growth direction (vertical vs. lateral) and facet stabilization can be controlled by tailoring the growth conditions accordingly (temperature, pressure, V/III). The SAG process is schematically depicted in Figure 6-1, where the different pathways for the net source flux are from the gas-phase boundary layer (J_{top}), diffusion along the mask and gas-phase diffusion above the substrate (J_{sub}) and diffusion along the sidewall surface of the growth structure (J_{sw}). Net source flux refers to the difference between absorbed and desorbed species.¹ There is a characteristic collection range (r_{sub}) for which adatoms will reach the mask opening (unmasked region) which accounts for both gas-phase and surface diffusion. Generally, this diffusion length should be greater than the largest distance between mask openings to limit the probability of adatom desorption or unwanted mask nucleation. The main driving force for diffusion towards the opening is the concentration gradient.
that results from consumption and incorporation of species in the unmasked regions. A topic of contention has been whether the dominant pathway for diffusion is along the mask surface or in the gas-phase above the mask. It seems to be the consensus that both occur and are important, where short-range diffusion (1-10 µm) from the mask contributes to smooth sidewall facets, while longer range gas-phase diffusion (>10 µm) leads to growth rate enhancement.\(^2\) This was experimentally demonstrated by Mitchell et al., where trench features were etched into the substrate in between mask openings as a means to inhibit diffusion of species along the mask surface to the opening. The growth rate profile was nearly identical to growth without the trench features impeding source diffusion.\(^2\)

Another important characteristic of the mask is the fill factor, which is defined as the ratio of unmasked area to total area of the substrate. The fill factor will greatly affect the growth rate, where a lower fill factor leads to increased growth rate. Consider the assumption of source flux conservation, where there is the same amount of source available for planar growth and SAG growth. Therefore, for a smaller fill factor, the additional source flux available for a given unmasked feature leads to a growth rate enhancement. Furthermore, it is observed experimentally that the volumetric growth rate is nearly identical for planar versus SAG.\(^3\)
A model was developed by Coltrin, et al. to describe the transport of reactants to the SAG surface and can predict whether the limiting regime is transport, reaction-rate, or intermediate. A summary of this model is provided here for reference, as it provides a useful viewpoint for describing the fundamentals of SAG. Starting with simple thin-film growth, the gas-phase concentration of reactant species is shown to drop off linearly within the boundary layer towards the sample surface. This results in the boundary condition shown in eq. 6-1, where $k$ is the rate constant (defined in eq. 6-2), $C_s$ is the gas-phase concentration of the limiting species at the surface, $C_i$ is the inlet concentration, $D$ is the diffusion constant, and $\delta$ is the boundary layer thickness taken to be $\sim 0.7$ cm (from simulation). Upon rearranging the equation, the variable $Da$ is introduced which is a dimensionless Damköhler number that determines the limiting regime. $Da$ is defined in eq. 6-3. The reaction probability or “sticking coefficient”, $\gamma$, is assumed to be unity for this calculation but is varied in upcoming calculations.

\[
kC_s = D \frac{C_i - C_s}{\delta} \rightarrow \frac{C_s}{C_i} = \frac{1}{1 + Da}
\]

\[6-1\]
\[ k = \gamma \sqrt{\frac{RT}{2\pi W}} = 15850 \frac{cm}{s} \]

For \( T = 1323 \, K, W = 69.7 \, \frac{g}{mol} \)

\[ Da = \frac{k\delta}{D} \]

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From the equivalence shown in eq. 6-3, it follows that when \( Da >> 1 \) the reaction probability is high, and/or \( \delta \) is high, and/or the diffusion coefficient is small. This would represent a transport-limited regime where the reaction-rate is large relative to transport. In the opposite case where \( Da << 1 \), this would represent a reaction-rate limited regime where the transport is large relative to reaction-rate related variables. A formula for growth rate is shown in eq. 6-4, which is independent of reaction probability when \( \gamma >> 0.01 \) and shows linear decrease with \( \gamma << 0.001 \). This agrees well with the simulation result shown in Figure 6-2. Now, considering a SAG type layout, a set of boundary conditions for the various sources of flux towards the exposed region is shown (Figure 6-3, left). The fill factor is defined by the variable \( \theta = x_0 / x_{\text{max}} \). A total steady-state flux is calculated that is related to the so-called SAG efficiency, \( \varepsilon \), in eq. 6-5. This shows that when the efficiency is unity, the same amount of material is deposited in the exposed region that would be available in the entire region without a mask, a result that was mentioned previously and which is often achieved experimentally (equivalent volumetric growth).

\[ G = \frac{kC_i}{1 + Da} \]
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**Figure 6-2**: Reactant concentration as a function of boundary layer height for varied surface reaction probability, simulation results align with thin film model given in eq. 6-1.

**Figure 6-3**: (left) Schematic of boundary conditions for SAG-type growth calculations and (right) the SAG efficiency as a function of pattern fill factor for varied Da.

\[
\varepsilon = \frac{J_{\text{tot}}}{x_{\text{max}} G}
\]

The authors then calculate the SAG efficiency as a function of fill-factor for various values of Da shown in Figure 6-3 (right), which as mentioned defines whether the system is transport or kinetic-limited. Extending the model developed for thin-film growth to SAG, using an effective or “average” rate constant \(k’=\theta k\) leads to an effective Damköhler number, Da’ and therefore an effective growth rate, G’. Taking these effective variables and recalculating eq. 6-1, 6-3 and 6-4 the SAG efficiency can then be described by eq. 6-6. This explains the trends shown in Figure 6-3.
In this plot, as the Damköhler number is decreased the value of fill factor for which the SAG efficiency begins to drop off, increases. For Da = 1000 (transport-limited), the SAG efficiency remains unity from a fill factor of 1 to around 0.05. For Da = 100, the SAG efficiency is now unity only up until 0.5 for which it begins to drop off. During experimental SAG, by obtaining data for SAG efficiency (from structure volume relative to equivalent planar volume) one may obtain the value of Da for a system therefore determining the limiting regime for a given fill factor. As an example, Figure 6-4 shows experimental data of SAG efficiency vs fill factor for InGaAs and InP (varied temperature), with the fitted line from eq. 6-6 determining a value for Da.

\[
\epsilon = \frac{G'}{G} = \frac{\theta (1 + Da)}{1 + \theta Da}
\]

Figure 6-4: Experimental data from growth of InGaAs and InP fit with eq. 6-6 to determine Da.

### 6.1.2 Applications

SAG has been employed for multiple semiconductor materials such as (Al)GaN, (In)GaAs, and InP.\(^5\text{-}^8\) The first reported utilization of the SAG technique was in 1965 for the growth of GaAs structures by vapor phase epitaxy a thermodynamically equilibrium process.\(^8\) It was not until 1982
that the technique was utilized for MOCVD of GaAs due to the challenges of unwanted mask nucleation and dealing with the unique chemistry found in this process. SAG of GaN by MOCVD was first reported in 1994 using an SiO₂ mask. An application of the method known as lateral epitaxial overgrowth (LEO) has shown great success in reducing average dislocation density in the overgrown film by several orders of magnitude compared to the bulk. For GaN, LEO has been shown to reduce the dislocation density from \(10^9\) cm\(^{-3}\) in standard heteroepitaxial growth to \(10^6\) cm\(^{-3}\). For growth along c-plane, this is achieved by lateral growth along semipolar/nonpolar growth fronts for which threading dislocations (TDs) from the underlying [0001] front cannot traverse due to their starting line direction. This is useful when growing material on a highly mismatched substrate. The LEO process leads to nearly defect free material in the “wing” regions of the overgrowth, thus an overall lower average dislocation density in the coalesced film. For non-cubic materials where asymmetry exists in crystal structure, an important consideration for LEO is the feature orientation relative to the underlying substrate. The same is true for lateral overgrowth on an existing seed, such as is demonstrated in this work, where the winged material should ideally be near-defect free.

For the advancement of LED technology, 3-D III-Nitride structures are being utilized to increase the output light intensity compared to a planar device. This partially results from the increased surface area for emission from 3D structures. In cases where a 3D LED structure with semi-polar/non-polar sidewalls is used, presence of the so-called quantum-confined stark effect (QCSE) is reduced/eliminated which will allow for thicker quantum wells (QW) to be grown. The QCSE is a result of polarization-induced separation of the electron and hole wavefunctions which can reduce the radiative recombination efficiency.
6.1.3 Sample Preparation and Processing

MOCVD-grown n-GaN templates, as described in Chapter 3.3, are used as the substrates for subsequent SAG growth. A standard mask layer stack of 50 nm SiO$_2$ / 50 nm SiN was deposited by plasma enhanced chemical vapor deposition (PECVD) at 250 °C / 300 °C, respectively. Prior to deposition the epi-surface was cleaned with acetone, IPA, and DI rinse. As compared to a single SiO$_2$ surface layer, the dual SiO$_2$/SiN has been shown to promote decreased mask nucleation. Noteworthy also that we have previously observed that use of the SiO$_2$/SiN mask during SAE results in Si incorporation ($7 \times 10^{18}$ cm$^{-3}$) in the overgrown structure, likely originating from partial decomposition of the mask. This would be an issue if attempting to grow unintentionally doped structures- however, here the seed structures are intentionally n-type (n-GaN). The SiO$_2$/SiN mask has also shown to lead to substantially decreased O incorporation compared to a standard W-metal mask (at $2 \times 10^{17}$ cm$^{-3}$ versus $7 \times 10^{18}$ cm$^{-3}$) and at the same level to a planar comparison sample ($3 \times 10^{17}$ cm$^{-3}$). The measured concentrations of [Si] for SiO$_2$/SiN-masked sample and [O] for W-masked sample results in carrier concentration of $8 \times 10^{18}$ cm$^{-3}$ and $1.59 \times 10^{19}$ cm$^{-3}$.

Dependent on the mask geometry and the desired dimensions, either AZ5214 or 1813 photoresist was patterned using standard photolithography techniques. Etch-windows were created through development in 300MIF solution. Samples are then etched by fluorine based RIE to form openings (unmasked regions) in the mask. A substantial over-etch (50%-100%) in RIE is applied to ensure complete removal of the material in the openings prior to epitaxy. A schematic of the SAE process flow is shown in Figure 6-5 for reference. The mask layout consists of either extended line features or circular features of various sizes, the defined pitch (p) and width (w) are labeled in
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Figure 6-6. Throughout the upcoming sections, the mask dimensions will be noted in the analysis for a given growth.

![Figure 6-5: Schematic of sample processing for SAE.](image)

![Figure 6-6: Schematic of mask layout with definition of mask opening (width, W) and distance between features (pitch, P).](image)

6.2 Selective Area Growth: Core-shell 3D + Planar Growth

6.2.1 Facet stabilization of n-type GaN seed by altered V/III

6.2.1.1 3D Fin Seed

To enable a 3D core-shell device architecture, a high aspect ratio seed microstructure is required for subsequent overgrowth. As mentioned, a SiO₂/SiN dual dielectric layer was employed for the mask, targeting u-GaN fin arrays of varying pitch and width. It is the expectation that the SAE structure will be n-type due to the Si incorporated during growth, as discussed in the previous
Section. Here, n-GaN is referring to growths where silane was intentionally introduced during growth. The goal is to obtain highly ordered, high aspect ratio 3D microstructures with smooth non-polar sidewalls (90° ⊥ [0001]). Several dimensions were available to study with the layout shown in Figure 6-7. The growth procedure follows a two-step method, with a brief filling step followed by a longer 3D vertical growth step. The differences in growth condition between the two layers are shown in Table 25. The cross-sectional SEM in Figure 6-8 shows the u-GaN fin arrays, with the presence of angled semipolar planes with smooth top c-plane surface. The lateral dimensions of the structures were obtained by SEM and vertical dimension was obtained by application of focused ion beam (FIB) milling. Using these measurements, the lateral growth was plotted up against both pitch (p) and mask opening (w). A general trend emerges with an increased lateral growth with both increased area of the masked regions and increased mask opening, shown in Figure 6-9. FIB cross section imaging confirmed that the sidewalls were semi-polar, due to the approximately 63° sidewall angle. The vertical dimension of the SAG feature was dependent on the opening width and ranged from ~700 nm (for 10 μm opening) to ~1100 nm (for 5 μm opening), reiterating the concept of enhanced growth rate for lower fill factor.

Table 25: General two-step growth layout, with specific values for growth I

<table>
<thead>
<tr>
<th>Growth Parameter</th>
<th>Filling Step</th>
<th>3D Vertical Step</th>
</tr>
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<tbody>
<tr>
<td>Pressure (Torr)</td>
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<td>100</td>
</tr>
<tr>
<td>Pyro Temp (In/Out)</td>
<td>1020/1020</td>
<td>1020/1020</td>
</tr>
<tr>
<td>H₂ (sccm)</td>
<td>18000</td>
<td>23500</td>
</tr>
<tr>
<td>N₂ (sccm)</td>
<td>8900</td>
<td>8900</td>
</tr>
<tr>
<td>NH₃ (sccm)</td>
<td>8000</td>
<td>2500</td>
</tr>
<tr>
<td>TMGa (sccm)</td>
<td>52</td>
<td>52</td>
</tr>
<tr>
<td>V/III</td>
<td>2100</td>
<td>660</td>
</tr>
</tbody>
</table>
Figure 6-7: Low magnification SEM image of the feature layout used for fin growth (following SAG), to display the large-scale uniformity achieved here.

Figure 6-8: SEM images of Growth I arrays to show semipolar faceting. The darker contrast SiN/SiO₂ mask surrounds the bright GaN fins. Sidewalls of fin features show higher contrast indicating high secondary electron emission intensity.

Figure 6-9: Total lateral growth as a function of the mask opening (left) and pitch (right)
A second growth was performed (Growth II) with a further reduction of the V/III. A lowering of the V/III ratio to ~430 was achieved by lowering the NH$_3$ flow to 1/5 the standard planar value, from 8000 sccm to 1600 sccm. To ensure the same total hydride flow the H$_2$ flow was increased to compensate for the lowered NH$_3$. The growth was performed on the identical SiN/SiO$_2$ mask as the previous run. All other conditions were kept the same as indicated in Table 25. SEM characterization shows that Growth II resulted in suppression of the semipolar sidewall facets and stabilization of vertical non-polar sidewalls, shown in Figure 6-10. Cross sectional SEM was performed for further analysis. The lateral growth dimension is ~200 nm (on one side) compared to ~1.8 µm vertical growth dimension, showing the capability for higher aspect ratio structures. The growth time was 30 minutes, meaning 60 nm/min vertical growth rate, and 6.6 nm/min lateral growth rate (~9:1 vertical to lateral) under these conditions.

Figure 6-10: Cross sectional SEM of microstructures from Growth II. Confirms smooth vertical non-polar sidewalls and uniform feature set. (left) and (middle) show the difference in height based on fill factor.
Figure 6-11: Further analysis of Growth II. The height of structures is dependent on initial opening width and spacing between stripes.

The conditions for u-GaN Growth II were used for the n-GaN fin seed growth with the addition of silane (SiH₄). Silane flow rate of 10, 50, 90 sccm were tested, as it has been shown that increased silane adsorption at the sidewall surface can lead to enhanced vertical growth rate. However, in this work the resulting aspect ratio (vertical:lateral) for the seed-structures grown under 10, 50, and 90 sccm silane flow was nearly identical. A cross-section of the structure grown with 10 sccm of silane is shown in Figure 6-12. In addition, there was no observable change in the height amongst the n-GaN set, the height is comparable to the u-GaN feature grown previously, with ~1.7 μm vertical growth, and ~0.2 μm lateral growth, shown in Figure 6-12. This aspect ratio is the same as the u-GaN seed at ~9:1. The effect of increased silane flow as described in literature does not appear to be playing a role here-possibly due to the lower aspect ratio of the structures studied here. The mechanism appears to be the increased diffusion length on the sidewall surface from the selective interaction with m-plane surface and formation of a thin SiN layer. With smaller dimensions and higher aspect ratio, a greater proportion of the full structure surface area is the nonpolar sidewall.
Figure 6-12: (left) n-GaN seed (10 sccm silane), and (right) u-GaN seed show nearly identical vertical and lateral growth rate.

An important consideration for the design of 3D+planar device layout is the increased growth rate at the array edge. The increased growth rate is a result of additional source flux (adatoms) from the large planar region where the collection range is much larger than between mask openings. The vertical growth rate is maximum at the edge then decreases moving towards the center of the array. This can be seen in Figure 6-13, where the increased vertical growth rate leads to severe degradation of material quality with the formation of large-scale defects. This observation also confirms that the adatoms are predominantly diffusing in the gas-phase above the features, because the source flux from the mask between features remains the same. The only difference being the distance from the array edge.
6.2.1.2 3D Pillar Seed

Lower dimensional n-GaN pillar microstructures seeds have been optimized to attain high-aspect ratio with nonpolar sidewalls. From reported work on nano- and micro-structure growth it is known that to stabilize the nonpolar plane the V/III precursor ratio must be low.$^{11,14}$ Given the difference in mask fill factor and geometry it was anticipated that the condition for fin seed growth in the previous section would not translate directly for lower-dimensional pillar seeds. The growth optimization reported here includes employing a pulsed growth method to achieve a low effective V/III precursor ratio. The previously reported two-step growth mode (fill layer planar growth + vertical growth) is used for all growth experiments reported here. The standard feature size for the SiN (50 nm) / SiO$_2$ (50 nm) SAG mask are 2-micron diameter circular openings with a ~3.5-micron gap between features. The processed SAG mask is shown in Figure 6-14 with the brighter contrast region being the GaN epi-surface.

*Figure 6-13: (left) The edge of the fin array in the n-GaN seed growth showing increased height towards the edge. The increased growth rate induces large-scale defects in the seed (right).*
Figure 6-14: As-processed SAG mask - circular openings to GaN surface, separated by ~3.5-micron

The V/III precursor ratio was adjusted for the vertical growth step, to understand the effect on facet stabilization and relative growth rate (GR). First, utilizing a continuous growth mode, in which precursor flow is uninterrupted throughout the duration of the growth layer, three V/III ratios were selected at 430, 215, 116. The NH₃ precursor flow was reduced to decrease the V/III ratio, except for the 116 V/III condition as the NH₃ was approaching the threshold for the mass flow controller (MFC) accuracy. To achieve the 116 V/III it was necessary to increase the TMGa flow to 90 sccm. The resulting microstructures are shown below in Figure 6-15. At V/III of 430 the structure consists solely of semipolar planes angled at ~62° (similar to Growth I of the fin structures) with the c-plane almost non-existent. This means that the c-plane growth rate was much higher compared to the other planes. When reducing the V/III by 50% to 215 a consistent nonpolar 90° facet appears at the base of the microstructure. This is towards the proposed/desired 3D BV seed shape. With a further reduction of the V/III (by an additional ~50%) the same structure results, which implies that the V/III ratio in this regime provides this structure as the equilibrium shape. Given the limits of the MFC(s) that are controlling hydride sources, the V/III cannot be accurately reduced further under a continuous flow mode. A shift to a pulsed growth mode was made to allow
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for a low effective V/III ratio by switching the flow of each precursor in the reactor at any given time – this is synonymous to reducing the NH\textsubscript{3} flow or increasing the TMGa flow for a continuous mode. The actual V/III ratio is unquantifiable given that the ratio of adsorbed precursors on the surface of the sample between and during the pulses is unknown.

Figure 6-15: SEM of microstructures resulting from the continuous mode SAG with continued reduction in V/III ratio during the vertical growth step.
Three conditions were tested for the pulsed growth mode at a fixed number of pulses at 50. The TMGa flow during the Ga-pulse (5s) was kept the same for each, with a change in the NH$_3$ flow during the N-pulse (10s) of 1.6 slm, 5 slm, and 8 slm. Figure 6-16 shows the SEM images of the resulting structures. The 1.6 slm N-pulse condition led to a V/III below the threshold for uniform steady-state growth and resulted in a randomly oriented – defective structure. When moving to 5 slm and 8 slm, a uniform structure is achieved with distinct equilibrium facet characteristics. For the 8 slm condition, a truncated pyramid results with semi-polar sidewalls and a smooth top-c-plane facet. With a reduced 5 slm flow, the semipolar sidewalls are replaced by nonpolar sidewalls characterized by their 90° angle with respect to the c-plane (substrate). The cylinder shape results due to a reduction in the c-plane growth rate and a uniform growth rate across the full opening area leading to growth in a single dimension (+c). Using the 5 slm N-pulse condition, the pulse number was increased from 50 to 150 in order to test continuity of the shape over a longer duration and to get closer to the simulated 3D BV aspect ratio. Following the growth, SEM confirmed the equilibrium shape remained after the increased pulse number, as is shown in Figure 6-17.
Figure 6-16: SEM of microstructures resulting from the pulsed mode SAG after 50 pulses for varied NH$_3$ flow during the N-pulse. An effective lowering of the overall V/III (unquantifiable) is achieved by lowering the pulsed NH$_3$ flow.

Figure 6-17: SEM of 5 slm NH$_3$ /N-pulse condition after 150 pulses
6.2.2 u-GaN shell overgrowth optimization

6.2.2.1 3D Fin Core-shell

For the overgrowth of the subsequent u-GaN shell on fin seed, both pressure and V/III ratio were varied to test for conformality and uniformity. A thin AlN marker was grown prior to the u-GaN layer to highlight the n-GaN/u-GaN interface during characterization. It is known that higher growth pressure can lead to reduced C incorporation in u-GaN films, and this would be desirable for the i-layer.\(^{15}\) However, it is not known how pressure will affect the growth dynamics over a 3D+planar surface. Prior to u-GaN shell growth optimization, the mask was removed from the samples by HF dip. Growths were tested at V/III of 2200 and 660, and pressure of 300 Torr and 100 Torr. Figure 6-18 shows the cross-sectional SEM for each sample. It becomes clear that the conformality of the growth is improved at a growth pressure of 100 Torr and 2200 V/III. With an increase in the growth pressure and reduction in V/III ratio, the fin between features is not filled/overgrown, and the sidewall growth terminates at the c-plane trench. A green line is drawn over the n-GaN / u-GaN interfaces to aid in the visualization of the growth.
Figure 6-18: Cross sectional SEM of each u-GaN shell growth on n-GaN seed.

Taking a closer look at the 100 Torr and 2200 V/III sample, the u-GaN shell overgrowth is conformal over the 3D feature and into the trench. A semi-polar plane appears that intersects with the non-polar sidewall, with an additional semi-polar plane appearing normal to the seeds top corner (above “c” in Figure 6-19). The thickest u-GaN shell region is found at point “b” at the n-GaN seeds bottom corner. The thickness uniformity over the shell can be improved, with points “a”, “c”, and “d” currently being within ~140 nm of each other. For GaN p-i-n BV devices, the u-GaN layer should be in the range of 500 nm – 700 nm thick, and nonuniformities in the thickness may translate to noticeable nonuniform device performance and beta-capture. The cross-section of the sample grown at 660 V/III and 100 Torr is shown in Figure 6-20. Here the lateral growth rate is inhibited (shown at point “c”), similar to the seed growth which was also grown at low V/III ratio. This condition also leads to nonuniform filling of the trench at point “d”. This condition does
not lead to a conformal junction, similar to the samples grown at 300 Torr. To confirm that the impact of pressure on filling the bottom of the trench (c-plane), the sample of 300 Torr / 2200 V/III was overgrown under 100 Torr growth condition. As shown in Figure 6-21, c-plane overgrowth, and filling of the bottom of the trench is observed.

Figure 6-19: Cross sectional SEM of the u-GaN shell grown at 2200 V/III, 100 Torr

Figure 6-20: Cross sectional SEM of the u-GaN shell grown at 660 V/III, 100 Torr
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6.2.2.2 3D Pillar Core-shell

The optimized pillar seed condition in Figure 6-17 was then used for subsequent overgrowth studies. Prior to u-GaN overgrowth the dielectric SAG mask was removed by HF wet etch to enable growth of conformal combined 3D+planar layers, as described previously. A standard u-GaN planar growth condition was used as the first attempt with a temperature of 1020 °C, pressure of 100 Torr, and 2200 V/III. Following the growth, SEM was performed and is shown in Figure 6-22. Conformality of the u-GaN layer between features is achieved, with distinct facets showing varied growth rate. There are micro-pits surrounding nearly all of the seed structures. These are most likely formed through competition between competing growth planes out from the seeds. Growth fronts meet unevenly which leads to gaps or pitting to the GaN epi-surface. These features may result in increased leakage current for fabricated devices. To confirm the conformality and to achieve a more informative view of the growth fronts, cross-sectional SEM was performed with the sample being cleaved using a diamond scribe. Shown in Figure 6-23 is the

Figure 6-21: The green indicates the n-GaN / 300 Torr interface, and the red indicates the 300 Torr / 100 Torr interface. Filling of the trench occurs post-100 Torr overgrowth.
cross-section of the n-GaN seed with u-GaN overgrowth. The u-GaN layer can be distinguished from the n-GaN seed by a darker contrast border surrounding the n-GaN region. The conformality is confirmed, with adjacent structures being connected by the u-GaN layer. The thinnest portion (lowest growth rate) of the u-GaN appears to be at the top right-angle corner of the n-GaN seed structure at only a few nanometers, better shown in Figure 6-24 (right). In contrast, the thickest portion (highest GR) is at the adjacent corner at the bottom of the n-GaN seed, Figure 6-24 (left). Here a semipolar growth plane forms out from the right-angle to a thickness of approximately 790 nm. This plane is a combination of growth fronts from the corner of the n-GaN seed, the nonpolar sidewall, and the c-plane surface between microstructures. The nonpolar sidewall (orthogonal to substrate epi-plane) is ~120 nm, while the top c-plane facet has a thickness of ~245 nm. The thickness values and the associated growth plane is summarized in Table 26.

Figure 6-22: SEM characterization (45° tilt) following 2200 V/III u-GaN overgrowth on n-GaN seeds. Micro-pits observed, formed through competition between facets
Figure 6-23: Cross-sectional SEM of the 2200 V/III u-GaN overgrowth. A view of adjacent pillar structures and confirmation of conformality (left), a magnified view of a single structure showing relative growth rates between planes (right) and the same image with borders isolating regions of interest (left).

Figure 6-24: Further magnification of single microstructure. High semipolar growth rate from the corner of the n-GaN seed base (left) Low growth rate at edge of seed top corner (right).

Table 26: Summary of u-GaN overgrowth plane thickness and GR

<table>
<thead>
<tr>
<th>Overgrowth location</th>
<th>Plane: Location</th>
<th>Thickness (nm)</th>
<th>GR (nm/s)</th>
<th>θ to [0001]</th>
</tr>
</thead>
<tbody>
<tr>
<td>[0001]: center trench</td>
<td>[0001]</td>
<td>225</td>
<td>0.38</td>
<td>0</td>
</tr>
<tr>
<td>[0001]: top pillar</td>
<td>[0001]</td>
<td>245</td>
<td>0.41</td>
<td>0</td>
</tr>
<tr>
<td>[10-10]: right sidewall</td>
<td>[10-10]</td>
<td>120</td>
<td>0.20</td>
<td>90</td>
</tr>
<tr>
<td>[11-22]: intersects sidewall/top pillar</td>
<td>[11-22]</td>
<td>5</td>
<td>0.0083</td>
<td>61</td>
</tr>
<tr>
<td>[0001]+[11-22]: intersects trench/sidewall</td>
<td>[0001]+[11-22]</td>
<td>790</td>
<td>1.32</td>
<td>34</td>
</tr>
</tbody>
</table>
To study the effect of V/III ratio on the u-GaN overgrowth, a second growth was performed at a reduced V/III of 1650 by lowering the NH\textsubscript{3} flow. All other conditions/timing were kept the same. Following the growth, both tilted and cross-sectional SEM were performed shown in Figure 6-25 and Figure 6-26. It is immediately clear that the micro-pitting surrounding the structure that was present in the previous u-GaN overgrowth has significantly reduced. The overall surface of the film appears smoother and the undulation surrounding the seed is more ordered. The cross-section shows a similar profile as the previous growth with conformality of the u-GaN layer surrounding the n-GaN seeds. However, the top c-plane facet and the semipolar corner facet thickness is increased under this V/III condition, while the nonpolar sidewall thickness remains similar to the higher V/III condition. The top c-plane facet growth rate is nearly doubled under the reduced V/III. All thickness values and the associated planes are compared in Table 27. XRD measurement will allow quantification of the crystalline quality of the u-GaN films, typically with higher growth rate the film quality if reduced. The higher growth rate in the c+-direction could be the major contributor to the improved morphology surrounding the base of the seed since the semipolar facet has some contribution from the c-plane growth front.
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Figure 6-25: SEM characterization (30° tilt) of 1650 V/III u-GaN overgrowth on n-GaN seed. Reduction in pitting observed, as compared to 2200 V/III condition.

Figure 6-26: Cross-sectional SEM of 1650 V/III u-GaN overgrowth, first appearance of void at corner of seed.

Table 27: Comparison of thickness for 2200 and 1650 V/III u-GaN overgrowth on n-GaN seed

<table>
<thead>
<tr>
<th>Overgrowth Plane: location</th>
<th>2200 V/III u-GaN Length (nm)</th>
<th>1650 V/III u-GaN Length (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>[0001]: center trench</td>
<td>225</td>
<td>404</td>
</tr>
<tr>
<td>[0001]: top pillar</td>
<td>245</td>
<td>422</td>
</tr>
<tr>
<td>[10-10]: right sidewall</td>
<td>120</td>
<td>120</td>
</tr>
<tr>
<td>[11-22]: intersects sidewall/top pillar</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>[0001]+[11-22]: intersects trench/sidewall</td>
<td>790</td>
<td>840</td>
</tr>
</tbody>
</table>
Pulsed u-GaN overgrowth was also attempted. Two conditions were tested, at 4950 and 2200 effective V/III ratio. The results are shown below in Figure 6-27. Both conditions led to a roughening of the c-plane surface in between the features and on the mesa top. The roughening appears in the form of grain-like features which increase in size with an increase in effective V/III. It is likely the case that the real V/III at the surface is very low resulting in a growth mode that is vertical/3D dominating, with the lateral growth being limited. This would lead to individually raised grain-features and an apparent roughening of the starting flat surface. However, these growths do provide further evidence that a reduction in V/III leads to a smooth semipolar facet at the base of the seed.

![Figure 6-27: Pulsed u-GaN SAG on n-GaN seed. Two V/III conditions tested, both show extreme roughening of the c-plane in between features and on top of the seed.](image)

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6.2.3 p-GaN shell overgrowth optimization

6.2.3.1 3D Fin Core-shell

Lastly, the p-GaN shell was grown over the 3D structures using the standard planar growth condition. Atomic force microscopy (AFM) was performed pre- and post- growth. The p-GaN surface and u-GaN surface (pre- p-GaN growth) are shown in Figure 6-28, with the image of the probe (on the righthand side) showing the area being scanned. The RMS roughness is comparable to planar p-GaN for both the trench and mesa surface. A planar sample in the same growth shows 8.3 cm²/Vs mobility and 2.8 x 10¹⁷ cm⁻³ carrier density by Hall measurement.

![AFM images showing p-GaN and u-GaN surfaces](image)

*Figure 6-28: AFM of the p-GaN surface (top) and the u-GaN surface prior to p-GaN growth (bottom).*
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It is expected this core-shell structure will show high level of interface impurities at interrupted interfaces, due to the multiple growth interruptions to perform various characterizations. To investigate the impact of these interruptions, continuous/uninterrupted PIN 3D growth was performed, using the optimized conditions. The AFM of the p-GaN shell surface of the continuous growth PIN structure is shown in Figure 6-29. The degree of surface undulation and step-bunching/termination is less than that of Figure 6-28 (the interrupted PIN growth). Four separate arrays of fin structures were formed on the sample, with varying degrees of width and pitch. The sample was cleaved to isolate the four arrays for device fabrication. An SEM cross-sectional view of 10 µm mesa x 5 µm pitch in Array 1 is shown in Figure 6-30.

Figure 6-29: AFM of the p-GaN surface (top) and the u-GaN surface prior to p-GaN growth (bottom).
Figure 6-30: Cross-sectional SEM of a 10 µm mesa x 5 µm pitch in Array 1. The lighter contrast indicates the p-GaN material, with increased efficiency of secondary electron emission.

In Figure 6-30, the p-GaN layer can be seen as the lighter contrast surface layer. Because of the downward band bending near the surface of p-GaN, there is enhanced electron transport towards the surface leading to increased secondary electron emission (i.e. higher contrast in image). With an n-type material, such as the u-GaN and n-GaN layers, the secondary electron emission is less due to upward band bending at the surface which depletes electrons. Therefore, the n-type material appears darker in contrast. The p-GaN layer is ~120 nm in the trench center, and upwards of ~370 nm at the semi-polar sidewall indicating a ~3x growth rate here. The n-GaN regrowth / u-GaN layer appears to be approximately ~170 nm in the trench center and up to ~930 nm at the semi-polar sidewall, a ~5.5x increase. A distinction between the u-GaN and n-GaN layer cannot be made under these SEM beam conditions.

6.2.3.2 3D Pillar Core-shell

For the last step in the formation of the combined 3D+planar PIN pillar microstructure, the p-GaN layer is grown. The standard planar p-GaN condition at 990 °C and 300 Torr is used, just as was grown for the fin structure. Each sample for the u-GaN overgrowth study was placed into
the system for a single run, to ensure continuity for the p-GaN layer. Figure 6-31 shows the SEM for pre- and post- p-GaN overgrowth for each of the samples. The samples are ordered in decreasing V/III ratio from top to bottom, with the first two being continuous u-GaN and the bottom two being pulsed u-GaN. Following p-GaN overgrowth it is shown that the pitting and roughness transfers from the underlying u-GaN layer. For example, the micro-pitting surrounding the seeds for sample s0986l is present after the p-GaN growth. The sample s1274d appears to be the best structure in terms of uniformity, roughness, and shape. All of these samples were used for device fabrication to probe the quality and behavior of the PIN and assess overall relationship between on-wafer characteristics with processed devices.
Figure 6-31: SEM overview of the p-GaN overgrowth on each u-GaN overgrowth / n-GaN seed tested.
6.2.3.3 $p$-GaN overgrowth DOE: effect of pressure and temperature

6.3 Top-down approach: growth on processed nonpolar surface

6.3.1 Effect of KOH wet etchant on a-plane regrowth surface morphology

MOCVD overgrowth was performed on a-plane u-GaN samples that were subjected to various dry/wet etches to compare the effects on interface/overgrowth quality. This experiment mimics the condition of 3D microstructure sidewalls (non-polar) after top-down etching and enables direct characterization of the surface and bulk characteristics. Top-down etching is another means for seed formation, but the introduction of dry-etching induced defects leads to increased leakage in the device. The experimental set (A1-A4) is listed in Table 28. Each sample is from the same parent wafer. The processing steps for the sample set allow for a comparison of the film/interface quality; an as-grown wafer, TMAH treated (without ICP etch), ICP etched and TMAH treated, and ICP etched (without TMAH treatment). Each wafer was cleaned with standard organic clean prior to growth. The standard TMAH recipe was 80 °C for 10 minutes. The standard ICP etch was a blanket etch to approximately 20 nm depth at 500 W ICP power, 100 W RIE power, in a BCl$_3$Cl$_2$ chemistry environment. The overgrowth was a repeat of the standard a-plane template layer, grown at 100 Torr growth pressure to a target thickness of 80 nm. This target thickness was chosen to allow absorption of 325 nm laser line down to the interface for PL characterization. Atomic force microscopy (AFM) was performed at each processing stage to follow the evolution of the surface morphology, as shown in Figure 6-32.

<table>
<thead>
<tr>
<th>#</th>
<th>Sample ID</th>
<th>Type</th>
<th>Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>s0784a</td>
<td>A-plane planar</td>
<td>As-grown</td>
</tr>
<tr>
<td>A2</td>
<td>s0784c</td>
<td>A-plane planar</td>
<td>TMAH treated</td>
</tr>
<tr>
<td>A3</td>
<td>s0784b</td>
<td>A-plane planar</td>
<td>ICP Etched, TMAH treated</td>
</tr>
</tbody>
</table>

Table 28: Processing conditions for planar a-plane GaN sample set
Figure 6-32: AFM images of the a-plane GaN surface after each processing step. Post-overgrowth the samples show similar RMS roughness ranging from 2.5 – 4.5 nm.

Figure 6-33: Optical micrograph of the a-plane GaN surface after overgrowth. Macro-level roughness is seen in the sample that was ICP etched with no wet treatment (A4).
An interesting result post-overgrowth is that the as-ICP etched sample (which shows presence of roughened surface with particulates) led to the smoothest overgrowth surface as measure by AFM root mean squared (RMS) roughness. On the macroscale however, it appears this sample shows the macro- roughness on the surface by optical microscopy shown in Figure 6-33. Samples A2 and A3 show a micro-faceting on the surface after the TMAH treatment, which results from the semi-polar facets (that are present due to surface roughness) etching at a higher rate than the non-polar growth facet. Figure 6-32 also shows that post-overgrowth the micro-faceting disappears and the surface for Sample A2 and A3 return to a state similar to the as-grown case.

6.4 References


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Chapter 7 Combined 3D + Planar Device

Fabrication and Evaluation

7.1 Design Considerations for 3D+planar BV Device

As a parallel to the work presented here for use of 3D III-Nitride structures as an enhanced platform for BVs, development of 3D structures for μ-LEDs has increased substantially in recent years.¹⁻⁷ The benefit of 3D μ-LEDs over conventional planar LEDs is the same for BVs which includes the significant increase in surface area for a given substrate footprint and lower average defect density. For 3D μ-LEDs, the significant increase in surface area translates to an increase in active area for light emission, the equivalent case for BVs where an increase in active area is desired for enhanced absorption of particles. In traditional planar LEDs with [0001] polar orientation, there exists an offset of the electron/hole wavefunctions due to internal polarization fields, known as the quantum-confined stark effect (QCSE), reducing carrier confinement. The presence of nonpolar sidewalls on the 3D structures removes the polarization-induced electric fields that can negatively impact quantum efficiency. As a result of these similarities, the methodology for device fabrication of μ-LEDs is translated here for BVs, where certain limitations arise that require a modification from the standard process in literature.

Most commonly, the III-Nitride structures used for μ-LEDs consist of an n-GaN seed (grown by SAG or dry-etched from n-GaN template), followed by InGaN/GaN multi-quantum wells (MQWs) for the active layers, then a p-GaN contact layer. This p-GaN layer is either conformal to connect adjacent structures, or isolated to single features, as differentiated in Figure 7-1. In most
cases, an indium-tin-oxide (ITO) layer is deposited as a transparent contact for the p-GaN surface layer in addition to a Cr/Au probing contact, while some reports attempt contacting of the 3D p-GaN with a standard metal stack (Ni/Au) atop certain regions of the p-GaN surface, also shown in Figure 7-1. The key purpose of the ITO is to conformally connect all active microstructures to ensure even current spreading, with its thickness on the order of several 100s of nm. The ITO material shows high transparency to the visible regime, which is necessary to maintain high extraction efficiency. Without this conformal current spreading layer, such as the example shown in Figure 7-1 (right), the spreading is reliant solely on the resistivity of the p-GaN layer. The p-GaN layer, even when grown 2-D [0001] exhibits high resistivity, therefore this contacting method is not promising to obtain large area devices. It is shown that the current injection and resulting EL emission is isolated to the area directly underneath the metal contact, which in turn limits the intended device area.

![Figure 7-1: Example of device fabrication processes for μ-LED with (left and right) conformal p-GaN and (middle) isolated p-GaN capping layer. Contact method is (left and middle) conformal ITO and (right) metal contact. Schematics from 1–3.](image)

Even though ITO has been studied extensively for use as a current spreading layer to p-GaN, the issue with implementing it in BVs is the undesirable kinetic energy loss that will occur within the thickness of the layer (100-200 nm) for incident β-particles. The instance where this thick ITO may be beneficial is for radioisotopes that emit β-particles with high energies (ex. $^{147}$Pm)
where the kinetic energy would be minimally attenuated. For lower energy sources, such as $^3$H and $^{63}$Ni the ITO standard thickness is unacceptable. This is also the reason that filling of the gaps between features by p-GaN overgrowth (as shown in the structure in Figure 7-1, right) is not ideal for this application, and hence why the proposed structure here is a controlled and conformal p-GaN layer that wraps around individual structures. An option to proceed is to attempt a thin ITO layer for current spreading, through deposition method such as thermal atomic layer deposition (ALD). However, given the success of the thin Ni/Au metal shown here for the GaN BV planar devices in Chapter 4, conformal deposition of this layer for 3D devices has been attempted. An additional benefit of the combined 3D+planar configuration here is the conformal p-GaN layer that connects multiple structures. If there is an unintentional break in the contact due to an issue with conformal deposition of the metal, the current may still spread through the p-GaN layer.

Another consideration for conformal contacts is the known anisotropy in conduction for various planes of the GaN crystal along with in-plane anisotropy, which has been shown to be defect dependent.\textsuperscript{10–13} As an example, it has been shown by transmission line method (TLM) that the sheet resistance of a-plane GaN along the c-axis is double that of the sheet resistance along the m-axis.\textsuperscript{11}

### 7.2 Fabrication Process Flow

Combined 3D+planar GaN core-shell structures were grown by MOCVD as reported in Chapter 6, both in a fin and pillar geometry with varied dimension. The device process flow closely matches that used for planar devices in Section 4.1.2 after consideration of the design factors detailed above. To note, the epitaxy for the fin structures studied here (g4002/s1273c) was performed in a single growth run, without any interruption between the P-I- layers and an N-
interface burying layer. The epitaxy for the pillar structures studied here, however, had an interruption between the p-GaN layer given the need for characterization between growth processes. The schematic of the fabrication process flow is shown in Figure 7-2. Standard photolithographic process with 1813 resist is used for mesa isolation. Due to the growth mask layout (continuous array of mask window/openings across full wafer), to isolate individual devices the ICP etch is performed on a 3D surface which leaves behind a 3D surface as shown schematically in Figure 7-2 and the corresponding SEM images in Figure 7-3. The standard N-type ohmic contact alloy of Ti(25nm)/Al(220nm)/Ni(60nm)/Au(50nm) was deposited by electron beam evaporation and annealed at 800 °C for 30 seconds in an N2 inert environment by RTA. The p-type contact of Ni(4nm)/Au(4nm) and Ni(50nm)/Au(50nm) for the thin current spreading and thick contact layers, respectively, was deposited by electron beam evaporation in separate runs. The p-contact was annealed at 450 °C for 5 min in an N2/O2 ambient using an RTA. For all steps requiring lithography, a solvent clean consisting of acetone, IPA, and DI was used to clean samples, and the resist was removed by heated Microposit Remover 1165 at 65 °C. Extended O2 plasma exposure was applied to ensure complete removal of resist given the complex 3D surface which tended to show more residue post-wet chemical etch compared to planar structures.
Figure 7-2: Schematic of Combined 3D+planar device process flow for both pillar and fin geometries

Figure 7-3: SEM image (30 degree tilted) showing the mesa isolation etch step for both (left) fin and (right) pillar structures following resist mask removal, wet cleaning, and O₂ plasma.

Figure 7-4: SEM images of device edge to show the distinction between contacts and processing steps for (middle) fin and (right) pillar structures
7.3 Electrical Characterization

7.3.1 Generation 1 Fin Combined 3D+planar PIN

The I-V characteristics of the fin pin core-shell devices (s1273c) were measured using a Keithley B1500 parametric analyzer within a voltage range of -5 to 5 V. Figure 7-6 shows the characteristics for selected devices. The variation in forward current amongst the devices somewhat correlates with the location on the wafer perpendicular to the length of the fin, with Device 3 and 4 showing similar forward conduction as well as Device 15 and 11 showing a similar trend. A schematic showing the relative location of the devices is depicted in Figure 7-6(right). Overall, compared with planar diodes of similar area the leakage current at -5 V is high for the fin devices. The fin devices overall also show a significant subthreshold turn-on voltage near ~1 V that may indicate a parallel Schottky conduction path in addition to the PIN junction with $V_{th} \sim 3$ V. The p-contact was annealed with the intent to form ohmic contacts but given the unique geometry of the devices it is possible that the contact is nonohmic. Standard transmission line measurements (TLM) to determine contact resistivity and profile were unsuccessful on the combined 3D sample. A comparison between a planar PIN diode and device 6 (combined 3D PIN) is shown in Figure 7-7, where the difference in forward conduction is observed as well as
significant difference in leakage current. Sources of increased leakage current include interface
defects or impurities at the seed structure / PIN interface in the 3D+planar structure, as it was
necessary to remove the dielectric mask with chemical etchant prior to regrowth of the PIN layers,
which involved transport and extended handling of the sample. A more aggressive cleaning/etch
(such as TMAH / KOH) prior to regrowth may assist but will alter the starting morphology of the
seed (which may not have too large an effect on the subsequent epitaxy, as indicated from the a-
plane regrowth study in Section 6.3.1). This will need to be taken into account in future studies.

Figure 7-6: (left) Room temperature IV of several devices from fin PIN core-shell device, varied
performance dependent on location on the wafer. (right) schematic depicting location of the
devices relative to each other

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Device 6 and 11 were characterized further with temperature-dependent IV (IVT) utilizing the same system with a heated sample chuck. The temperature was increased from room temperature (RT) to 478 K, with measurements being taken in 20 K increments. The IVT relation for device 6 is shown in Figure 7-8 and device 11 shown in Figure 7-9. For the reverse current, taking $J_0$ at -5 V and plotting against 1/T, an exponential relation results as expected shown in Figure 7-10(left). A stronger coefficient of determination ($R^2$) is shown for device 11 compared to device 6. Referring to the inset plot in Figure 7-8(right), the temperature dependence on the forward current (at >3 V) is inconsistent for device 6, however a strong correlation results for device 11 with a decrease in forward current (at >3 V) as the temperature is increased, shown in the inset in Figure 7-9(right). This is thought to be a result of the decrease in carrier mobility with increased temperature which leads to increased resistance in the depletion region.\textsuperscript{14} It is unclear why device 6 does not show this same relation, however that may be due in part to area-dependent characteristics variation across the sample (e.g. see Figure 7-6). Confirmation of PIN junction behavior is confirmed given the $V_{th}$ of ~3.0 V for device 6 as plotted in Figure 7-10(right). In the
same plot, the $V_{th}$ for device 11 is consistently lower at a value $\sim$2.6 V. If assumed that device 11 is a Schottky diode because of this low threshold voltage, then the reverse saturation current should follow the temperature dependence relation of $J_0 = A^*T^2 \exp(-q\phi_b/kT)$ where $\phi_b$ is the barrier height and $A^*$ is the effective Richardson constant. This means that a plot of $\ln(J_0/T^2)$ vs $1/T$ should show a linear relation. As shown in Figure 7-11, the data does not show a strong linear relation for either device 11 or device 6.

**Figure 7-8:** Temperature dependent IV (IVT) of device 6, (left) reverse current and (right) forward current.

**Figure 7-9:** Temperature dependent IV (IVT) of device 11, (left) reverse current and (right) forward current.
7.3.2 Generation 1 Pillar Combined 3D+planar PIN

The I-V characteristics of the pillar pin core-shell devices (s1273c) were measured using the same Keithley B1500 parametric analyzer within a voltage range of -5 to 5 V. Figure 7-12 shows the resulting IV characteristics, which indicate Schottky diode behavior given the low turn on voltage of ~1.5 V. Differing from the fin pin core-shell devices in Section 7.3.1, these pillar devices do not appear to have a parallel conduction pathway from a PIN depletion region. Given that these diodes had growth interruption between the PIN layers it is possible that there are a high density...
of interfacial impurities resulting in the high leakage current and possible compensation of the thin p-GaN layer. The leakage current may also be due to the lack of a post-mesa isolation KOH passivation etch. This was not used here given the 360-degree sidewall exposure, where the etchant could have removed a significant portion of the sidewall p-GaN layer. To counteract this high leakage pathway, a 300 nm SiN passivation dielectric was deposited. The mesa contact was reopened by standard lithographic techniques. The change to the IV characteristics is shown in Figure 7-13. Note the leakage improved by more than 2-orders of magnitude but the forward conduction characteristics still indicates a Schottky transport pathway. Source of this behavior include the possibility that the p-GaN layer is highly resistive and/or layer density of compensating impurities exist at growth interrupted interfaces (although the SEM contrast difference in Figure 7-3 may suggest the contrary). It is expected that an improvement will be observed if the p-GaN layer is grown continuously with the I-N layers, and not following a growth interruption. Also, it is clearly shown that removal of the standard passivation wet etch following mesa-isolation is detrimental to the device, especially with 3D devices which have large surface area. Further studies should include optimization of the conformal p-GaN overgrowth on pillar seed.
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Figure 7-12: Pillar 3D+planar “PIN” diodes showing high leakage and Schottky-like behavior

Figure 7-13: Pre- and Post- 300 nm SiN passivation deposition, non-standard process to improve the leakage current given the desire not to chemically etch the pillar sidewalls.

7.4 Challenges to Address

A challenge that requires addressing in future iterations is overcoming the possibility of forming a nonconformal contact on the mesa caused by the unidirectional electron beam deposition process. The metal evaporation system used here does not have capability for two-axis planetary
rotation which would ensure conformal metal deposition on high-aspect ratio structures. Referring to Figure 7-14, there may be a gap in the metal contact on one of the sidewalls for both the fin and pillar geometry, resulting from the sample tilt relative to the source during the deposition. If there is a disconnect the current spread and connection between structures will rely partially on transport through the p-GaN alone, which could limit device performance and skew the electrical characteristics measured given the high contact resistivity. For the pillar geometry, if one sidewall has conformal deposition of metal the structure should have a continuous metal contact due to the planar gaps surrounding each. However, for the fin structures if the gap in the contact travels along the full length of the fin there may be a disconnect between adjacent structures.

![Image](image)

*Figure 7-14: Magnified view of fin structures with p-contact, possible gap in the contact along a structure sidewall as indicated within the highlighted red region.*

To test this theory, a high forward injection current (~100 mA at 8 V set-point) was applied to a dummy device on the Generation 1 fin core-shell PIN sample. The EL emission that results shows varied intensity based on probe location. This is depicted in Figure 7-15, where two probe positions (labeled) show a high EL intensity along the fins that the probe is in direct contact with, and lowered intensity laterally across adjacent (presumed to be connected) fins. The true color of
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the EL emission is bluer than the violet color in the image. If the bulk of the conduction for the 3D pin is happening along the probed fins, and minimal current traverses laterally, then that true device area may be much smaller than the isolated mesa. Small devices with limited depletion volume are not ideal for BV and this nonuniform conduction needs to be addressed in future iterations.

Figure 7-15: EL intensity differences based on probe location, indicates strong conduction along fins/contact and decreased lateral conduction across fin features.

If one assumes that the contact is conformal and uniform, then this phenomenon would be the result of nonuniform epitaxial layer. To investigate this, extensive SEM was performed on the device surfaces to locate any possible points of concern. The result is that most of the devices on the sample show uniform contrast indicating a continuous and uniform epitaxial layer. However, in some regions, as shown in Figure 7-16, there appears to be a discontinuity in the epitaxy that
could be affecting the subsequent current conduction. This area was closer to the sample edge, although such features were not isolated to the edge of the sample. Although low, the presence of such features was more probable for larger devices. It is recommended that future growth optimizations focus on the macro-scale uniformity of 3D+planar overgrown layers, to determine whether such features are a result of growth parameters (such as pressure, V/III, temperature) or a result of sample preparation pre-growth.

![Image of possible discontinuities in p-i-GaN epitaxy](image)

*Figure 7-16: Possible discontinuities in p-i-GaN epitaxy that leads to nonuniform conduction across mesa of certain devices*

### 7.5 References


Chapter 7


Chapter 8 Conclusion and Future Directions

8.1 Conclusion

Al\textsubscript{1-x}Ga\textsubscript{x}N is promising for use as the converter material in a BV device due to its wide
bandgap, superior radiation tolerance, chemical inertness, and physical hardness. With the
emergence of micro- and nano- technology, many low power systems require self-sustained power
sources where replacement is difficult or impossible in harsh environments, for which BV batteries
are a prime candidate. BV batteries have been investigated for several decades; however, the
technology still lacks in efficient energy conversion and limited power output. This work has
addressed some of the challenges associated with, and improved upon, the GaN-based beta-energy
converter, along with engineering new methods for device qualification under high energy
irradiation and radioisotope sources. Uniquely, a converter structure has been designed, simulated,
and implemented with the growth of novel GaN 3D+planar core-shell microstructures. A summary
of the key results of the thesis is provided below.

A combined 3D (planar+core-shell) PIN device has been proposed as the optimal design for
GaN application as a BV battery. This optimized structure layout leads to enhanced conversion
(depletion) region volume compared to that in a planar device under equilibrium conditions.
MCNPX code was used to simulate a full spectrum isotopically emitting beta source to couple
with the 3D combined device structure. A parametric study of mesa width, mesa height, and
isotope gap spacing was performed with comparison made to a planar structure, in contact with
\textsuperscript{63}NiCl\textsubscript{2} liquid radioisotope material. This work demonstrated that both the power generated and
efficiency of energy transfer decline with an increase in mesa width. When increasing mesa height from 4 μm to 8 μm there will be a trade-off where power generated will increase at the cost of efficiency and isotope activity density. There is a 3.75x increase in $P_{\text{GaN/cm}^2}$ at approximately half the activity density for the 4 μm mesa height structure compared to planar designs optimum at 10 μm $^{63}\text{Ni}$ thickness with 5.8x improvement in $\eta_{\text{src}}$. The 8 μm mesa height structure shows a 5.6x increase in power generated at only 1.1x the activity.

High performing GaN PIN planar diodes were fabricated by tailoring the fabrication process for BV specific needs such as a “beta-transparent” p-contact, low forward leakage by a KOH wet-etch passivation treatment, and implementing a large area mesa for greater electron absorption. Electron energy irradiation was performed to mimic radioisotope exposure using an electron flood gun (4-16 keV) and a custom in-operando TEM setup (62 – 200 keV). Beam voltage and beam current dependence is reported for several devices. For the electron flood gun irradiation, the highest reported efficiency of energy conversion at 7% is reported for GaN PIN exposed to 16 keV electron irradiation. For in-operando irradiation in TEM, a large-area planar GaN PIN (0.04 cm$^2$, 17.8 nA/cm$^2$ at -5 V) shows a decrease in maximum power produced (MPP) from 2.45 μW/cm$^2$ to 0.45 μW/cm$^2$ at an approximate input current density of 5 nA/cm$^2$ for increasing beam voltages from 62 kV to 200 kV. In addition, the MPP shows enhancement with increased beam current density up to 48.2 μW/cm$^2$ at 177 nA/cm$^2$ for 62 kV voltage. A 60-minute duration irradiation was performed to target a total electron dose of $10^{16}$ cm$^{-2}$ which led to no observable change in the dark current characteristics of the device at any dose up to the target. This indicates no observable radiation induced degradation of the device at this dosing level. This unique characterization
Chapter 8

capability allows for non-destructive evaluation of new device designs and structures for the next generation of BV devices coupled with high energy sources.

Direct measurement of these GaN PIN diodes under exposure to solid metal radioisotope and liquid radioisotope solution was performed at ARL and ORNL, respectively, for both $^{63}$Ni and $^{147}$Pm isotopes. Methodology for device testing in custom enclosures was developed and executed. For the metal radioisotopes, the overall magnitude of the peak in MPP is relatively low, at 0.74 nW/cm$^2$ for exposure to $^{63}$Ni and 2.27 nW/cm$^2$ for exposure to $^{147}$Pm, corresponding to an efficiency of 0.1% and 0.02%, respectively. In addition to these studies, heteroepitaxial and homoepitaxial GaN PIN devices were subjected to focused 4.5 MeV $\alpha$-beam irradiation at similar input current. The GaN/GaN PIN shows a permanent degradation in forward current because of the irradiation, while the GaN/sapphire PIN restores the pre-irradiation forward current once the irradiation ceases. The GaN/sapphire PIN shows a discoloration within the irradiated area, which upon PL measurement, correlates to a disappearance of the blue luminescence (BL) emission peak at ~2.88 eV. The BL is related to the Mg-dopant atom in GaN which may suggest displacement of the Mg dopant from the acceptor position and/or presence of increased compensating N-vacancy density from the alpha bombardment. The GaN/GaN PIN also shows disappearance of the BL following irradiation, and also a large reduction in the YL emission. It is possible that the restoration in forward current for the GaN/sapphire PIN is a result of conduction through the regions of the device mesa that were not directly irradiated with the beam. Given the large differences in pre-irradiation dark I-V characteristics between the two devices, it is difficult to compare the differences in post-irradiation I-V in direct association to the specific substrate.
MOCVD growth of the proposed combined 3D+planar GaN PIN was achieved in both a fin and pillar geometry. High aspect ratio n-GaN seeds were grown by optimization of the precursor V/III ratio during MOCVD growth. Crystalline facet stabilization was controlled by varying the V/III ratio. A shift to a pulsed growth mode was made to allow for a low effective V/III ratio by switching the flow of each precursor in the reactor at any given time. The TMGa flow during the Ga-pulse (5s) was kept the same for each, with a change in the NH₃ flow during the N-pulse (10s) of 1.6 slm, 5 slm, and 8 slm. The 1.6 slm N-pulse condition led to a V/III below the threshold for uniform steady-state growth and resulted in a randomly oriented – defective structure. When moving to 5 slm and 8 slm, a uniform structure is achieved with distinct equilibrium facet characteristics. For the 8 slm condition, a truncated pyramid results with semi-polar sidewalls and a smooth top-c-plane facet. With a reduced 5 slm flow, the semipolar sidewalls are replaced by nonpolar sidewalls characterized by their 90° angle with respect to the c-plane (substrate), which was selected as the seed condition for the combined 3D+planar core-shell structure. Continuing to the u-GaN (i-layer) overgrowth, growth pressure is shown to play a pivotal role in the conformality and filling of the trenches between adjacent seeds, where 300 Torr results in inconsistent filling. Standard p-GaN growth conditions were then applied to the structures to complete the junction on both fin and pillar geometries. The fin combined 3D+planar PIN showed diode behavior with a threshold voltage of ~3 V, nearing the bandgap of GaN.
8.2 Future Directions

8.2.1 Improved control of facet stabilization and growth rate for 3D PIN

Based on the MOCVD SAG results in Chapter 6, the resulting microstructure overgrowths consist of additional semipolar facets. The formation of these facets in between adjacent structures results in thickness variation of the u-GaN and p-GaN layers. This thickness variation can lead to nonuniformities in the device characteristics which may inhibit achieving optimum performance. It is also shown that the coalescence of these planes is not always uniform along the length of the structures, resulting in the formation of defects in the overgrowth layers that are observed on the surface. It is advisable that one of two paths be taken 1) bring the current knowledge of combined 3D+planar morphology from experiments into the simulation design to gain a deeper understanding of the impact of these additional planes on performance characteristics, or 2) continue the growth optimization to inhibit the formation of semipolar planes to maintain the initial structure design. The latter may be required in order to minimize the effect of polarization field at interfaces for AlGaN-based BV. A simulation of a combined 3D+planar structure with semipolar planes and non-uniform thickness for the u-GaN and p-GaN layers leads to increased “dead-zone”, as shown in Figure 8-1.
Related to the control of facet stabilization, preliminary investigation into the growth of isolated 3D core-shell PIN structures has been performed (i.e. where the dielectric mask material remains in place throughout the growth of PIN layers). This structure varies from the combined 3D+planar in that there is no “planar” junction between adjacent structures, synonymous with how 3D LED structures are grown. Given that the semi-polar facet was shown to form due to the
competing growth of the trench and sidewall planes in combined 3D+planar growth, keeping the mask would remove that intersection between the two planes. However, the area between structures is now “wasted”, with no depletion region formation. Regardless, under growth conditions similar to attempts of combined 3D+planar, the results below indicate that presence of the mask for 3D core-shell inhibits the formation of semipolar facets. Figure 8-2 shows an example of this, with a u-GaN overgrowth completed on a u-GaN seed. An AlN marker layer is visible in the image on the left, with a red dotted line placed over this layer in the right image, to show the thicknesses. The growth rate of the c-plane is an order-of-magnitude greater than the m-plane, which requires addressing. Difficulties in contacting a large array of such isolated structures while limiting the attenuation of incoming β-particles in the contact material will be a critical factor to consider.

Figure 8-2: 3D SAG with mask present, no semi-polar facet forms during u-GaN overgrowth, the aspect ratio is maintained. Growth rate differences between c-plane and sidewall are order-of-magnitude.
Additional growth condition optimization for both the u-GaN and p-GaN layer is required, to attend to the above and to ensure that the desired dopant levels are obtained. Characterization by atomic probe microscopy (APT) and micro-cathodoluminescence (µCL) will allow a more thorough and quantitative means for determining the electrical and optical characteristics of these unique 3D+planar layers. The eventual goal is to test 3D device structures under radioactive source for direct comparison to devices in a planar layout.

8.2.2 Homoepitaxial GaN Planar and 3D+planar PIN

An attempt at utilizing a homoepitaxial GaN/GaN PIN planar device was made, as described in Section 4.1.3.2. For currently unknown reasons the performance of the device was poor, with high leakage current. This may be a result of the MOCVD growth or fabrication process. Section 1.4.3 provides some details on the benefit of homoepitaxial growth of PIN, mainly regarding the significant reduction in dislocation density which in turn should improve leakage current and enhance the carrier diffusion lengths. As the availability of bulk GaN substrates increases and cost reduces (driven in part from the increasing promise of bulk GaN – based power electronic devices), its use in BV should be explored further.
Appendix A: Python Code

In situ TEM Irradiation: Keithley 6430 I-V Sweep

# Keithley IV Sweep for 6430 Source Meter
# K. Hogan 2019
# Command line input: python IV_sweep.py min_voltage max_voltage voltage_step filename.txt deviceid_plottitle
# Example: python IV_sweep.py -10 10 0.5 test "Test IV"

import sys
import visa
import matplotlib.pyplot as plt
import numpy as np
from scipy import stats

# Variable intake and assignment
startv = sys.argv[1]
stopv = sys.argv[2]
stepv = sys.argv[3]
filename = sys.argv[4]
deviceID = sys.argv[5]
startvprime = float(startv)
stopvprime = float(stopv)
stepvprime = float(stepv)
steps = (stopvprime - startvprime) / stepvprime

# Assign keithley to resource ASRL6::INSTR
rm = visa.ResourceManager()
rm.list_resources()
Keithley = rm.open_resource('ASRL6::INSTR')
Keithley.write("*RST")
#Keithley.baud_rate = 57600
Keithley.timeout = 200000 # Needs to be long enough to complete sweeping
# Turn off concurrent functions and set sensor to current with fixed voltage
Keithley.write(":SENS:FUNC:CONC OFF")
Keithley.write(":SOUR:FUNC VOLT")
Keithley.write(":SENS:FUNC 'CURR'")
Keithley.write(":SENS:RES:NPLC 1.0")

# Set measurement speed to NPLC 1.0
Keithley.write(":SENS:FUNC:CONC OFF")
Keithley.write(":SOUR:FUNC VOLT")
Keithley.write(":SENS:FUNC 'CURR'")
Keithley.write(":SENS:RES:NPLC 1.0")

# Voltage starting, ending, and spacing values based on input
Keithley.write(":SOUR:VOLT:STAR " + str(startv))
Keithley.write(":SOUR:VOLT:STOP " + str(stopv))
Keithley.write(":SOUR:VOLT:STEP " + str(stepv))
Keithley.write(":SOUR:SWE:RANG AUTO")

# Set compliance current (in A), sweep direction, and data acquisition
Keithley.write(":SENS:CURRE:PROT 105E-3") # limit is -105mA to 105mA for 6430 model
Keithley.write(":SOUR:SWE:SPAC LIN")
Keithley.write(":SOUR:SWE:POIN " + str(steps))
Keithley.write(":SOUR:SWE:DIR UP")
Keithley.write(":TRIG:COUN " + str(steps))
Keithley.write(":FORM:ELEM CURR")

# Set sweep mode and turn output on
Keithley.write(":SOUR:VOLT:MODE SWE")
Keithley.write(":OUTP ON")

result = Keithley.query(":READ?")
yvalues = Keithley.query_ascii_values(":FETC?")
Keithley.write(":OUTP OFF")
Keithley.write(":SOUR:VOLT 0")

# Create xvalues array and calculate conductance
xvalues = np.arange(startvprime,stopvprime,stepvprime)
slope, intercept, r_value, p_value, std_error = stats.linregress(xvalues, yvalues)
print("Resistance:" , 1/slope, "Ohms")
# Plot IV and save data to txt file
plt.plot(xvalues,yvalues)
plt.xlabel('Voltage (V)')
plt.ylabel('Current (A)')
plt.title(str(deviceID))
plt.show()
np.savetxt(filename + "_IV.txt", (xvalues,yvalues))

# Calculate Maximum Power Point in IV curve
# Save power data to file
power = np.array(xvalues * yvalues)
maxpower = np.amax(power)
maxpower_loc = np.argmax(power)
np.savetxt(filename + "_Power.txt", (xvalues,power))

# Print out max power value, location in np array
# then print x,y values cooresponding to np location
#print(maxpower)
#print(maxpower_loc)
#print(xvalues[maxpower_loc], yvalues[maxpower_loc])
#print(power)
#print(xvalues)

# Plot the power vs voltage in the IV quadrant of the IV curve
# and save the data into a file.
plt.xlim([0, np.amax(xvalues)])
plt.ylim([np.amin(power), 0])
plt.plot(xvalues,power)
plt.xlabel('Voltage (V)')
plt.ylabel('Power (W)')
plt.title('Maximum Power')
plt.show()
In situ TEM Irradiation: Keithley 6430 Drive Current

# Set Current for 6430 Source Meter
# K. Hogan 2019
# Command line input: python curr_set.py current_setpoint(in amps) device_Size(in microns)
# Example: python curr_set.py 0.01 400

import sys
import visa
import matplotlib.pyplot as plt
import numpy as np
from scipy import stats

curr_set = sys.argv[1]
dev_size = sys.argv[2]
curr_set_prime = float(curr_set)

# Assign keithley to resource ASRL6::INSTR
rm = visa.ResourceManager()
Keithley = rm.open_resource('ASRL6::INSTR')
Keithley.write('*RST')
Keithley.timeout = 100000

# Turn off concurrent functions and set sensor to voltage with fixed current
# Set measurement speed to NPLC 1.0
Keithley.write(':SENS:FUNC:CONC OFF')
Keithley.write(':SOUR:FUNC CURR')
Keithley.write(':SENS:FUNC ' 'VOLT'')
Keithley.write(':SENS:RES:NPLC 1.0')
Keithley.write(':SOUR:CURR ' + str(curr_set_prime))
Keithley.write(':OUTP ON')

enter_maker = input('Press enter to turn off current source ')  
Keithley.write(':OUTP OFF')
Keithley 6430 Plot: multi-file

import matplotlib.pyplot as plt
import sys
import os
import numpy as np
import glob

# Command line input of file name, open file for reading
#file1 = sys.argv[1]

# Call all files in directory with specific notation
# Extract data from each to plot all together
filenames = glob.glob('*.*')
for r in filenames:
    f = open(os.path.expanduser(r))

# Create x,y arrays
x, y = [], []

# Read lines of txt file (which is 2 rows x 400 columns)
    line1 = f.readlines()

# For each row in line1, appended values to x
    for line in line1:
        x.append(line.split())

# Change string value to float value in new numpy array
xyfloat = np.asarray(x, dtype=np.float64, order='C')

# Separate out the x and y values from the numpy array
# Create absolute value array yabs, for log plot
x = xyfloat[0]
y = xyfloat[1]
yabs = np.absolute(y)
plt.plot(x,yabs)
f.close()
print(y[50])
ind = np.argmax(y>0)
print(x[ind])

plt.yscale("log")
plt.xlim([-4, 4])
# plt.ylim([1E-10, 1E-7])
plt.xlabel(' Voltage (V)')
plt.ylabel(' Current (A)')
plt.title('s1507b: Device S3, Pad 11\n200 kV, time dependent')
plt.legend(filenames)
plt.show()
data = list(reader) 
data = np.array(data).astype(float)

if plottype == 'linear':
    plt.plot(data[:,0],data[:,1])
else:
    plt.plot(data[:,0],np.abs(data[:,1]))
csvfile.close()

plt.yscale(plottype) 
plt.grid(True, which="both")
plt.xlabel(' Voltage (V)')
plt.ylabel(' Current (A)')
plt.xlim([-5,5])
plt.title(plottitle)
plt.legend(filenames, prop={'size': 6})
plt.show()

**Diode I-V analysis: multi-file**

# K. Hogan 2020
import matplotlib.pyplot as plt
import sys
import os
import numpy as np
import glob
import csv
import pandas as pd
from scipy.stats import linregress

# Command line input of voltage range
shuntmin = float(input("Min voltage for shunt calc: "))
shuntmax = float(input("Max voltage for shunt calc: "))
seriesmin = float(input("Min voltage for series calc: "))
seriesmax = float(input("Max voltage for series calc: "))
devicearea = float(input("Device area: "))
leakcalc = float(input("Voltage to calculate leakage density: "))
plottitle = input('Plot title: ')
plottype = input("log or linear: ")

# Call all files in directory with specific notation
# Extract data from each
filenames = glob.glob("*.csv")
for r in filenames:
    with open(r, 'r') as csvfile:
        reader = csv.reader(csvfile)
        headers = next(reader)
        data = list(reader)
        data = np.array(data).astype(float)

        if plottype == 'linear':
            plt.plot(data[:,0],data[:,1])
        else:
            plt.plot(data[:,0],np.absolute(data[:,1]))

shunt = data[(data[:,0] > shuntmin) & (data[:,0] < shuntmax)]
shunt_info = linregress(shunt)
Rshunt = (1/(shunt_info.slope)) * devicearea

series = data[(data[:,0] > seriesmin) & (data[:,0] < seriesmax)]
series_info = linregress(series)
Rseries = (1/(series_info.slope)) * devicearea
yintercept = -series_info.intercept / series_info.slope

leakage = data[data[:,0] == leakcalc]
leakageden = leakage*1000 / devicearea

print("\n")
print(r)
print("Vth (V) = ", "%2f" % yintercept)
print("Jleak (mA/cm^2) = ", "%.2E" % leakageden[:,1])
print("Ileak (A) = ", "%.2E" % leakage[:,1])
print("Rshunt (ohm cm^2) = ", '%.2E' % Rshunt)
print("Rseries (ohm cm^2) = ", "%.2f" % Rseries)
csvfile.close()

plt.yscale(plottype)
plt.grid(True, which="both")
plt.xlabel(' Voltage (V)')
plt.ylabel(' Current (A)')
plt.xlim([seriesmin,seriesmax])
plt.title(plottitle)
plt.legend(filenames, prop={'size': 6})
plt.show()
Appendix B: Sentaurus Code

Core-shell 3D+Planar PIN: Structure Script .scm

(load "utility.scm")

; name
(define name "bv")

; x mole fractions
(define x_algan 0.2)
(define x_graded (cons x_algan 1.00))
(define x_rgraded (cons 0.00 x_algan))

; doping
(define i_doping 1E16)
(define p_doping -5E17)
(define n_doping 1E18)

; device dimensions
(define p_height 0.100)
(define i_height 0.500)
(define n_height 1.000)
(define n_width 0.800)

(define pillar_width (+ n_width i_height i_height p_height p_height))
(define pillar_height 4.000)
(define pillar_spacing 2.000)

; calculated values
(define half_spacing (/ pillar_spacing 2))
(define pillar_i_width n_width)
(define pillar_i_height (- pillar_height p_height i_height))
(define pillar_m_width (+ n_width i_height i_height))
(define pillar_m_height (- pillar_height p_height))
(define w (+ pillar_width pillar_spacing))

; polarity
(define n_polar #f)

; meshing
(define fine_eps_1 0.200)
(define fine_eps_2 0.100)

; Layers, top is surface at y=0. Set n_polar variable to control polarity
; N-polar will be on positive side, Ga-polar on negative side
; When plotting multiple structures, they will be aligned to the surface
; For graded layers, first number is top, second number is bottom
; region name; mat
erial; height; width; custom mesh; doping (+n -p); x mole; x-offset
(define layers (list
    (list "p" "GaN" p_height w "no" p_doping 0 0)
    (list "i" "GaN" i_height w "no" i_doping 0 0)
    (list "n" "GaN" n_height w "no" n_doping 0 0)
))

; contacts
; contact name; location
(define contacts (list
; (list "elec" "surface")
    (list "cathode" "bulk")
))

(if n_polar (define op +) (define op -))
(define y 0)
(define total_height 0)
(for-each (lambda (layer)
  (define region   (car layer))
  (define material (car (cdr layer)))
  (define height   (car (cdr (cdr layer))))
  (define width    (car (cdr (cdr (cdr layer)))))
  (define yref     (car (cdr (cdr (cdr (cdr layer))))))
  (define doping   (car (cdr (cdr (cdr (cdr (cdr layer)))))))
  (define xmole    (car (cdr (cdr (cdr (cdr (cdr (cdr layer)))))))))
  (define x        (car (cdr (cdr (cdr (cdr (cdr (cdr (cdr layer)))))))))
  (display (string-append "creating layer " region "n")

  (sdegeo:create-rectangle (position x y 0) (position width (op y height) 0) material region)

  (if (number? yref)
    (util:define-refinement-region-2d region width yref width yref)
)

  (set! y (op y height))
  (set! total_height (+ total_height height))

) layers)

; ==============================================================
; === Custom Device Material ===
; ==============================================================

(define x1 0)
(define y1 0)
(define x2 0)
(define y2 0)
; polygon test
(sdegeo:create-polygon
(list (position 0 0 0))
(position 0.5 0.5 0)
(position 0.5 0 0)
"GaN" "Substate")

; outer pillar - pGaN
(set! x1 half_spacing)
(set! y1 0)
(set! x2 (+ x1 pillar_width))
(set! y2 pillar_height)
(sdegeo:create-rectangle (position x1 y1 0) (position x2 y2 0) "GaN" "outer")

; middle pillar - uGaN
(set! x1 (+ half_spacing p_height))
(set! y1 (- p_height))
(set! x2 (+ x1 pillar_m_width))
(set! y2 (+ y1 pillar_height))
(sdegeo:create-rectangle (position x1 y1 0) (position x2 y2 0) "GaN" "middle")

; inner pillar - nGaN
(set! x1 (+ half_spacing p_height i_height))
(set! y1 (- 0 p_height i_height))
(set! x2 (+ x1 pillar_i_width))
(set! y2 (+ y1 pillar_height))
(sdegeo:create-rectangle (position x1 y1 0) (position x2 y2 0) "GaN" "inner")

(util:define-global-refinement-2d 0.1 0.1 0.1 0.1) ; default mesh - 100nm x 100nm

; ===================================================================
; == Define a custom meshing scheme ==
; ===================================================================

; Custom meshing at p-i pillar interface
(define p1 (position 0 (- fine_eps_1 p_height) 0))
(define p2 (position (- (+ half_spacing p_height) fine_eps_1) (- 0 p_height fine_eps_1) 0))
(define p3  (position (+ half_spacing p_height fine_eps_1) (- fine_eps_1 p_height) 0))
(define p4  (position (- (+ half_spacing p_height) fine_eps_1) (- pillar_height p_height fine_eps_1) 0))
(define p5  (position (+ half_spacing p_height fine_eps_1) (+ (- pillar_height p_height) fine_eps_1) 0))
(define p6  (position (- (+ half_spacing pillar_width) p_height fine_eps_1) (- pillar_height p_height fine_eps_1) 0))
(define p7  (position (- (+ half_spacing pillar_width fine_eps_1) p_height) (+ (- pillar_height p_height) fine_eps_1) 0))
(define p8  (position (- (+ half_spacing pillar_width fine_eps_1) p_height) (- fine_eps_1 p_height) 0))
(define p9  (position (- (+ half_spacing pillar_width) p_height fine_eps_1) (- 0 p_height fine_eps_1) 0))
(define p10 (position w (- 0 p_height fine_eps_1) 0))

(util:define-refinement-2d "middle_1" p1 p2 0.1 0.01 0.1 0.01)
(util:define-refinement-2d "middle_2" p2 p3 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "middle_3" p3 p4 0.01 0.1 0.01 0.1)
(util:define-refinement-2d "middle_4" p4 p5 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "middle_5" p5 p6 0.1 0.01 0.1 0.01)
(util:define-refinement-2d "middle_6" p6 p7 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "middle_7" p6 p8 0.01 0.1 0.01 0.1)
(util:define-refinement-2d "middle_8" p8 p9 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "middle_9" p8 p10 0.1 0.01 0.1 0.01)

; Custom meshing at n-p pillar interface
(set! p1  (position 0 (- fine_eps_2 p_height i_height) 0))
(set! p2  (position (- (+ half_spacing p_height i_height) fine_eps_2) (- 0 p_height fine_eps_2 i_height) 0))
(set! p3  (position (+ half_spacing p_height i_height fine_eps_2) (- fine_eps_2 p_height i_height) 0))
(set! p4  (position (- (+ half_spacing p_height i_height) fine_eps_2) (- pillar_height p_height i_height fine_eps_2) 0))
(set! p5  (position (+ half_spacing p_height i_height fine_eps_2) (+ (- pillar_height p_height i_height) fine_eps_2) 0))
(set! p6  (position (- (+ half_spacing pillar_width) p_height i_height fine_eps_2) (- pillar_height p_height i_height fine_eps_2) 0))
(set! p7  (position (- (+ half_spacing pillar_width fine_eps_2) p_height i_height) (+ (- pillar_height p_height i_height) fine_eps_2) 0))
(set! p8  (position (- (+ half_spacing pillar_width fine_eps_2) p_height i_height) (- fine_eps_2 p_height i_height) 0))
(set! p9  (position (- (+ half_spacing pillar_width) p_height i_height fine_eps_2) (- 0 p_height i_height fine_eps_2) 0))

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(set! p10 (position w (- 0 p_height i_height fine_eps_2) 0))

(util:define-refinement-2d "inner_1" p1 p2 0.1 0.01 0.1 0.01)
(util:define-refinement-2d "inner_2" p2 p3 0.01 0.01 0.1 0.01)
(util:define-refinement-2d "inner_3" p3 p4 0.01 0.1 0.01 0.1)
(util:define-refinement-2d "inner_4" p4 p5 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "inner_5" p5 p6 0.1 0.01 0.1 0.01)
(util:define-refinement-2d "inner_6" p6 p7 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "inner_7" p6 p8 0.01 0.1 0.01 0.1)
(util:define-refinement-2d "inner_8" p8 p9 0.01 0.01 0.01 0.01)
(util:define-refinement-2d "inner_9" p8 p10 0.1 0.01 0.1 0.01)

; set doping and x mole fractions
(set! y 0)
(for-each (lambda (layer)

  (define region (car layer))
  (define material (car (cdr layer)))
  (define height (car (cdr (cdr layer)))))
  (define width (car (cdr (cdr (cdr layer)))))
  (define yref (car (cdr (cdr (cdr (cdr layer)))))
  (define doping (car (cdr (cdr (cdr (cdr (cdr layer)))))))
  (define xmole (car (cdr (cdr (cdr (cdr (cdr (cdr layer))))))))
  (define x (car (cdr (cdr (cdr (cdr (cdr (cdr (cdr layer))))))))))

; set doping
(util:set-region-doping region doping)

; set x mole fractions
(cond
  ((pair? xmole)
    (util:set-region-xmole-graded region xmole height y n_polar)
  )
  ((number? xmole)

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(util:set-region-xmole region xmole)
)
)

(set! y (op y height))
)
) layers)

(util:set-region-doping "inner" n_doping)
(util:set-region-doping "middle" i_doping)
(util:set-region-doping "outer" p_doping)

; create contacts
; TODO: handle x-y contacts
(for-each (lambda (contact)
  (define name (car contact))
  (define location (car (cdr contact)))

  (util:create-side-contact name location total_height n_polar)
  ) contacts)
)

; contacts
(set! x1 half_spacing)
(set! y1 0)
(set! x2 (+ half_spacing pillar_width))
(set! y2 pillar_height)
(define eps 0.001)

(util:create-xy-contact "anode" eps y1)
(util:create-xy-contact "anode" x1 (+ y1 eps))
(util:create-xy-contact "anode" (+ x1 eps) y2)
(util:create-xy-contact "anode" x2 (+ y1 eps))
(util:create-xy-contact "anode" (+ x2 eps) y1)

(render:rebuild)
(sde:save-model name)
(sde:build-mesh "snmesh" "-skipSameMaterialInterfaces -AI" name)

Semi-polar Core-shell 3D+Planar: Structure Script and Meshing Function

; Creating core-shell structure using polygons

; defining vacuum background
(sdegeo:create-rectangle
 (position (- 0 0.5) (- 0 0.5) 0)
 (position (+ w 0.5) (+ n_height p_width i_width 0.5 ) 0)
 "Vacuum" "vac")

; defining the pGaN shell layer
(sdegeo:create-polygon
 (list (position 0 (+ n_width i_width) 0)
   (position 0 (+ n_width i_width p_width) 0)
   (position (* 0.5 w) (* 0.5 n_width) i_width p_width) (* 0.75 n_height) 0)
   (position (- (* 0.5 w) (* 0.5 n_width) i_width p_width) (+ n_height i_width p_width) 0)
   (position (+ (* 0.5 w) (* 0.5 n_width) i_width p_width) (+ n_height i_width p_width) 0)
   (position (+ (* 0.5 w) (* 0.5 n_width) i_width p_width) (* 0.75 n_height) 0)
   (position (* 1.0 w) (+ n_width i_width p_width) 0)
   (position w (+ n_width i_width) 0)
)

"GaN" "player")

(util:set-region-doping "player" p_doping)
; defining the uGaN shell layer
(sdegeo:create-polygon
(list (position 0 n_width 0)
  (position 0 (+ n_width i_width) 0)
  (position (* -1 w) (* -0.5 n_width) (* 0.5 i_width) 0)
  (position (* -1 w) (* -0.5 n_width) (+ n_height i_width) 0)
  (position (+ (* 0.5 w) (* 0.5 n_width) i_width) (+ n_height i_width) 0)
  (position (+ (* 0.5 w) (* 0.5 n_width) i_width) (* 0.5 n_height) 0)
  (position (+ (* 1.0 w) (* 0.5 n_width) i_width) (* 0.5 n_height) 0)
  (position w n_width 0)
  (position w n_width 0)
  (position 0 n_width 0)
)
"GaN" "ilayer")
(util:set-region-doping "ilayer" i_doping)

; defining the nGaN core layer
(sdegeo:create-polygon
(list (position 0 0 0)
  (position 0 n_width 0)
  (position (- (* -0.5 w) (* -0.5 n_width))) n_width 0)
  (position (- (* -0.5 w) (* -0.5 n_width))) n_height 0)
  (position (+ (* 0.5 w) (* 0.5 n_width))) n_height 0)
  (position (+ (* 0.5 w) (* 0.5 n_width))) n_width 0)
  (position w n_width 0)
  (position w 0 0)
  (position 0 0 0)
)
"GaN" "nlayer")
(util:set-region-doping "nlayer" n_doping)
(define Value 0.001 0.001)

(util:define-global-refinement-2d 0.025 0.025 0.025 0.025) ; default mesh - 25 nm x 25 nm

; refinement around material interfaces
(sdedr:define-refinement-size "RefDef.Epi" 0.1 0.0125 0.0025 0.0025)
(sdedr:define-refinement-region "PlaceRF.Epi" "RefDef.Epi" "ilayer")

(sdedr:define-refinement-function "RefDef.Epi" "MaxLenInt" "ilayer" "nlayer" Value 2 "DoubleSide" "UseRegionNames")
(sdedr:define-refinement-function "RefDef.Epi" "MaxLenInt" "player" "ilayer" Value 2 "DoubleSide" "UseRegionNames")

; refinement around surfaces
(sdedr:define-refinement-size "RefDef.Vac" 0.5 0.5 0.5 0.5)
(sdedr:define-refinement-region "PlaceRF.Vac" "RefDef.Vac" "vac")

(sdedr:define-refinement-function "RefDef.Vac" "MaxLenInt" "vac" "player" Value 2 "DoubleSide" "UseRegionNames")