Improved N-Type 4h-Sic Epitaxial Layer Radiation Detectors and Noise Analysis of Front-End Readout Electronics

Khai V. Nguyen

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IMPROVED N-TYPE 4H-SiC EPITAXIAL LAYER RADIATION DETECTORS AND
NOISE ANALYSIS OF FRONT-END READOUT ELECTRONICS

by

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Submitted in Partial Fulfillment of the Requirements
For the Degree of Doctor of Philosophy in
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2017

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DEDICATION

I would like to dedicate this work to my dear wife, Lam, who has been a constant source of support and encouragement in this endeavor. I am very thankful to have you by my side. Without you, I would never have made it thus far.
ACKNOWLEDGEMENTS

First and foremost, I am sincerely grateful to my graduate advisor, Dr. Krishna C. Mandal for welcoming me into his research group. His tremendous enthusiasm, guidance, and support have enabled me to make great strides forward in my career and in my understanding of semiconductors. He has continued to inspire my passion for science from my first day in his class as an undergraduate electrical engineering student through to this day. I would also like to thank my committee members, Dr. Enrico Santi, Dr. Guoan Wang, and Dr. Yuriy V. Pershin for sacrificing a portion of their time to provide guidance and support of my work.

I would like to thank the Chairman of the Department of Electrical Engineering, Professor Roger Dougal, for his guidance and words of wisdom that set me on the path to success in both life and as an engineer.

For their contributions in the research performed in this work, I would like to thank the following people:

- Dr. Sandeep K. Chaudhuri, for his extraordinary effort and tireless patience invested in setting up the DLTS and passing on his knowledge of radiation measurements and instrument calibration to me.

- Dr. Shuguo Ma (College of Engineering and Computing, University of South Carolina) for performing XPS characterization.
• The staff of the Institute for Electronics and Nanotechnology (IEN) at Georgia Tech for organizing the equipment training schedule such that two visits were enough to be qualified and added as a user for all of the tools necessary for fabricating the edge-terminated detectors.

• Nat Patterson, David London, Ashley Burt, Alicia Williams, David Metts, Jenny Balestrero, Lauren Ridings and all those I have forgotten who have provided both their expertise and kind words throughout my time here at USC.

Finally, I would like to thank my fellow lab members, Dr. Sandeep K. Chaudhuri, Dr. Sandip Das, Dr. Mohammad A. Mannan, Kelvin Zavalla, Cihan Oner, and Towhid A. Chowdhuri for their scientific inquisitiveness, humor, friendship, and support.
ABSTRACT

Schottky barrier radiation detectors were fabricated on n-type 4H-SiC epitaxial layers (12 – 50 µm) grown by hot wall CVD process on highly nitrogen doped 4H-SiC (0001) substrates with 4-8° off-cut towards the [11\overline{2}0] direction. Ni/4H-SiC Schottky barrier radiation detectors, a very low leakage current of 0.18 nA at 250 V bias, revealing low thermal noise, was observed in current-voltage (I-V) measurements. Using a thermionic emission model, junction properties such as barrier height of ≥1.10 eV and an ideality factor of ≤1.29 were determined. An effective carrier concentration of $1.03\times 10^{15}$ cm$^{-3}$ was calculated by capacitance-voltage (C-V) measurement. Deep level transient spectroscopy (DLTS) was used to investigate electrically active defects in epilayer. Defect parameters such as activation energy, capture cross-section, and density of defects were calculated from Arrhenius plots. DLTS revealed the presence of shallow level defects related to titanium impurities, electrically active lifetime killer $Z_{1/2}$ defect, and deep level defects assigned as $EH_{6/7}$ which are related to carbon and carbon-silicon vacancies. The density of $Z_{1/2}$ defect, the most detrimental to detector performance, was $1.6\times 10^{12}$ cm$^{-3}$, orders of magnitude lower compared to other 4H-SiC detectors.

Detector performances were evaluated in terms of the energy resolution at full-width at half-maximum (FWHM) using pulse height spectroscopy (PHS) measurements with 0.1 µCi $^{241}$Am source. Charge collection efficiency was investigated using a drift-diffusion charge transport model. The energy resolution for 5.486 MeV alpha particles
was 166 keV with charge collection efficiency of 22.6%. Electronic noise analysis of front-end readout system was carried out in terms of equivalent noise charge (ENC) in order to study the contribution of white series noise, pink noise ($f$ parallel and $1/f$ series) and white parallel noise to the total electronic noise in the detection system.

New edge termination was developed using surface passivating layers of silicon dioxide ($\text{SiO}_2$) and silicon nitride ($\text{Si}_3\text{N}_4$) in order to improve detector performance. With edge termination, reverse leakage current of Ni/4H-SiC epilayer detector was improved significantly (nA to pA) leading to an increased signal-to-noise ratio. Improved Schottky properties such as barrier height of ~1.7 eV and diode ideality factor of ~1.07 were observed indicating a better surface uniformity that enhanced charge collection efficiency. C-V measurement confirmed a doping concentration of $2.4 \times 10^{14} \text{ cm}^{-3}$ ensuring a fully depleted (~20 µm) detector at bias voltages as low as ~70 V. DLTS analysis showed a decreased concentration of performance limiting $Z_{1/2}$ defect level and absence of EH$_{6/7}$ deep-levels with edge termination, ensuing a more complete charge collection. Alpha spectroscopy measurements revealed an improved detector energy resolution from ~0.7% to ~0.4% for 5.48 MeV alpha particles with edge termination.

4H-SiC epitaxial detector with ruthenium (Ru) Schottky barrier contact (in addition to Ni being used in above studies) was investigated for operation in harsh environments with high temperature and high radiation. Ru/4H-SiC Schottky detectors exhibited excellent rectification and improved junction properties, even without edge termination. However, inhomogeneity of the Schottky barrier heights was observed due to interfacial defects resulting from a solid-state reaction involving Ru, Si, and C. As a result, pulse-height spectra with $^{241}\text{Am}$ source were broad, and the three characteristic
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LIST OF ABBREVIATIONS

CT...................................................................................................Computer Tomography
C-V ..................................................................................................Capacitance-Voltage
CVD................................................................................................Chemical Vapor Deposition
CZT ..............................................................................................Cadmium Zinc Telluride
DLTS .........................................................................................Deep Level Transient Spectroscopy
ENC ............................................................................................Equivalent Noise Charge
FWHM .........................................................................................Full-Width at Half-Maximum
I-V ..............................................................................................Current-Voltage
keV .............................................................................................Kilo Electron Volt
MeV .............................................................................................Mega Electron Volt
MCA ............................................................................................Multi-Channel Analyzer
MSM ...........................................................................................Metal-Semiconductor-Metal
PHS ...........................................................................................Pulse-Height Spectroscopy
PVT ..............................................................................................Physical Vapor Transport
RT ...............................................................................................Room Temperature
RTA .............................................................................................Rapid Thermal Annealing
RU ...............................................................................................Ruthenium
SEM ...........................................................................................Scanning Electron Microscopy
SiC .............................................................................................Silicon Carbide
SIMS.................................................................Secondary Ion Mass Spectrometry
XPS.................................................................X-ray Photoelectron Spectroscopy
CHAPTER 1: GENERAL INTRODUCTION

1.1 DISSERTATION INTRODUCTION

With growing concerns about the nuclear proliferation and terrorism, there are needs for portable, high performance, nuclear spectrometers for surveillance of nuclear terrorism activities, safeguards of nuclear spent fuel, and verification of non-proliferation treaty. The heart of a nuclear spectrometer is a radiation sensor that detects and quantitatively measure the energy of ionizing radiation transferred from a radiation source to the detector material. The current detection systems are limited by their detection efficiency, stability of response, speed of operation, and physical size due to requirement of cryogenic cooling. To address this issue, in our research lab we are developing “all solid-state”, “direct readout” detectors that can detect x-ray, gamma-ray, alpha, beta, and neutrons, and are suitable for operation at room temperature and above.

The detector materials and electronic instrumentation play critical roles in detector parameters such as sensitivity (type of radiation the detector will detect), efficiency (percentage of radiation that was amplified to electrical signal); energy resolution (capability of distinguishing emitted energy from two isotopes) and signal-to-noise ratio. For a high-performance radiation detector, incident radiation must be captured accurately. The electron-hole pairs created due to ionizing radiation interacting with the detector material must be collected without being lost through recombination. The electrical signals produced must be amplified to precisely measure the type and
amount of energy imparted from the radiation interacting with the detector material. Hence, the semiconductor materials for radiation detectors must offer (i) large bandgap energy ($\geq 1.5 \text{ eV at } 300 \text{ K}$) contributing to low thermal noise, (ii) high resistivity ($\geq 10^{10} \Omega\text{-cm}$) for low leakage current, thereby low noise, (iii) high mobility-lifetime product ($\mu\tau \geq 10^{-3} \text{ cm}^2/\text{V}$) for good charge transport properties so that probability of charge collection will be high and recombination is low, (iv) high atomic displacement energy for radiation hardness and damage resistance, and (v) high thermal conductivity for high temperature operation.

Silicon Carbide (SiC) is an indirect wide bandgap semiconductor (3.27 eV at 300 K) with high thermal conductivity, high breakdown electric field, high carrier saturation drift velocity, and large displacement energy (22-35 eV) making it a suitable candidate for replacing conventional radiation detectors based on Si, Ge, CdTe, and CdZnTe [1]-[12]. SiC allows detector operation well above room temperature (~773 K) and extremely low leakage currents (low noise) at operating bias (~ tens of pA for 20 µm 4H-SiC epilayer). SiC is a radiation hard material because of high displacement energies of the constituent elements making it available for detectors that are deployed in harsh environments such as high radiation field found in nuclear energy plants and in upper atmosphere and outer space. Due to these excellent material properties, SiC based radiation detectors can be designed for portable and compact radiation detection needs in field deployment and standoff detection applications including national security, nuclear non-proliferation, nuclear energy plant, high energy astrophysics, medical imaging, and environmental safety.
The prospect of SiC Schottky diodes as alpha particle detectors was first reported by Babcock and Chang [13]. Ruddy et al. reported a resolution of 5.8% at a deposited energy of 294 keV and 6.6% at a deposited energy of 260 keV by alpha particles from a collimated $^{238}$Pu source [14]. Nava et al. reported very robust 5.48 MeV alpha particle signal in 4H-SiC epitaxial detectors with circular contacts of ~2 mm diameter [15]. However, they have not achieved a saturation of the charge collection efficiency even at a bias voltage of 200 V. In a later work Ruddy et al. reported an energy resolution of 5.7% for a deposited energy of 89.5 keV alpha particles from a collimated $^{148}$Gd source with 10 μm thick epilayer detector [16]. Since then there has been significant progress in SiC devices; especially, advancements in epitaxy growth technologies have led to higher quality material and reproducibility than bulk SiC [4], [17], [18]. However, the yield and performance of the devices are still limited by the underlying material. The critical factor limiting the resolution in SiC epilayer based radiation detectors are the presence of electrically active defects in the active region which originate from the growth and fabrication processes [19], [20]. Device killing defects such as micropipes can form in the active region when screw dislocations propagate through from the bulk substrate into the epilayer [21]-[23]. Furthermore, commercially available epitaxially grown SiC layers have a maximum thickness on the order of 150 μm with a residual n-type doping ~ $10^{14}$ cm$^{-3}$ that limits the depth of the depletion layer (detector’s active region) to less than 100 μm at reasonable bias voltages [24]-[26].

Thus, defect-device performance correlation study was a major part of this dissertation. A new deep-level passivation and edge termination technique that improved charge collection efficiency and stability was developed. Contact structures with nickel
and ruthenium Schottky contact that control the performance of semiconductor detectors were studied in order to achieve optimum detectors. In addition, electronic noise analysis of the front-end readout system was carried out in terms of equivalent noise charge (ENC), and charge collection efficiency was investigated to optimize energy resolution.

1.2 DISSERTATION OVERVIEW

This dissertation research was centered on device fabrication, electrical characterization using current-voltage (I-V) and capacitance-voltage (C-V) measurements, defect analysis using deep level transient spectroscopy (DLTS), and detector performance evaluation using Pulse-Height Spectroscopy (PHS) measurements with an $^{241}$Am source.

Four key investigations were carried out in this dissertation study:

(i) Fabrication and characterization of Ni/4H-SiC epilayer Schottky barrier detectors on 12 µm thick highly nitrogen doped n-type SiC epitaxial layer. These thinner detectors (12 µm compared to 20 µm SiC epilayer used in baseline detector) were studied to observe the impact of deep lying point and/or extended defects in the active region (in the width of the depletion region) on Schottky barrier properties and radiation detector performance.

(ii) New edge termination using passivating layers of SiO$_2$ and Si$_3$N$_4$ on the epitaxial surface surrounding the edge of the Ni contact was developed in order to reduce surface leakage current and improve energy resolution. Ni/4H-SiC Schottky detector on n-type 20 µm SiC epilayer were fabricated with SiO$_2$ or Si$_3$N$_4$ passivating layers, and their detector
properties were compared with non-passivated Ni/4H-SiC 20µm-epilayer detector to assess effectiveness of edge termination.

(iii) Electronic noise analysis of front-end readout system was carried out in terms of equivalent noise charge (ENC). The charge collection efficiency was investigated using a drift-diffusion charge transport model.

(iv) Finally, 4H-SiC epilayer based radiation detectors with ruthenium (Ru) have been developed for operation in harsh environments where high temperature and high radiation fluence would significantly degrade conventional devices (e.g. Si, Ge, CZT, and scintillator based detectors). Ru metal has high abrasion and fatigue resistance, low electrical resistance, and high melting point (2334 °C), making it a better choice than Ni for harsh environment application such as in nuclear power plant.

This dissertation is divided into six chapters. Chapter 1 provides the significance of nuclear radiation detector and motivation for selecting 4H-SiC as the detector of interest. This chapter discusses the requirements for high performance radiation detectors, examine the structure and properties of SiC pertinent to radiation detector fabrication, and briefly review the previous work on SiC radiation detector. Growth of 4H-SiC materials for radiation detector was also discussed in this chapter.

Chapter 2 includes fabrication, electrical characterization, and defect analysis of Ni/n-type 4H-SiC detectors on 12 µm (investigated for the first time) and 20 µm (baseline detector) epilayer. The chapter describes fabrication of monolithic metal-semiconductor-metal (MSM) Schottky detectors, reviews thermionic emission model used for detector characterization, and discusses the results of current-voltage (I-V),
capacitance-voltage (C-V), and defect characterization using deep level transient spectroscopy (DLTS).

Chapter 3 describes the performance of radiation detectors based on n-type 4H-SiC epitaxial layer grown on off-axis bulk SiC crystals as characterized using Pulse-Height Spectroscopy (PHS) measurements for radiation testing using alpha particles and low energy x-ray and gamma-ray sources. Electronic noise analysis of front-end readout system was carried out in terms of equivalent noise charge (ENC). Finally, charge transport and collection efficiency were investigated using a drift-diffusion charge transport model.

Chapter 4 includes experiments with silicon dioxide (SiO₂) and silicon nitride (Si₃N₄) passivating layers for edge termination in order to reduce surface leakage current and improve energy resolution. The junction properties of the fabricated detectors before and after edge termination were studied by I-V and C-V measurements. DLTS was used to evaluate defect levels in detectors following edge termination. Alpha spectroscopy measurements were carried out to assess the effectiveness of the passivating layers in terms of higher energy resolution.

Chapter 5 details the investigation with ruthenium (Ru) Schottky barrier detector fabricated on 50 μm n-type 4H-SiC epilayers for operation in harsh environments with high temperature and high radiation. The chapter includes results of current-voltage, capacitance-voltage, and defect characterization of Ru/4H-SiC epilayer Schottky diodes. DLTS were employed to investigate defect levels induced from radio frequency (RF) sputtering and rapid thermal annealing (RTA) of the Ru Schottky contacts in the 4H-SiC epilayers.
Finally, Chapter 6 concludes the research presented in this dissertation. A brief review of the current challenges and suggestions for future work are also provided.

1.3 STRUCTURE AND PROPERTIES OF 4H-SIC

Silicon carbide crystal lattice is structured from closely stacked silicon and carbon bilayers (also called Si-C double layers). Due to the variation of staking sequences of atomic planes in one certain direction, silicon carbide has many crystal lattice structures such as cubic, hexagonal and rhombohedral symmetry [27]. These different crystal lattice structures of SiC are known as polytypes, and alphabetical letters are used to identify the structure. For example, the letter ‘H’ in 4H-SiC specifies that this polytype of SiC has hexagonal symmetry. The integer number ‘4’ represents the repetition number of bilayers in the stacking sequence. Figure 1.1 shows the crystal structure of 4H-SiC. From the side view, the staking sequence of SiC crystal shows a zig-zag pattern which terminates with a silicon face on a surface and with carbon atoms on the opposing surface.

![Figure 1.1 Structure of 4H-SiC polytype.](image)
Due to the variation in the stacking sequence, different polytypes have significantly different optical and electrical properties such as band-gap, drift velocity, breakdown electric field strength, and the impurity ionization energies [27]-[30]. Among different polytypes, 4H-SiC is usually preferred for electronic devices due to wide bandgap energy and better charge transport properties, specifically high electron mobility [31]-[33]. Low dielectric constant of 4H-SiC compared to Si and Ge helps to reduce the detector capacitance for a given active detector volume, which in turn decreases the white series noise component. The high threshold displacement energy indicates the radiation hardness of the material. Table 1.1 shows the properties of the polytype 4H-SiC that are relevant for radiation detector fabrication.

<table>
<thead>
<tr>
<th>Property</th>
<th>4H-SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Band gap (eV)</td>
<td>3.27</td>
</tr>
<tr>
<td>Electron hole pair creation energy (eV)</td>
<td>7.7</td>
</tr>
<tr>
<td>Relative Dielectric Constant</td>
<td>9.7</td>
</tr>
<tr>
<td>Electron mobility (cm$^2$/V.s)</td>
<td>1000</td>
</tr>
<tr>
<td>Hole mobility (cm$^2$/V.s)</td>
<td>115</td>
</tr>
<tr>
<td>Saturation electron drift velocity (x10$^7$ cm/s)</td>
<td>2</td>
</tr>
<tr>
<td>Threshold displacement energy (eV)</td>
<td>22-35</td>
</tr>
<tr>
<td>Thermal conductivity (W/cmK)</td>
<td>4.9</td>
</tr>
</tbody>
</table>

1.4 GROWTH OF 4H-SILICON CARBIDE MATERIAL

High quality, defect-free semiconductor materials are needed to fabricate high-performance radiation detectors. Intrinsic defects and impurities may act as traps or
recombination/generation centers leading to poor charge carrier lifetime-mobility which reduces signal-to-noise ratio and thereby detector performance. Silicon carbide phase diagram does not show a liquid phase, meaning SiC sublimes before it melts, therefore SiC bulk crystals cannot be grown by solidification from melts. SiC bulk growth is usually done by a method based on physical vapor transport (PVT), where a solid source of silicon carbide is evaporated at high temperatures and the vapors then migrate and crystallize on a monocrystalline SiC seed kept at a lower temperature [34]. In this method of crystal growth, precise doping and uniformity cannot be controlled easily, and grown crystals often suffer from microscopic crystallographic defects. Efforts have been made to optimize the reactor design to better control thermal gradients inside the growth chamber in order to increase wafer size and to reduce defect density [35]. At present, 3-inch diameter substrates are commercially available from multiple vendors [36]. However, bulk SiC available in the market still has relatively high defect densities and impurity concentrations to be used for high performance detector device.

Hence, SiC device are fabricated not directly on the bulk SiC wafers, but on epitaxial layers grown on top of the bulk SiC wafer. Epitaxial layer can be grown in a more controlled and reproducible manner using techniques such as chemical vapor deposition (CVD) yielding much higher quality SiC material than bulk SiC [37]. Epitaxial growth in a CVD system entails heating of the substrate in the reactor chamber with flowing silicon and carbon containing gasses that decompose and deposit Si and C in a well-ordered fashion onto the substrate to grow a high quality thin crystalline layer.

The n-type 4H-SiC epitaxial layers used to fabricate radiation detectors for our study were grown on 50 - 100 mm wafers diced from highly nitrogen-doped 4H-SiC
(0001) substrates with a 4 - 8° offcut towards the [1120] direction. Epitaxial layers with 12 – 50 μm thickness were grown by hot-wall CVD system using dichlorosilane (SiH₂Cl₂, DCS) and propane (C₃H₈) as gas-phase precursors and hydrogen of 6 SLM as the carrier gas. A dilution ratio of ~ 1000 was used and flow rates of precursors were maintained to obtain a C/Si ratio of ~ 1.28. The growth temperature and pressure were 1550 °C and 80 - 120 torr, respectively. An in situ hydrogen etching of the substrate was performed at 1550 °C for 5 – 20 min prior to growth. With horizontal hot-wall reactor, a higher growth temperature (up to 2000 °C) could be reached with more efficient heating of the substrate [38]. In this technique, the precursor gases are utilized more efficiently, and consequently, a higher growth rate (up to 100 μm per hour) can be achieved.
CHAPTER 2: DETECTOR FABRICATION AND CHARACTERIZATION

2.1 OVERVIEW

4H-SiC detectors were fabricated on n-type (nitrogen-doped) 4H-SiC epitaxial layer using Schottky barrier contact. Ni/4H-SiC radiation detector fabricated on 12 µm epitaxial layer was investigated for the first time in this study along with 20 µm and 50 µm epilayer based detectors. Details of detector fabrication and theory behind Schottky contact are provided. Fabricated detectors were characterized using current-voltage (I-V) and capacitance-voltage (C-V) measurements to determine electrical properties such as leakage current, doping concentration, built-in potential, Schottky barrier height, and ideality factor. A thermionic emission model was used to determine Schottky barrier junction properties [39]. Built-in potential and barrier height specifies the amount of current flow through the junction and the ideality factor specifies the spatial uniformity of the barrier height across the diode surface [40]. These diode parameters are important characteristics to predict device performance. Leakage current at applied reverse bias across the detector is also an important property as the detector electronic noise increases with leakage current thereby reducing the overall detector resolution [41]. Presence of electrically active defects, which act as generation-recombination centers, can lead to increased detector leakage current. The defects can also act as trap centers which may lead to incomplete charge collection. Deep level transient spectroscopy (DLTS) measurements were carried out to investigate defect levels in the detector active region.
and to understand their impact on device performance. Low-leakage current and low-defect bearing 4H-SiC detectors were then chosen for performance evaluation with alpha-radiation source.

### 2.2 EPITAXIAL 4H-SILICON CARBIDE DETECTOR MATERIAL

The n-type 4H-SiC epitaxial layers used to fabricate radiation detector for our study were grown on 100 mm diameter wafers diced from highly nitrogen-doped 4H-SiC (0001) substrates with a 4 - 8° offcut towards the [11̅20] direction. Epitaxial layers (12 – 50 µm thick) were grown by hot-wall CVD system at a growth temperature of 1550 °C. A picture of n-type 4H-SiC epitaxial layer wafer diced into 10×10 mm² size pieces is shown in Figure 2.1.

![Figure 2.1 Photograph of an n-type 4H-SiC epitaxial layer wafer.](image)

Nomarski optical microscopy and scanning electron microscopy (SEM) revealed a micropipe defect density of less than 1 cm⁻². Secondary ion mass spectrometry (SIMS) measurement was performed to characterize the epilayer layer thickness and doping density. From the SIMS measurement, shown in Figure 2.2, the epilayer thickness can be
observed due to the large nitrogen concentration difference in the epilayer and the highly doped substrate. For this particular sample it was determined to be 12 μm thick with an average nitrogen doping concentration of approximately $8 \times 10^{15} \text{ cm}^{-3}$.

![Figure 2.2 SIMS measurement indicating a 4H-SiC epilayer thickness of 12 μm and nitrogen doping concentration of $\sim 8 \times 10^{15} \text{ cm}^{-3}$.](image)

2.3 **Ni/4H-SiC Schottky Barrier Detector Structure**

Single-pixel, planar metal-semiconductor-metal (MSM) detector structure was fabricated on n-type 4H-SiC epitaxial layers. In a planar detector structure, metal contacts are placed on both sides of the detector material. The cross-sectional schematic of 8×8 mm² detector is presented in Figure 2.3 showing 4H-SiC epilayer (active layer), 4H-SiC buffer epilayer, and 4H-SiC bulk substrate with circular nickel (Ni) contact deposited on the epilayer face (top contact) and larger square Ni bottom contact on the opposite side. The circular Schottky barrier contact on the epilayer surface forms the ‘detector window’ through which ionizing radiation is captured by the 4H-SiC epilayer. Therefore, characterization of this Schottky barrier junction is of great importance.
Figure 2.3 Cross-sectional view of Schottky barrier detector on 4H-SiC epitaxial layer.

Figure 2.4 Energy band diagram of: (a) a metal and an n-type semiconductor before contact and (b) after Schottky contacts between metal and n-semiconductor [42].

In the fabricated detector structure, top nickel (Ni) contact forms a Schottky contact with high-resistive n-type 4H-SiC epitaxial layer. Figure 2.4 shows energy band diagram of an ideal Schottky contact between a metal and an n-type semiconductor at thermal equilibrium. A metal-semiconductor contact is called Schottky contact when it has a rectifying effect providing current conduction at forward bias (metal to semiconductor) and a low saturation current at reverse bias (semiconductor to metal). The rectifying effect of Schottky contact arises from the potential barrier present at metal-
semiconductor junction that restricts charge carrier movements. The potential barrier height \((e\phi_{bo})\) for electron injection from the metal into the semiconductor conduction band \((E_c)\) is known as the Schottky barrier and is the difference between the metal work function \((\phi_m)\) and semiconductor electron affinity \((\chi)\):

\[
e\phi_{bo} = e\phi_m - e\chi
\]

where \(e\) is electron charge. Work function is the energy difference between the Fermi level \((E_F)\) to the vacuum level and electron affinity \((\chi)\) is the energy difference between the semiconductor conduction band edge \((E_c)\) and the vacuum level. In the ideal case, the Schottky barrier height remains constant with respect to the polarity of the applied voltage.

On the semiconductor side, the built-in potential barrier \((V_{bi})\) is the barrier for electron flow from semiconductor conduction band into the metal and is given by:

\[
e\phi V_{bi} = e\phi_{B0} - e\phi_n
\]

where \(\phi_s\) is semiconductor work function. Built-in potential \(V_{bi}\) increases or decreases with applied voltage. In ‘forward’ bias, where a positive voltage is applied to the metal in respect to the semiconductor, \(V_{bi}\) is reduced so electrons can flow more easily from semiconductor into metal.

Due to conduction band bending in a Schottky contact, an electric field develops which sweeps free electrons from the vicinity of the contact interface and creates a depletion region (also known as space charge region). The bands become flat at the edge of the depletion region, and the electric field falls to zero at the edge which persists throughout the semiconductor. The width of the depletion region, \(W_c\), for a Schottky barrier is dependent on applied voltage; it is mostly negligible at forward bias, but
increases with applied voltage at reverse bias. Width of the depletion region can be expressed as:

\[ W = \sqrt{\frac{2 \times V_{bi} \times \varepsilon \times \varepsilon_0}{e \times N_D}} \]  

where \( \varepsilon \) is the dielectric constant of the semiconductor material, \( \varepsilon_0 \) is the permittivity of vacuum, \( e \) is the electronic charge \( (1.6 \times 10^{-19} \text{ C}) \) and \( N_D \) is the effective doping concentration and \( V_{bi} \) is the built-in potential.

The thermally annealed bottom Ni-contact forms an Ohmic contact with the very low-resistive bulk 4H-SiC substrate. The energy band diagram of an ideal Ohmic contact in Figure 2.5 shows that there is no potential barrier to block electron flow, hence providing conduction in both directions.

![Energy band diagram of an ideal Ohmic contact between metal and n-type semiconductor](image)

Figure 2.5 Energy band diagram of an ideal Ohmic contact between metal and n-type semiconductor [42].

2.4 DETECTOR FABRICATION

Prior to detector fabrication, the SiC wafer was thoroughly cleaned using a modified Radio Corporation of America (RCA) cleaning process. The cleaning process starts with removal of organic contaminants (dust particles, grease, etc.) from the wafer surface using organic solvents (trichloroethylene, acetone, and methanol) at their respective boiling temperatures. Any organic residue left by the first step is then removed
using first ammonium hydroxide (NH₄OH) solutions with hydrogen peroxide (H₂O₂:NH₄OH = 5:1:1) and then piranha solution (H₂SO₄:H₂O₂ = 1:1). Finally, oxide layers are etched with hydrofluoric acid (HF) followed by a Type 1 DI water rinse prior to metal contact deposition.

To form back Ohmic contact, large Ni contact (~ 6 × 6 mm²) of 100 nm in thickness was deposited on the bulk side (C-face) of the 8 × 8 mm² 4H-SiC wafer using a Quorum Q150T DC sputtering and a shadow mask. This was followed by rapid thermal annealing (RTA) at 950°C for 2 minutes in high-purity argon (Ar).

Photolithography was then performed on the epilayer side of the sample to pattern a 3.8 mm diameter circular window for Schottky contact formation. The sample was coated with Microposit SC1813 positive photoresist. A Karl Suss MA-6 Mask Aligner was used to expose a 3.8 mm diameter area centered on the sample by a quartz mask. The sample was then submersed in Microposit MF-319 developer for 1 minute followed by Type1 DI water rinse and drying by compressed N₂ gas. A Ni Schottky contact (3.8 mm diameter circular shape) with an area of ~ 11.34 mm² and thickness of ~ 10 nm was deposited on top of the epitaxial layers (Si-face) through the shadow mask using a Quorum model Q150T sputtering unit followed by a lift-off process.

After fabrication, the detector was then mounted on a printed circuit board (PCB) designed and fabricated in our laboratory and wire bonded for proper electrical connection. The wire-bonding was done using very thin (25 µm) gold wire to ensure less scattering and obscurcation of the alpha particles from the wire-bond region. The PCBs were fitted with board-to-board connector pins in order to obtain plug-in modular
configuration for stable electrical connections. A photograph (top view) of a fabricated detector is shown in Figure 2.6.

Figure 2.6 Photograph of a 4H-SiC epitaxial Schottky barrier detector with circular nickel top contact mounted on a PCB. This PCB is designed and fabricated in our laboratory.

2.5 CURRENT-VOLTAGE MEASUREMENT

Current-voltage (I-V) measurements on the fabricated Schottky barrier 4H-SiC epitaxial detectors were carried out at room temperature (300 K) using a Keithley 237 HV SMU. I-V characterizations were performed by measuring the current flowing through the 4H-SiC detector at various forward as well as reverse voltage bias applied across the detector. The forward-biased response was used to study the behavior of the Schottky contacts in terms of barrier height and the diode ideality factor applying the thermionic emission model [40], [24-25]. The reverse I-V characteristics were used to determine the leakage current under operating conditions. As per a thermionic emission model, the voltage dependent junction current in a Schottky contact can be given by:

\[ I = A^* A T^2 \left( e \exp \left( \frac{-e}{k_B T \phi_B} \right) \left( \exp \left( \frac{e V}{\hbar k_B T} \right) - 1 \right) \right) \]

where \( A^* \) is the Richardson’s constant taken to be 146 A·cm⁻²K⁻² for 4H-SiC [43], \( A \) is the diode area, \( T \) is the absolute temperature, \( e \) is the electron charge \((1.6 \times 10^{-19} \text{ C})\), \( k_B \) is the Boltzmann constant \((8.62 \times 10^{-5} \text{ eV/K})\), \( \phi_B \) is the Schottky barrier height, \( V \) is the
applied voltage, and $n$ is the diode ideality factor. Using logarithm, the Equation 2.4 could be written as:

$$\log(I) = \frac{\beta V}{n} + \log(I_s)$$

where $I_s$ is the saturation current, $I_s = A^*A^2(T^2 (\exp^{-\beta \phi_B})$ and $\beta = q/k_B T$. Therefore, using current measurements at varying applied voltage and then plotting log(I) versus applied voltage bias, saturation current $I_s$ can be obtained from the intercept and the ideality factor ‘n’ could be measured from the slope using following equation:

$$n = \frac{1}{\text{slope} \times \frac{1}{\beta}}.$$  

Figure 2.7, a typical I-V characteristic, shows the variation of forward and reverse current as a function of applied bias voltage across the n-type 4H-SiC epitaxial Schottky detector with Ni-contact. Schottky behavior of the device is clearly visible from this I-V characteristic measurement at room temperature. The reverse bias leakage current at room temperature was found to be ~ 0.18 nA at a bias voltage of −250 V. A logarithmic plot of voltage-dependent junction current is shown in Figure 2.8 for 12 μm n-type 4H-SiC epitaxial Schottky detector with Ni-contact. A Schottky barrier height of 1.10 eV was determined by applying the thermionic emission model as discussed in Equation 2.5. The diode ideality factor of 1.29 was measured from the slope using Equation 2.6. An ideality factor greater than unity, indicates inhomogeneity of the Schottky barrier height across the metal contact, most probably due to the presence of generation-recombination centers or trap centers (defect centers) on the detector surface [7], [44]. Lower barrier height also occurs due to the presence of defects in the epilayer arising from the growth process and contact formation.
Figure 2.7 Variation of junction current for n-type 20 μm 4H-SiC epitaxial/Ni Schottky detector with forward and reverse applied bias at room temperature; schematic of the electrical circuit for I-V measurements is shown at top.

Figure 2.8 I-V characteristic with current on logarithmic scale showing the rectifying behavior of the Ni Schottky contact formed on the 12 μm 4H-SiC epilayer.
2.6 Capacitance-Voltage Measurement

Capacitance-voltage (C-V) measurements at room temperature under dark condition were carried out at a frequency of 1 MHz. The C-V measurements were performed to determine effective carrier concentration and built-in potential for n-type 4H-SiC epitaxial Schottky detectors. The junction capacitance of Schottky barrier contact depends on the depletion region width which is a function of applied voltage (V) and effective doping concentration ($N_{eff}$) as expressed in Equation 2.3. Using that equation, the following relationship can be derived between junction capacitance ($C$), effective carrier concentration ($N_{eff}$) and built-in potential ($V_{bi}$):

$$C = \varepsilon A / \left( \frac{2\varepsilon \times (V_{bi} - V)}{eN_{eff}} \right)^{1/2}$$

where $A$ is the area of the diode, $\varepsilon$ is the product of the relative permittivity in 4H-SiC and free space, and $e$ is charge of an electron. The above equation could be rewritten as below:

$$1/C^2 = \frac{2V_{bi}}{\varepsilon\varepsilon A^2 N_{eff}} + \frac{2V}{\varepsilon\varepsilon A^2 N_{eff}}.$$  

Applying the linear fit described in Equation 2.8, when $1/C^2$ is plotted against applied voltage bias, $V$, built-in voltage ($V_{bi}$) could be estimated, from the intercept, and the effective doping concentration ($N_{eff}$) could be determined from the slope using the following formula:

$$N_{eff} = \frac{2}{\varepsilon\varepsilon A^2 \times \text{slope}}.$$  

Once doping concentration and built-in voltage is determined, the barrier-height ($\phi_B$) can also be calculated from C-V measurements using the following equation:
where $N_C$ is the effective density of states in the conduction band of 4H-SiC and is taken equal to $1.6 \times 10^{19}$ cm$^{-3}$ [30].

Figure 2.9 shows a $1/C^2$ vs $V$ plot, also known as Mott-Schottky plot, obtained for the 12 $\mu$m n-type 4H-SiC epitaxial Schottky detector at 300 K. From the slope of the straight line $N_{eff}$ was calculated using Equation 2.9 and found to be $1.03 \times 10^{15}$ cm$^{-3}$ with the built-in potential $V_{bi}$ determined to be 1.91 V where the extrapolated line intersects the voltage axis. The higher built-in potential can be explained by the presence of a thin oxide layer at the metal-semiconductor interface which introduces an additional small series capacitance. Since the slope of the line in Figure 2.9 depends on the effective doping concentration, the slope would remain the same in presence of a thin oxide layer while the intercept with the voltage axis would be shifted to a higher value [45], [46] – [49]. The barrier height calculated from the C-V measurements using Equation 2.10 is 1.38 eV, which is slightly higher than the value of 1.10 eV obtained from the forward I-V characteristics. This is due to the fact that while the barrier-height obtained from forward I-V characteristics is affected by low Schottky barrier height locations in an inhomogeneous diode, the barrier height determined from C-V characteristic gives an average value for the whole diode [29], [50]. The larger value of barrier height calculated from the C-V measurements further confirms the inhomogeneity of the surface barrier height due to presence of defect center.
Figure 2.9 Mott-Schottky plot ($1/C^2$ vs. $V$ plot) of Ni/4H-SiC Schottky barrier detector fabricated on 12 μm thick n-type 4H-SiC epitaxial layer. Inset shows original C-V characteristic at 300 K.

2.7 DEFECT STUDY BY DEEP LEVEL TRANSIENT SPECTROSCOPY

Intrinsic defects, such as grain boundaries and dislocations, impurity related point defects or complexes have been reported in as-grown SiC epilayers [18], [50] - [53]. Many of these defects act as trap or recombination/generation centers and can lead to increased detector leakage current (thereby noise), loss of charge carriers and/or incomplete charge collection. The current-voltage and capacitance-voltage measurement of the fabricated Schottky barrier detector on 4H-SiC epitaxial layer showed evidence of non-uniform barrier height due to the presence of defect centers. Therefore, it is important to identify the electrically active defects in the epilayer and evaluate their role in affecting the ultimate detector performance.

The presence of deep level defects was investigated by deep level transient spectroscopy (DLTS) in a temperature range from 80 K to 800 K. DLTS measures the change in junction capacitance of the Schottky detector due to the emission of charge
carriers from the defects existing in the space charge region. A capacitance-DLTS (C-DLTS) spectrum is generated from the temperature dependent capacitance transients followed by a saturated trap filling pulse applied to a semiconductor junction. By analyzing the capacitance transients, the defect activation energies (ΔE), which is the energy separation between the trap level and the conduction band, capture cross-sections (σ_n), which determines if the defects may act as a trap or recombination/generation center, and defect concentration (N_i), which provides the extent of defect on device performance, can be determined [54], [55].

In C-DLTS mode, the thermally activated emission rate, e_n, can be expressed as:

\[ e_n = (\sigma_n \langle V_{th} \rangle N_C / g) \exp(-\Delta E / k_B T) \]  

where \( \sigma_n \) is the carrier capture cross section, \( \langle V_{th} \rangle \) is the mean thermal velocity, \( N_C \) is the effective density of states, \( g \) is the degeneracy of the trap level and was considered to be equal to 1, \( \Delta E \) the energy separation between the trap level and the carrier band, \( k_B \) is the Boltzmann constant (8.62 × 10^{-5} eV/K), and \( T \) is the absolute temperature. The emission rate is related to the capacitance transient by the following relationship:

\[ C(t) = C_0 + \Delta C \exp(-t e_n) \]  

where \( C_0 \) is the junction capacitance at steady-state reverse bias voltage, \( \Delta C \) is the difference in capacitance change measured within the rate window. The trap concentration \( N_i \) can be calculated using the following expression:

\[ N_t = 2 \left( \frac{\Delta C(0) / C}{C} \right) N_d \]
where $\Delta C(\theta)$ is the difference in capacitance change between the two edges of the filling pulse and $N_d$ is doping concentration. The peak position in DLTS spectroscopy depends on the rate window, $\tau$, which is defined by an initial delay set for the emission rate calculations following the termination of the filling pulse:

$$Initial \ delay \ (ms) = \frac{1}{(4.3 \times \tau)}$$  \hspace{1cm} 2.14

For defect characterization in 4H-SiC epitaxial Schottky barrier detector, a SULA DDS-12 modular DLTS system was used in a capacitance mode. The DLTS system is comprised of a pulse generator, a capacitance meter, a correlator module, a Janis VPF 800 LN$_2$ cryostat controlled by a Lakeshore LS335 temperature controller, and a PC based data acquisition and analysis software. The correlator module uses a modified double boxcar signal averaging algorithm and automatically removes DC background from the capacitance signals and measures the capacitance transient in a given rate window.

The DLTS measurements were carried out using a temperature range of 80 - 800 K at a heating rate of 0.05 Ks$^{-1}$ and a steady-state reverse bias of -2 V pulsed to 0 V to fill carrier traps present within the depletion width. DLTS software allows the collection of four DLTS spectra simultaneously with four independent correlator delay settings in a single temperature scan as shown in Figure 2.10. Five distinct negative peaks appeared at different temperatures corresponding to different defect levels indicating majority carrier (electron) traps. The activation energy ($\Delta E$), which corresponds to the energy difference between the trap level and the conduction band, was calculated for each defect level from the Arrhenius plots ($T^2/e_n$ vs 1000/$T$) shown in Figure 2.11. The defect parameters such as capture cross-sections ($\sigma_n$) and defect concentration ($N_t$) were determined from
the DLTS scans using the Equations 2.11 - 2.13 and summarized in Table 2.1. The observed defect level was then compared with the literature values, if available, reported by other researchers.

![DLTS spectra](image)

**Figure 2.10** DLTS spectra obtained using n-type Ni/4H-SiC epitaxial detector in the temperature range of: (a) 80 to 140 K using a smaller set of initial delays, and (b) 150 to 800 K using a larger set of initial delays.

![Arrhenius plot](image)

**Figure 2.11** Arrhenius plot for all the peaks obtained from the DLTS scans.

The activation energy for trap levels in Peak #1 was found to be $E_c - 0.17$ eV, which means this defect level is located at $0.17$ eV below the conduction band minimum.
(\(E_c\)). This is a shallow level defect, and from the available literature, can be identified as titanium (Ti) substitutional impurities in the Si sites. Similar defect levels at \(E_c - (0.160 \pm 0.010)\) eV were reported from DLTS studies of Ti\(^{+}\) implanted 4H-SiC which were attributed to the ionized titanium acceptor Ti\(^{3+}\) (3d\(^1\)) residing at hexagonal and cubic Si lattice [52], [56]. The presence of Ti impurity is likely introduced during the growth process from the Ti parts in the growth reactor. Other research groups also assigned defect level located at \(E_c - 0.16\) eV as Ti electron trap [53], and trap level located at \(E_c - 0.17\) eV as chromium or titanium impurities (acceptor like) in hexagonal position [51].

Table 2.1 Defect parameters obtained from the DLTS measurements

<table>
<thead>
<tr>
<th>Peak # as per DLTS Spectra</th>
<th>Capture Cross-Section, (\sigma_n) cm(^2)</th>
<th>Activation Energy, (\Delta E) eV</th>
<th>Defect Concentration (N_t) cm(^{-3})</th>
<th>Possible Trap Identity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak 1</td>
<td>2.51( \times 10^{15})</td>
<td>(E_c - 0.17)</td>
<td>3.6( \times 10^{13})</td>
<td>Ti(c); titanium substitutional impurities probably introduced at growth reactor</td>
</tr>
<tr>
<td>Peak 2</td>
<td>3.48( \times 10^{15})</td>
<td>(E_c - 0.68)</td>
<td>1.6( \times 10^{13})</td>
<td>(Z_{1/2}); electrically active defect affecting detector performance, probably originated from carbon-silicon vacancies or antisites</td>
</tr>
<tr>
<td>Peak 3</td>
<td>3.71( \times 10^{15})</td>
<td>(E_c - 1.05)</td>
<td>3.8( \times 10^{13})</td>
<td>(EH_5), electrically active defect originated form carbon cluster</td>
</tr>
<tr>
<td>Peak 4</td>
<td>3.14( \times 10^{17})</td>
<td>(E_c - 1.58)</td>
<td>5.6( \times 10^{12})</td>
<td>(EH_{6/7}); electrically active defect originated from carbon of carbon--silicon divacancies</td>
</tr>
<tr>
<td>Peak 5</td>
<td>1.53( \times 10^{11})</td>
<td>(E_c - 2.12)</td>
<td>3.5( \times 10^{12})</td>
<td>Unidentified</td>
</tr>
</tbody>
</table>

The position of defect level associated with Peak #2 was found at 0.68 eV below the conduction band edge. Several groups have reported the presence of a similar defect
level often designated as Z\(_{1/2}\) in n-type 4H-SiC [55]-[60]. Z\(_{1/2}\) center is reported to be an electrically active defect responsible for the reduction of carrier lifetime which is detrimental to detector performance [52], [60]. The exact microscopic structure is still unknown but most likely it is originated from carbon related vacancies, silicon and carbon vacancy complexes (V\(_{\text{Si}}\)+V\(_{\text{C}}\)) or antisite complexes (Si\(_{\text{C}}\)+C\(_{\text{Si}}\)) [53], [55], [58].

The peak #3 was related to a defect level located at 1.06 eV below the conduction band edge. Similar defect levels at \(E_{\text{c}}-1.03\) eV and at \(E_{\text{c}}-1.07\) eV were observed by other researchers [61], [62]. This defect level designated as EH\(_5\) has been found in ion irradiated 4H-SiC and has been attributed to a carbon cluster [58].

The position of peak #4 in the bandgap was calculated to be at \(E_{\text{c}}-1.58\) eV. The nearest match for this defect level reported in literature was at \(E_{\text{c}}-1.6\) eV and at \(E_{\text{c}}-1.55\) eV, which were designated as EH\(_{6/7}\) defect level and related it to carbon vacancies (V\(_{\text{C}}\)) or carbon-silicon di-vacancies [63], [64]. Further investigation is in progress to clearly resolve peaks #3 and #4. Several groups have tried various methods to resolve the level termed EH\(_{6/7}\) and have reported activation energies ranging from 1.35 eV to 1.58 eV for resolved EH\(_6\) and EH\(_7\) levels [52], [57], [65]-[67].

The peak #5 was related to a defect level located at 2.12 eV below the conduction band edge, and remains unidentified. No similar defect level has been reported in the literature to best of our knowledge. Further investigations are in progress.

A defect level in n-type 4H-SiC epitaxial located at 1.32 eV below the conduction band edge, which was observed by our research group and others, was not present in this particular sample [18], [50], [58] [61]. This defect was assigned to be defect center Ci1 in a chlorine implanted n-type 4H-SiC epitaxial layer [61].
2.8 SUMMARY OF Ni/4H-SiC DETECTOR CHARACTERIZATION

A total of four different Ni/4H-SiC detectors were investigated that were fabricated on 12 μm or 20 μm thick n-type 4H-SiC epitaxial layer using the fabrication process described in this chapter. These detectors are listed in Table 2.2. Among these detectors, 12 μm Ni/4H-SiC epilayer were investigated for the first time as radiation detector [68]. Detector 2AS1a was fabricated using relatively higher quality n-type 4H-SiC and used for front-end readout electronics noise analysis as described in Chapter 3. An additional two 20 μm detectors provided baseline data for further study with edge termination described in Chapter 4. Being fabricated from the same substrate, they could also be used to demonstrate detector reproducibility. Applying different characterization techniques described in above sections of this chapter, these detectors were studied for their Schottky barrier properties, leakage current, doping concentration, and defect concentration. The results of this analysis are summarized in Table 2.3.

Table 2.2 Baseline Ni/4H-SiC epitaxial detectors

<table>
<thead>
<tr>
<th>Detector ID</th>
<th>Epilayer Thickness</th>
<th>Purpose Served</th>
</tr>
</thead>
<tbody>
<tr>
<td>1AS1</td>
<td>12 μm</td>
<td>Investigated for the first time to use as radiation detector</td>
</tr>
<tr>
<td>2AS1a</td>
<td>20 μm</td>
<td>Fabricated with higher quality epitaxial layer and used for electronic noise analysis</td>
</tr>
<tr>
<td>2AS22x</td>
<td>20 μm</td>
<td>Detector used to assess edge termination with SiO₂</td>
</tr>
<tr>
<td>2AS30x</td>
<td>20 μm</td>
<td>Detector used to assess edge termination with Si₃N₄</td>
</tr>
</tbody>
</table>
Table 2.3 Summary of characterization for baseline Ni/4H-SiC epitaxial detectors

<table>
<thead>
<tr>
<th>Detector ID</th>
<th>Leakage Current at -200V (nA)</th>
<th>Barrier Height (eV)</th>
<th>Diode Ideality Factor</th>
<th>Doping Concentration (×10^{14} cm^{-3})</th>
<th>Density of Z_{1/2} Defect (×10^{14} cm^{-3})</th>
<th>Capture Cross-section of Z_{1/2} Defect (×10^{-16} cm^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1AS1</td>
<td>0.18^1</td>
<td>1.10</td>
<td>1.29</td>
<td>10.3</td>
<td>1.58</td>
<td>9.12</td>
</tr>
<tr>
<td>2AS1a</td>
<td>0.12</td>
<td>1.39</td>
<td>1.18</td>
<td>1.8</td>
<td>4.16</td>
<td>5.04</td>
</tr>
<tr>
<td>2AS22x</td>
<td>4.72</td>
<td>1.01</td>
<td>1.35</td>
<td>2.86</td>
<td>1.6</td>
<td>34.8</td>
</tr>
<tr>
<td>2AS30x</td>
<td>4.52</td>
<td>1.00</td>
<td>1.24</td>
<td>2.42</td>
<td>Not tested</td>
<td>Not tested</td>
</tr>
</tbody>
</table>

^1measured at -250 V for detector 1AS1

2.9 CONCLUSION

Schottky barrier radiation detectors were fabricated on n-type 4H-SiC epitaxial layers grown on a 4° off-axis highly doped 4H-SiC substrate (0001). Planar single-pixel MSM detector structure with Ni Schottky top contact (detection window) was used. Schottky barrier properties were characterized through current-voltage (I-V) and capacitance-voltage (C-V) measurements. The room temperature I-V measurements revealed a very low leakage current ranging from a few to a fraction of nA at 200 V reverse bias. The barrier height for Ni/4H-SiC Schottky contact was found to be in a range of 1 to 1.4 eV by forward I-V measurement. From a Mott-Schottky plot of the C-V measurements, the effective carrier concentration was calculated and found to be in a range of 1.8 - 10 \times 10^{14} \text{ cm}^{-3}. Using a thermionic emission model, the diode ideality factor was determined to be ≥1.18, which is higher than ‘unity’ indicating the presence of deep levels as traps or recombination centers in the detector volume. Capacitance-mode deep level transient spectroscopy (DLTS) measurements were carried out to investigate the defect levels in the detector active region. DLTS revealed the presence of a shallow level defect related to titanium impurities, electrically active lifetime killer Z_{1/2} defect
level, and two deep level defects assigned as EHs, and EH_{6/7} which are related to carbon or carbon-silicon di-vacancies. The concentration and capture cross-section of Z_{1/2}, which is the most detrimental defects to detector performance, were determined to be $\geq 1.6 \times 10^{14}$ cm$^{-3}$ and $\geq 5 \times 10^{-16}$ cm$^2$, respectively. An unidentified deep electron trap located at 2.12 eV below the conduction band minimum was observed, but the nature and origin of the defect is unknown. Thus, the electrical characterization along with defect characterization of the fabricated n-type 4H-SiC epitaxial layer detector provided useful information and serves as a quality control tool in selecting detectors that would be suitable for spectroscopic characterization discussed in the next chapter.
3.1 **Overview**

The response of the detectors to alpha particles was evaluated by irradiating the detector with 0.1 $\mu$Ci $^{241}$Am alpha source which provides low-energy gamma-rays at 59.6 keV or alpha particles at 5.486 MeV and by recording the pulse-height spectrum produced from detector measurements. Front-end readout electronics consisted of preamplifiers which converts charge signal generated by incident alpha particles to a voltage signal, shaping amplifier that filters noise, and multi-channel analyzers (MCA) which converts analog signals into digital information as pulse-height spectrum. Detector performance was evaluated in terms of energy resolution of the detection peak obtained by PHS. The energy resolution was calculated at full width at half maximum (FWHM) of the alpha energy peak using Gaussian peak fitting function. The charge collection efficiency (CCE) is the ratio of the output energy observed by the detector to the actual incident energy of the alpha particles (5.48 MeV), and was calculated as a function of reverse bias using a drift-diffusion charge transport model. Finally, electronic noise analysis of front-end readout system was carried out in terms of equivalent noise charge (ENC) in order to study the contribution of white series noise, pink noise ($f$ parallel and $1/f$ series) and parallel white noise to the total electronic noise in the detection system.
3.2 **FRONT-END ELECTRONICS**

When radiation detectors are irradiated with $^{241}$Am alpha source, incident ionizing radiation (alpha particles from $^{241}$Am source) interact with the semiconductor material (4H-SiC), and generate electron-hole pairs. The generated charge carriers are swept out due to applied bias and are collected at the respective electrodes giving rise to an electrical signal. This electrical signal is then converted by the front-end electronics to provide pulse-height spectra for the incident radiation. Figure 3.1 shows the schematic diagram of the detection testing setup.

![Schematic diagram of the detection testing setup](image)

*Figure 3.1 Schematic of the detector testing electronics.*

The charge signals generated by the interaction of the alpha particles with the 4H-SiC radiation detector were amplified with an Amptek CoolFet (A250CF) preamplifier and an Ortec 671 spectroscopy amplifier. The preamplifier allows the charge signal to be converted to a voltage signal, which can be sent over to a shaping amplifier. This is a critical step because noise introduced in this stage of signal processing can significantly affect the resulting detection spectrum. The preamplifier pulse output is shaped by the shaping amplifier, which filters out noise while preserving information about the energy of the radiation imparted to the detector resulting in a semi-Gaussian...
amplified output. The shaping amplifier spends a set period of time measuring the signal, which is known as the shaping time. After shaping, the amplified pulses are fed into a Canberra Multiport II ADC multichannel analyzer (MCA) which is controlled by Genie-2000 interface software and converts the analog signals into digital information. The MCA records the height of the shaped pulse and the number of pulse-heights acquired within a given range yielding a histogram known as “Pulse-Height Spectrum”. PHS depicts how many counts of radioactive photons interacted with the detector in a given energy window.

In order to find a correlation between the channels of the MCA with their corresponding energy, a calibration process is performed using the same configuration as the setup used for detector testing. To calibrate the system, a precision pulser, which generates waveforms and simulates the output of a radiation detector, is connected to the detection system through a capacitor. By injecting pulses of various known amplitudes \( V_{\text{pulser}} \), energy of the charge pulses from the capacitor, \( E_{\text{pulser}} \) (in keV) can be determined by the following expression:

\[
E_{\text{pulser}} = \frac{V_{\text{pulser}} \times \epsilon \times C}{1.6 \times 10^{-19}}
\]

where \( \epsilon \) is the electron-hole pair creation energy (7.7 eV for 4H-SiC) [72], [6]. A graphical plot between \( E_{\text{pulser}} \) and the corresponding MCA peak positions for different pulse-heights gives the calibration graph. Figure 3.2 shows one MCA spectrum with various pulser peak-positions taken during calibration. The linear plot of the peak centroid channel number against the pulser energy in keV gives the required calibration parameters [6]. Centroid is the “center of mass” of the peak energy in keV.
3.3 **Pulse-Height Measurements with $^{241}\text{Am}$ Alpha Source**

Response of the fabricated Ni/4H-SiC epitaxial Schottky detectors to radiation was evaluated by irradiating the detector with 0.1 $\mu$Ci $^{241}\text{Am}$ source (peak energies: 60 keV for $\gamma$ and 5.5 MeV for $\alpha$ particles) at room temperature (~300 K). To minimize electromagnetic interference, the radiation source and the 4H-SiC detector were placed inside an EMI shielded aluminum box, with source kept at a distance of 1.5 cm from the detector. Detector performance was evaluated in terms of energy resolution of the detection peak obtained by PHS. The energy resolution was calculated as full width at half maximum (FWHM) of the alpha energy peak using Gaussian peak fitting function. The energy resolution of the detector was calculated by the following equation:

$$\% \text{ Energy Resolution} = \frac{FWHM \ (keV)}{Incident \ Energy \ (keV)} \times 100\%$$  \hspace{1cm} (3.2)

where the incident energy is the centroid (center of the mass) of the energy peak in keV observed in the pulse-height spectrum. Lower values of energy resolution and FWHM indicate better detector performance.
Figure 3.3 Alpha pulse-height spectroscopy collected from the 12 μm epilayer 4H-SiC radiation detector using a broad window 1 μCi $^{241}$Am source.

The Schottky barrier radiation detector fabricated on 12 μm thick 4H-SiC epilayer was evaluated by alpha pulse-height spectroscopy (PHS) shown in Figure 3.3. At a 3 V reverse bias, the energy resolution was determined to be 166 keV and charge collection efficiency (CCE) of 22.6%. In 4H-SiC, the stopping range of 5.486 MeV alpha particles is approximately 18 μm from SRIM calculations [70]. Since a 3 V reverse bias is not sufficient to fully deplete the epilayer active region with an effective doping concentration of $1.03 \times 10^{15}$ cm$^{-3}$, a large portion of the charge carrier generation occurs in the neutral region of the detector where charge carrier diffusion would dominate the transport properties. A higher CCE could be achieved by increasing the reverse bias voltage which would extend the depletion region and lead to higher contribution of charge carrier drift to the CCE. This assumes leakage currents do not significantly increase as was the case for the fabricated detector in this measurement which may be due to the presence of electrically active defects in the epilayer [23] – [26].
The percentage energy resolution of a Ni/4H-SiC detector fabricated on high-quality 20 μm thick n-type 4H-SiC epitaxial layer is shown in Figure 3.4. The pulse-height spectra shows three clearly resolved peaks for three major alpha particle energies – 5388 keV, 5443 keV, and 5486 keV emitted from an $^{241}$Am source. An energy resolution of 0.38% was observed for 5486 keV alpha particles with this detector. This spectrum completely vanishes and counts become background noise when a piece of A4 white copying paper was placed in between the radiation source and detector, confirming the detector’s response to alpha particles. By comparing response with gamma radiation, it is clear that the peak is the distinctive signal of α-radiation.

![Alpha pulse-height spectra](image)

Figure 3.4 Alpha ($^{241}$Am) pulse-height spectra obtained for a high resolution Ni/4H-SiC detectors fabricated on 20 μm thick n-type 4H-SiC epitaxial layer.

### 3.4 Charge Collection Efficiency

Resolution of a radiation detector is a function of collected charge carriers generated by alpha particles, thus charge collection efficiency (CCE) provides an important measure of detector performance. Experimentally, CCE is calculated as the ratio of energy deposited in the detector ($E_v$) to the actual energy of the alpha particles (5.48 MeV) emitted by the source ($E_0$) given by:
The energy deposited was calculated from the alpha peak position in a calibrated MCA. The charge collection efficiency in theory is the sum of two contributions – CCE_{drift} and CCE_{diffusion}. CCE_{drift} is the contribution of charge carriers generated within the depletion region and drifted to collecting electrode. CCE_{diffusion} is the contribution of charge carriers generated in the neutral region behind the depletion region and diffused to the depletion region. These two types of charge collection efficiency could be determined separately using drift-diffusion model as described in the following equation [72].

\[
CCE_{theory} = CCE_{drift} + CCE_{diffusion} = \frac{1}{E_p} \int_0^d \left( \frac{dE}{dx} \right) dx + \frac{1}{E_p} \int_d^{x_r} \left( \frac{dE}{dx} \right) \times \exp \left\{ -\frac{x - d}{L_d} \right\} dx
\]

where \( E_p \) is the energy of the alpha particles, \( d \) is the depletion width at the particular bias, \( \frac{dE}{dx} \) is the electronic stopping power of the alpha particles calculated using SRIM 2012 [70], \( x_r \) is the projected range of the alpha particles with energy \( E_p \), and \( L_d \) is the diffusion length of the minority carriers.

The width of the depletion region, \( d \), for a Schottky barrier diode is dependent on the effective doping concentration of the semiconductor material and applied bias voltage. It is mostly negligible at forward bias, but increases with applied voltage at reverse bias. Width of the depletion region can be expressed as:

\[
d = \sqrt{\frac{2\epsilon\varepsilon_0(V_{bi} - V)}{eN_{eff}}} eN_{eff}
\]
where \( \varepsilon \) is the dielectric constant of the semiconductor material, \( \varepsilon_0 \) is the permittivity of vacuum, \( e \) is the electronic charge (\( 1.6 \times 10^{-19} \) C) and \( N_{eff} \) is the effective doping concentration and \( V_{bi} \) is the built-in potential.

Using a drift-diffusion model (Equation 3.4), total charge collection efficiency (\( CCE_{theory} \)), collection efficiency in depletion region (\( CCE_{drift} \)), and collection efficiency in neutral region (\( CCE_{diffusion} \)) were determined separately with varying applied bias using 5.48 MeV alpha particles. To have a better perspective, the variations in \( CCE_{theory} \) as a function of reverse bias voltage were compared with the experimentally obtained charge collection efficiency (\( CCE_{observed} \)) from pulse-height spectra using Equation 3.3. Figure 3.5 compares different CCE values with varying reverse bias voltages. The results shows that as applied reverse bias increases, width of depletion region increases allowing more number of generated electron-hole pairs to contribute toward \( CCE_{drift} \), thereby increasing total CCE. Both \( CCE_{observed} \) and \( CCE_{theory} \) improves with applied reverse biases up to a reverse bias of 90 V, then \( CCE_{observed} \) levels off, while \( CCE_{theory} \) keep increasing to reach 100% efficiency. At this point, charge collection is almost solely due to carrier drift inside the depletion region (\( CCE_{drift} \)). Any electron-hole pairs generated by the ionizing radiation (alpha particle in this case) outside of the depletion region do not contribute significantly toward total charge collection efficiency as a decreasing value for \( CCE_{diffusion} \) was observed.

Figure 3.5 shows the saturation of experimentally obtained CCE (\( CCE_{observed} \)) at \(~80\%\). Alpha particles of 5.48 MeV energy have a projected range (penetration depth) of 18 \( \mu \)m in SiC. The depletion width in the fabricated detector was calculated to be \(~12\ \mu \)m at a reverse bias of 90 V. So the alpha particles did not deposit their full energy within the
depletion region which is the active region of the detector. A CCE value less than 100% also suggests that a fraction of the generated charge carriers are getting trapped and eventually lost (recombine) in the defect centers. At higher reverse bias voltages, leakage current increases and more number of defects become involved which decreases charge collection efficiency.

![Figure 3.5](image)

Figure 3.5 Variation of experimentally obtained (□) CCE and theoretically calculated (○) CCE as a function of reverse bias voltage. CCE from drift (Δ) and diffusion (▽) calculated separately using drift-diffusion model are also shown. The solid line shows the variation in depletion width. The arrows indicate the respective y-axis for a given plot.

3.5 **Electronic Noise Analysis**

The charge signal seen by the detector requires immediate amplification to a voltage signal to prevent signal loss, due to the small amounts of charge involved. A preamplifier performs this initial amplification. Pre-amplification is a critical step for signal processing in the detection system because noise introduced in this stage of the detection setup can have a significant effect on the resulting pulse-height spectrum.
Figure 3.6 shows a basic charge-sensitive preamplifier circuit containing a high gain amplifier with a feedback capacitor and feedback resistor. The feedback capacitor makes the preamplifier insensitive to changes in the capacitance of the detector, while adding some noise to the circuit. A FET is usually used at the input of the high-gain amplifier, which must be carefully selected for noise consideration purposes.

![Simplified circuit diagram for a charge sensitive preamplifier used in radiation detection system.](image)

As detector resolution ultimately depends on the front-end electronics, the electronic noise in terms of equivalent noise charge (ENC) were carried out. ENC represents the contribution of different noise sources that influence in the radiation detection signal acquisition and processing and can be expressed as given [72]:

$$\text{ENC}^2 = \left( aC_{tot}^2 A_1 \right) \frac{1}{\tau} + \left[ \left( 2\pi a_f C_{tot}^2 + \frac{b_f}{2\pi} \right) A_2 \right] + (bA_3)\tau$$

where $a$ is the white series noise contribution from the thermal noise in the FET channel, $C_{tot}$ is the total input capacitance, $A_1$, $A_2$, and $A_3$ are constants which depend on the response of the shaping network, $\tau$ is the shaping time, $a_f$ is the coefficient of the FET 1/f noise, $b_f$ is the dielectric noise coefficient, and $b$ is the sum of the white parallel
noise contribution due to the shot noise of the FET, leakage current in the detector, and thermal noise in the feedback resistor.

For the ENC measurements, the experimental set up was the same as used for calibration procedure described in section 3.2. Pulse-height spectra were recorded using the precision pulser generating pulses at a fixed amplitude and frequency. The electronic noise was measured from the pulser peak width and expressed in terms of ENC in charge units. The ENC is plotted as a function of amplifier shaping time $\tau$ and fitted to Equation 3.6 using a least square estimation method implemented with Matlab coding [73]. This program was designed to apply the corresponding fitting to the experimental results in order to calculate the three components white series noise, pink noise ($f$ parallel and $1/f$ series) and white parallel noise.

In Figure 3.7, a variation of the ENC as a function of shaping time for the charge sensitive preamplifier is shown. In Figure 3.8, the noise components were observed to increase following the connection of the biased detector to the system compared to the preamplifier only in Figure 3.7. For this analysis Ni/4H-SiC detector on 12 $\mu$m epitaxial layer was used. By comparing both the figures, it is possible to see that the ENC is higher when the detector is connected to the system. The data revealed that the contribution of the white series noise, which is primarily due to the total input capacitance, to the overall electronic noise (ENC) dominate over the white parallel and the pink noise. The minimum ENC corresponded to a shaping time between 3 and 6 $\mu$s for the preamplifier only case (Figure 3.7). An increase in the parallel white noise contribution is observed when the detector is exposed to the $^{241}\text{Am}$ source due to the current from charge carrier generation. The minimum ENC for the biased detector under irradiation was observed to
be at a shaping time of 6 μs which corresponds to the minimum energy resolution of 166 keV. Previous analysis with the energy resolution dependence on the shaping time at -90V is in accordance with this data [74].

Figure 3.7 ENC plotted as a function of shaping time for the charge sensitive preamplifier with the input connected in series with a calibrated capacitor to the precision pulser. WSN, WPN and Pink is white series noise, white parallel noise, and pink noise (f parallel and 1/f series), respectively.

Figure 3.8 . ENC vs. shaping time for Ni/4H-SiC detector (on 12 μm epitaxial layer) under bias and exposed to the 241Am source while connected to the preamplifier.
3.6 CONCLUSION

Ni/4H-SiC epitaxial Schottky barrier detectors’ performances were evaluated using pulse-height spectrum (PHS) produced under a 0.1 μCi $^{241}$Am alpha source. At a 3 V reverse bias, the energy resolution for Ni/4H-SiC detector on 12 μm epitaxial layer was determined to be 166 keV and charge collection efficiency (CCE) of 22.6%. Using a precision pulser, which generates waveforms and simulates the output of a radiation detector, charge collection efficiency (CCE) was determined as a function of bias voltage. In 4H-SiC, the stopping range of 5.486 MeV alpha particles was approximately 18 μm from SRIM calculations. A 3 V reverse bias was not sufficient to fully deplete the epilayer active region, so a large portion of the charge carrier generated in the neutral region did not significantly contribute to CCE. A higher CCE could be achieved by increasing the reverse bias voltage which would extend the depletion region and lead to higher contribution of charge carrier drift to the CCE. The pulse-height spectra of Ni/4H-SiC detectors fabricated on high-quality 20 μm thick n-type 4H-SiC epitaxial layer showed clearly resolved peaks for three major alpha particle energies – 5388 keV, 5443 keV, and 5486 keV emitted from an $^{241}$Am source at 100V reverse bias. The energy resolution was calculated at full width at half maximum (FWHM) of the alpha energy peak using Gaussian peak fitting function. An energy resolution of 0.38% was observed for 5486 keV alpha particles with this detector. A CCE of ~80% was observed for this detector, which suggests that a fraction of the generated charge carriers are getting trapped and eventually lost (recombine) in the defect centers. The electronic noise analysis of the front-end readout electronics revealed that the contribution of the white series noise (which is primarily due to the total input capacitance) to the overall
electronic noise dominate over the white parallel and the pink noise. The minimum equivalent noise charge (ENC) for the biased detector under irradiation was observed to be at a shaping time of 6 μs which corresponds to the minimum energy resolution of 166 keV.
CHAPTER 4: IMPROVED PERFORMANCE USING SURFACE PASSIVATION AND EDGE TERMINATION

4.1 OVERVIEW

A new edge termination has been developed using thin (nanometer range) layers of silicon dioxide ($\text{SiO}_2$) and silicon nitride ($\text{Si}_3\text{N}_4$) on the epitaxial surface surrounding the edge of the metal contact in order to reduce surface leakage current. These dielectric layers deposited by plasma enhanced chemical vapor deposition (PECVD) also help to reduce the overall capacitance of the device drastically. These passivating layers are shown to be a very effective method for improving energy resolution and thereby radiation detection performance of n-type 4H-SiC epilayer Schottky barrier radiation detectors. The junction properties of the fabricated detectors before and after edge termination were studied by current-voltage (I-V) and capacitance-voltage (C-V) measurements. A thermionic emission model applied to the forward I-V characteristics showed surface barrier height of $\sim 1.4$ eV and diode ideality factor of $\sim 1.1$. The C-V measurements showed a doping concentration of $1.8 \times 10^{14}$ cm$^{-3}$ which ensured a fully depleted ($\sim 20$ $\mu$m) detector at bias voltages as low as $\sim 70$ V. Alpha spectroscopy measurements revealed an improved energy resolution from $\sim 0.7\%$ to $\sim 0.4\%$ for 5.48 MeV alpha particles. Deep level transient spectroscopy (DLTS) measurements have shown a decreased concentration of $Z_{1/2}$ defect levels in detectors following edge termination.
4.2 DETECTOR FABRICATION WITH EDGE TERMINATION

A 4H-SiC (0001) 76mm diameter wafer highly doped with nitrogen off-cut 4° towards the [11-20] direction was used as the substrate for 20 μm thick n-type epitaxial grown layers in which 8x8 mm² squares were diced for device fabrication. Wafers were evaluated by Nomarski optical microscopy and scanning electron microscopy for micropipe defect density and found to be less than 1 cm⁻². Following a modified RCA cleaning procedure, a Karl Suss MA6 mask aligner was used with a predesigned quartz photo-mask with 3.9 mm circular patch for center alignment of the metal contacts and exposure of the photoresist coated samples. Following UV exposure, substrates were dipped in a proper developer. Schottky barrier detectors were fabricated on the 8×8 mm² prepared samples by depositing thin (~10 nm) circular nickel (Ni) contacts (area ~12 mm²) using a Quorum Q150T sputtering unit and liftoff process. The thin Ni contact acts as the detector window with minimized dispersion in the case of incident photons [6]

SiO₂ or Si₃N₄ passivating layer was deposited on the epilayer surface using a STS plasma enhanced chemical vapor deposition (PECVD) with approximate thickness of 400 nm. Prior to passivation layer deposition, the deposition chamber was seasoned by running the process for five minutes with an empty chamber to ensure more of the deposition occurs on the sample during the actual deposition run. A cleaning process was run between the silicon dioxide and silicon nitride processes to prevent contamination. The process parameters used for silicon dioxide layer deposition were as follows: pressure 650 mtorr, power (13.56 MHz RF) 25 W, temp aux 250 °C, temp process 300 °C, 2% SiH₄ in N₂ 400 sccm, and N₂O 1420 sccm. The process parameters used for silicon nitride layer deposition were as follows: pressure 800 mtorr, power (13.56 MHz
RF) 25 W, temp aux 250 °C, temp process 300 °C, 2% SiH₄ in N₂ 2000 sccm, and NH₃ 40 sccm. Following the deposition process, a Nanospec Reflectometer was employed to measure the thickness of the passivation layer on the epilayer surface. The average thicknesses measured for the samples were 395 nm and 410 nm for silicon dioxide and silicon nitride, respectively.

A photolithography process was performed prior to reactive ion etching (RIE) to isolate a circular area centered on the epilayer for contact deposition. Due to the hardness of silicon carbide, a slight over-etch was safely used to ensure complete removal of the passivation layer within the exposed circular opening without damage to the epilayer. The process parameters were as follows: CHF₃ 45 sccm, O₂ 5 sccm, pressure set point 40 mtorr, RF set point 250 W. RIE was performed to open a window in the passivation layer of each detector followed by nickel metal contact deposition using a Quorum Q150T sputtering unit and subsequent liftoff technique to construct a passivation layer surrounding the metal contact. Schematic of the cross-sectional view of the fabricated detectors with and without edge termination is shown in Figure 4.1.

Three different devices were fabricated and will be referred to as 2AS1a (without edge termination), 2AS22 (SiO₂ edge termination), and 2AS30 (Si₃N₄ edge termination). The detector 2AS1a which already had both better electrical characteristics and energy resolution (even without edge termination) was used as reference to assess the effect of edge termination. The goal was to determine if edge termination could further improve detector performance. All three devices were fabricated from the same parent wafer. To evaluate the effect of edge termination, these detector devices were then characterized
using current-voltage (I-V) and capacitance-voltage (C-V) measurements, defect analysis by DLTS, and finally radiation detection performance with 0.1 µCi $^{241}$Am source.

![Diagram](image1)

(a)

![Diagram](image2)

(b)

Figure 4.1 Cross-sectional view of a fabricated Schottky barrier detectors on 4H-SiC n-type epitaxial layer: (a) without edge termination and (b) with edge termination; SiO$_2$ or Si$_3$N$_4$ passivating dielectric layer surrounds the Ni contact.

4.3 Characterization by XPS

X-ray photoelectron spectroscopy (XPS) was carried out on two 4H-SiC epitaxial layer samples deposited with SiO$_2$ and Si$_3$N$_4$ layers by PECVD. XPS measurements were performed using a Kratos AXIS Ultra DLD XPS system equipped with a hemispherical energy analyzer and a monochromatic Al Kα source. The Al Kα source was operated at
15 KeV and 150 W incident on surface at 45° with respect to the normal surface. The pass energy was fixed at 40 eV for the detail scans. The sample chamber was kept under ultra-high vacuum of $2 \times 10^{-9}$ torr and a high performance charge neutralizer was used to compensate for the sample surface charge. The binding energy of the analyzer was calibrated with an Ag foil to the value of 368.21±0.025 eV. The analysis area was around 0.3×0.7 mm$^2$. During XPS scanning, the reproducibility of spectra was assured by multiple scanning cycles to reach accuracy better than ±0.05 eV.

The XPS survey scan of the SiO$_2$ and Si$_3$N$_4$ passivated 4H-SiC epilayer samples are shown in Figure 4.2. Si 2p, C 1s, O 1s, N 1s and O 2s core level peaks were identified along with oxygen Auger peaks and are indexed in the figure. No other foreign impurities were detected in the samples. A strong oxygen peak is observed in the SiO$_2$ passivated sample originating from the oxygen in SiO$_2$, whereas a weak oxygen peak is detected in the Si$_3$N$_4$ passivated SiC, attributed to the adsorbed residual oxygen on the sample surface. Similarly, a strong N 1s peak is observed in the Si$_3$N$_4$ passivated sample originating from the nitrogen present in Si$_3$N$_4$ compared to a faint nitrogen peak in SiO$_2$ passivated SiC, which could be attributed to the unintentional introduction of nitrogen in the SiC lattice during the epilayer growth.

High resolution spectra of N 1s, O 1s, Si 2p, and C 1s core levels were acquired to further evaluate the oxidation states of the respective elements and are represented in Figure 4.3. C 1s was considered as the reference with a relative peak shift of ~0.4 eV between the two samples. Core level peaks of the elements could be de-convoluted after relative peak shift correction corresponding to C 1s in order to determine the oxidation states and to locate precise peak positions.
Figure 4.2 Survey spectra of SiO$_2$ and Si$_3$N$_4$ passivated 4H-SiC epilayers.

Figure 4.3 High-resolution core level spectra of N 1s, O 1s, Si 2p, and C 1s respectively.
4.4 **ELECTRICAL CHARACTERIZATION**

The electrical properties of Schottky barrier diodes were studied by current-voltage (I-V) and capacitance-voltage (C-V) measurements in an EMI-shielded aluminum box at room temperature. I-V measurements were performed using a Keithley 237 High Voltage Source Measure Unit. I-V measurements were performed for both forward and reverse bias voltages. For detector 2AS1a (detector without edge termination), the leakage current was observed to be ~0.25 nA at -250 V applied bias shown in Figure 4.4. An effective surface barrier height of 1.39 eV and a diode ideality factor of 1.18 were determined from the forward I-V characteristics by applying a thermionic emission model as discussed in Chapter 2 [39]. A diode having an ideality factor greater than unity indicates inhomogeneity in the barrier height which suggests the presence of charge traps in the depletion region [7], [23] – [26]. The edge termination is thus used to reduce leakage current and thereby improve the energy resolution of radiation detection peaks.

High frequency (1 MHz) C-V measurements were carried out to determine the effective doping concentration from Mott-Schottky plots. A linear fit of the $1/C^2$ vs. $V$ plot for the detector 2AS1a (without edge termination) at room temperature shown in Figure 4.5, gives an effective doping concentration ($N_{eff}$) of $1.8 \times 10^{14}$ cm$^{-3}$ by:

$$N_{eff} = \frac{2}{e\varepsilon_0\varepsilon_sA^2 \times \text{slope}}$$  \hspace{1cm} (4.1)

where $e$ is the electronic charge, $A$ is the area of the metal contact, $\varepsilon_0$ is the permittivity of free space, $\varepsilon_s$ is the permittivity of 4H-SiC (9.72), and slope is the linear fit of the curve shown in Figure 4.5.
Figure 4.4 Forward and reverse I-V characteristics obtained at room temperature for Schottky barrier detector fabricated on n-type 4H-SiC epilayer and without edge termination (device 2AS1a).

Figure 4.5 $1/C^2$ vs. $V$ characteristic at room temperature for the fabricated 4H-SiC epitaxial Schottky detector 2AS1a (without edge termination). Inset shows original C-V characteristic for this detector.
Figure 4.6 shows the C-V measurement results on the dielectric deposited 4H-SiC wafers. Although a difference in the capacitance behavior was observed in the forward and reverse bias regimes, the change in the capacitance value in a bias range of -12 to +12 V was noticed to be less than 2 pf in both the cases. Also it was noticed that the capacitance values obtained for the devices with the dielectric layer on are lower by an order of magnitude than those obtained from the devices with the same geometry but without any dielectric layer.

![Figure 4.6 C-V measurement results of the 4H-SiC epilayer detectors deposited with SiO$_2$ and Si$_3$N$_4$ dielectric layers.](image)

Figure 4.7 (a) shows the I-V characteristics for devices 2AS22 and 2AS30 before edge termination; forward I-V characteristics are shown in the inset. The detector leakage currents were observed to be ~5 nA at a reverse bias voltage of -200 V before edge termination. After the edge termination process, an improvement of approximately two orders of magnitude in leakage current was observed for 2AS22 and 2AS30 as shown in Figure 4.7 (b) at -200 V bias with 2 pA and 86 pA, respectively. From the forward I-V characteristics shown in the inset of Figure 4.7 (b), the diode ideality factors for 2AS22 and 2AS30 were calculated to be 1.07 and 1.09. Higher effective surface barrier heights,
1.68 eV for 2AS22 and 1.72 eV for 2AS30, were observed after edge termination. A summary of the detectors’ characteristics are shown below in Table 4.1.

Figure 4.7 Reverse I-V characteristics obtained at room temperature for the fabricated 4H-SiC n-type epitaxial Schottky barrier 2AS22 and 2AS30 prior to edge termination (a) and post edge termination (b). Inset shows the forward I-V characteristics.
Table 4.1 Summary of Detector Characteristics with and without Edge Termination

<table>
<thead>
<tr>
<th>Detector</th>
<th>Leakage current @ 200 V (A)</th>
<th>Barrier height (eV)</th>
<th>Diode ideality factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>2AS22 w/o passivation</td>
<td>4.72E-9</td>
<td>1.01</td>
<td>1.35</td>
</tr>
<tr>
<td>2AS22 w/ SiO₂ passivation</td>
<td>0.22E-11</td>
<td>1.72</td>
<td>1.07</td>
</tr>
<tr>
<td>2AS30 w/o passivation</td>
<td>4.52E-9</td>
<td>1.00</td>
<td>1.24</td>
</tr>
<tr>
<td>2AS30w/ Si₃N₄ passivation</td>
<td>8.58E-11</td>
<td>1.68</td>
<td>1.09</td>
</tr>
</tbody>
</table>

Note: Lower leakage current, higher barrier height, and ideality factor of ‘1’ correlate to better radiation detector performance.

The high frequency (1 MHz) C-V measurements were performed at room temperature for both fabricated detectors before and after passivation. From a linear fit of the Mott-Schottky plot shown in Figure 4.8, the effective doping concentration (N_{eff}) was calculated to be $2.24 \times 10^{14}$ cm$^{-3}$ and $2.86 \times 10^{14}$ cm$^{-3}$, respectively for 2AS22 and 2AS30.

Figure 4.8 $1/C^2$ vs. V characteristic at room temperature for the fabricated 4H-SiC epitaxial Schottky detectors 2AS22 and 2AS30 with edge termination. Inset shows original C-V characteristic for these detectors.
At -90 V, the depletion region width in the epilayer with \( N_{\text{eff}} = 2.86 \times 10^{14} \, \text{cm}^{-3} \) is approximately 20 µm (full-depletion condition) calculated using the formula

\[
W = \sqrt{\frac{2 \times V \times \varepsilon \times \varepsilon_0}{\varepsilon_0 \times N_{\text{eff}}}}
\]

where \( W \) is the width of the depletion region, \( V \) is the applied voltage, \( \varepsilon \) is the relative dielectric permittivity of SiC, \( \varepsilon_0 \) is the dielectric permittivity of vacuum, and \( e \) is the electronic charge.

4.5 DEFECT CHARACTERIZATION

For defect characterization, a SULA DDS-12 DLTS system was used in capacitance mode. The system uses a 1 MHz oscillator for capacitance measurements with a built-in pre-amplifier module. A 2 V reverse bias was applied in steady-state and a 0 V pulse was applied with a pulse width of 1 ms in the case of large correlator delays and 1 µs in the case of small correlator delays. Capacitance transient signals are digitized by a National Instruments’ (NI) digitizer card and processed using a PC. Software correlators used are based on a modified double boxcar signal averaging system in a dedicated Labview interface. This software allows the user to collect four DLTS spectra simultaneously with four independent correlator delay settings in a single temperature scan. For temperature variation of the sample, the detector was loaded into a Janis VPF-800 LN\(_2\) cryostat for temperature scans from 85K to 750K which was controlled by a Lakeshore LS335 temperature controller.

Figure 4.9 shows DLTS scans obtained from detector 2AS1a (device without edge terminating layer) mounted in a Janis VPF800 cryostat. Four DLTS peaks were observed in a single temperature scan from 85K to 750K.
Figure 4.9 DLTS scans are shown for a Ni/4H-SiC detector without edge termination; the observed negative peaks correspond to electron traps in the detector: (left) larger correlator delays were used to observe the deep defect levels; (right) shorter correlator delays with a lower temperature scan set were used to fully observe peak 1.

The shallow level defects (Peak #1) was slightly cut-off, so a second temperature scan from 85K to 550K was performed with shorter correlator delays to fully display the shallow level defects. From an Arrhenius plot obtained using different rate windows, the activation energies for the Peak #1, Peak #2, Peak #3, and Peak #4 were found to be 0.19 eV, 0.62 eV, 1.40 eV, and 1.45 eV below the conduction band minimum, respectively, as shown in Figure 4.10.

Figure 4.10 Arrhenius plots of Peaks #1 - #4 corresponding to the DLTS spectra shown in Figure 4.9.
Peak #1 was identified as titanium substitutional impurity [51]-[53], [56], [65]. The Peak #2 has been assigned as $Z_{1/2}$ defect which is related to carbon vacancies or their complexes by several groups [55], [63]-[67]. The levels corresponding to Peaks #3 and #4 were assigned as EH$_6$ and EH$_7$, respectively [55], [66], [75]-[77].

DLTS scans were performed for the edge terminated detectors to compare the deep levels with that of the 2AS1a non-edge terminated detector. DLTS spectra of the detector with SiO$_2$ edge termination (2AS22) is shown in Figure 4.11.

![DLTS spectra of 2AS22](image)

Figure 4.11. DLTS scans of 2AS22, the device with SiO$_2$ edge termination; negative peaks corresponding to electron traps in the detector: (left) larger correlator delays were used to observe the deep defect levels up to 700K; (right) shorter correlator delays were used with a lower temperature scan set to fully observe peak 1.

DLTS scans indicate presence of only two defect levels - Peak #1 and Peak #2. Corresponding Arrhenius plot of this DLTS scan, obtained using different rate windows, is shown in Figure 4.12. From the Arrhenius plot, activation energy for Peak #1 was found to be $E_c - 0.202$ eV, which is a shallow level defect and related to titanium substitutional impurity. The activation energy of Peak #2 was found to be $E_c - 0.64$ eV, which is identified as electrically active $Z_{1/2}$ defect related to carbon vacancies or their complexes. Notably, the defect levels assigned as EH$_6$ at $E_c - 1.40$ eV and EH$_7$ at $E_c -$
1.45 eV, which were observed for the device without edge termination, were absent for the detector with SiO$_2$ edge termination.

Figure 4.12 Arrhenius plots of Peaks #1 - #2 corresponding to the DLTS spectra shown in Figure 4.11.

The DLTS scans performed on detector with Si$_3$N$_4$ edge termination (2AS30) are shown in Figure 4.13. Three peaks were present, and as before, an additional scan with shorter correlator delays was required to fully observe Peak #1. The activation energies, obtained from the resulting Arrhenius plot shown in Figure 4.14, indicate that the defect centers related to these three peaks were located at $E_c - 0.22$ for Peak #1, $E_c - 0.63$ for Peak #2, and $E_c - 1.25$ for Peak #3. As discussed earlier, Peak #1 at $E_c - 0.22$ was assigned as the defects corresponds to titanium substitutional impurity. The defect level at $E_c - 0.63$ relates to electrically active $Z_{1/2}$ defect originated from carbon vacancies. Similar to detector with SiO$_2$ edge termination, the defect levels assigned as EH$_6$ at $E_c - 1.40$ eV and EH$_7$ at $E_c - 1.45$ eV were absent. However, a new defect level at $E_c - 1.25$ eV (Peak #3) was observed that was not present in the detector without edge termination. A similar trap level at $E_c - 1.31$ eV was reported before [25], [61], [78] and was identified as Cil in a chlorine implanted n-type 4H-SiC epitaxial layer [61].
Figure 4.13. DLTS scans of 2AS30, the device with Si$_3$N$_4$ edge termination: (left) larger correlator delays were used to observe the deep defect levels up to 700K; (right) a lower temperature scan set with the shorter correlator delays to fully observed peak 1.

Figure 4.14. Arrhenius plots of Peaks #1 - #3 from DLTS spectra in Figure 4.13

A summary of the DLTS defects parameters extracted from the Arrhenius plots of the three Ni/4H-SiC detectors (2AS1a with no edge termination, 2AS22 with SiO$_2$ edge termination layer, and 2AS30 with Si$_3$N$_4$ edge termination layer) are presented in Table 4.2. Result shows that the defect trap concentrations ($N_t$) and capture cross sections ($\sigma_n$) of the $Z_{1/2}$ levels were lower for the detectors with edge termination compared to the detector without edge termination. In previous study, the defect level $Z_{1/2}$ was found to be the most performance limiting as it reduces carrier lifetime [25], [52], [60], [79]-[80]. Other performance limiting deep-level defects EH$_6$ and EH$_7$, which represents carbon-
carbon or carbon-silicon di-vacancies or related complex, were also absent in the detector samples with edge termination.

Table 4.2 Defects parameters obtained from DLTS scans of the detectors with and without edge termination layer.

<table>
<thead>
<tr>
<th>Detector without edge termination layer (2AS1a)</th>
<th>Detector with SiO2 edge termination layer (2AS22)</th>
<th>Detector with Si3N4 edge termination layer (2AS30)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak #</td>
<td>$\sigma_n$ cm$^{-2}$</td>
<td>$\Delta E$ eV</td>
</tr>
<tr>
<td>Peak 1</td>
<td>$7.06 \times 10^{-14}$</td>
<td>$E_c - 0.19$</td>
</tr>
<tr>
<td>Peak 2</td>
<td>$5.04 \times 10^{-16}$</td>
<td>$E_c - 0.62$</td>
</tr>
<tr>
<td>Peak 3</td>
<td>$5.52 \times 10^{-15}$</td>
<td>$E_c - 1.40$</td>
</tr>
<tr>
<td>Peak 4</td>
<td>$1.73 \times 10^{-16}$</td>
<td>$E_c - 1.45$</td>
</tr>
</tbody>
</table>

Hiyoshi and Kimoto have shown that by thermal oxidation of 4H-SiC epilayers, the $Z_{1/2}$ and EH$_{6/7}$ centers can be significantly reduced and have proposed that carbon and/or silicon atoms from the oxide-semiconductor interface diffuse into the epilayer and these interstitials recombine with the carbon vacancies \[80\]. In our work, SiO$_2$ and Si$_3$N$_4$ edge termination was carried out at a process temperature of 300 °C much lower than the thermal oxidation temperatures (1150 °C – 1300 °C) used by Hiyoshi and Kimoto \[81\] which could explain $Z_{1/2}$ levels decreasing less in comparison to their reported trap
concentration reduction to below their detection limit of $1 \times 10^{11}$ cm$^{-3}$. Kawahara et al. showed that although $Z_{1/2}$ levels were decreased by thermal oxidation, other deep levels were generated labeled ON1 and ON2 in their work at temperature from 350 K to 550 K. [82]. Theses peaks were absent in the DLTS scan of SiO$_2$ edge terminated sample 2AS22.

4.6 RADIATION DETECTION PERFORMANCE EVALUATION

A standard analog spectrometer was used along with an Amptek A250CF preamplifier and a standard broad window 0.1 µCi $^{241}$Am source to evaluate the radiation detection performance of the detectors. The detector was mounted in an EMI shielded aluminum sealed box with the alpha source mounted 15 mm above the detector where alpha particles would be incident normal to the Ni window of the detector. A mechanical pump was attached to the aluminum box and continually evacuated during measurements to reduce alpha particle scattering from air molecules. A charged sensitive Amptek A250CF preamplifier was used to collect the detector signals. The incoming signals were shaped by an Ortec 671 Spectroscopy Amplifier. The amplified and shaped signals were collected using a Canberra Multiport II ADC-MCA unit to obtain the pulse-height spectra. The energy resolution was expressed in terms of full width at half maximum (FWHM) and percentage resolution for the relevant peaks of the spectra given by

$$\text{% resolution} = \frac{\text{FWHM}}{\text{centroid}} \times 100$$  \hspace{1cm} 4.3

where FWHM is the full width at half maximum of the detected peak from the pulse-height spectra and centroid is the “center of mass” of the peak energy in keV. The centroid of the observed peak was slightly less than the characteristic main energy peak of 5486 keV. Since a broad alpha source was used, a portion of the charged particles
incident on the Ni window will scatter depending on angle of incidence and thickness of the Ni window. A further improvement of the detector performance is expected if the source is collimated and metal contact thickness and material selection is optimized.

A pulse-height spectrum shown in Figure 4.15 was collected from the fabricated Schottky barrier detector 2AS1a (without edge termination) at an applied bias of 120 V and a shaping time of 6 μs at 300K.

![Pulse height spectrum](image)

Figure 4.15. Pulse height spectrum obtained from detector 2AS1a using a $^{241}$Am alpha source. Solid lines show the de-convoluted alpha peaks which were partially resolved.

The collected spectrum was converted from bins to energy by adopting the absolute calibration approach described in the previous work by our research group [6]. The energy resolution was found to be ~0.5% for 5486 keV alpha particles incident on the detector through the 10 nm thick Ni window. The centroid (center of mass) of the observed peak was slightly less than the characteristic main energy peak of 5486 keV. Since a broad alpha source was used, some alpha particles incident on the epilayer surface at an angle may reduce the amount of kinetic energy imparted to the active region
of the detector. Furthermore, electrically active defects in the detector cause incomplete charge collection. Centroid energy is affected in both cases. A further improvement of the detector performance is expected if the source is collimated and metal contact thickness and material selection is optimized.

Alpha spectroscopy measurements were also performed at 300K on detectors 2AS22 (with SiO$_2$ edge termination) and 2AS30 (with Si$_3$N$_4$ edge termination) with the pulse-height spectra from before and after edge termination as shown in Figure 4.16 and Figure 4.17, respectively. The main pulse-height spectra shown in this figure is the spectrum for the detector with edge termination, and the spectra shown in inset is of the detector before edge termination.

Figure 4.16. Pulse-height spectra using $^{241}$Am source at a bias voltage of -120 V on 2AS22 with SiO$_2$ passivation. Inset: Pulse-height spectra of the same detector before passivation.
The pulse-height spectra for detector without edge termination (shown in inset) are broader with no clear indication of the three main energy peaks. In contrast, a clear improvement in energy resolution was observed noting the three main energy peaks of $^{241}$Am were all visible and highly resolved after SiO$_2$ and Si$_3$N$_4$ edge termination. The percentage resolutions calculated for the detectors 2AS22 and 2AS30 at a reverse bias of 120 V were 0.72% and 0.55% before edge termination and 0.39% and 0.38% after edge termination for SiO$_2$ and Si$_3$N$_4$, respectively.

The initial difference in energy resolution between two detectors without edge termination can be explained by a variation in the number of electrically active defects from sample to sample which can be observed in DLTS scans. Following edge termination, the differences in energy resolutions between the two samples are minimal,
which suggests a decrease in the quantity or involvement of those electrically active defects.

Edge termination has played multiple roles in improving detector performance. By depositing relatively thick layers of SiO$_2$ and Si$_3$N$_4$ surrounding the detector window, these layers act to collimate alpha particles incident on the detector window and block particles incident outside of the window for a broad alpha source. This leads to reduced broadening of the spectra by improved charge collection. Reduction in the involvement of electrically active defects also leads to more complete charge collection due to edge termination.

4.7 CONCLUSION

Edge termination of Ni/n-type 4H-SiC epilayer detector exhibited a significant improvement in electrical characteristics and detector performance in terms of energy resolution. Alpha spectroscopy measurements revealed an improved energy resolution from ~0.7% to ~0.4% for 5.48 MeV alpha particles after edge termination with SiO$_2$ and Si$_3$N$_4$. Edge termination with thick SiO$_2$ and Si$_3$N$_4$ layers has multiple positive effects toward improving detector performance.

Edge termination improved Schottky barrier properties of the detector. Following edge termination, reverse leakage current was decreased by two orders of magnitude, leading to increased signal-to-noise ratio. Forward I-V characteristics showed high surface barrier height of ~1.7 eV and diode ideality factor of 1.07, very close to ‘unity’, which is ideal for Schottky junction. The C-V measurements showed doping concentration of 2.4x10$^{14}$ cm$^{-3}$ which ensured a fully depleted (~20 μm) detector at bias voltage as low as ~70 V.
The edge terminating layers serve to collimate alpha particles incident on the detector window. This allows energy of more alpha particles to be deposited in the active region of the device leading to better charge collection while also reducing the broadening of the spectra thereby enhanced energy resolution.

Reduction in electrically active defects due to edge termination, as observed by DLTS, also leads to more complete charge collection. After edge termination, a reduction was observed in $Z_{1/2}$ defect level densities, which plays a significant role in the detector performance. No detectable presence of performance limiting electrically active EH$_{6/7}$ deep-levels were also attributed to higher charge collection thereby improved detector resolution.
CHAPTER 5: RU/4H-SIC/NI DETECTOR FOR HARSH ENVIRONMENTS

5.1 OVERVIEW

The availability of high quality epitaxial layers have led to a renewed interest in silicon carbide due to its superior material properties making it attractive for use in high temperature and in high radiation background where conventional semiconductor detectors would fail to operate or suffer reduced performance. The properties of ruthenium (Ru) metal possess abrasion and fatigue resistance, high melting point (2334 °C), and chemical stability, making it an ideal material to form electrical contacts for rugged and compact 4H-SiC devices operating in harsh environments where high temperature and high radiation fluence would significantly degrade other devices (e.g. Si, Ge, CZT, and scintillator based detectors). In this study, we explore the use of Ru as a Schottky contact on n-type 4H-SiC epilayers detector with nickel as the back Ohmic contact. The junction properties were characterized through current-voltage (I-V) and capacitance-voltage (C-V) measurements. Detectors were characterized by alpha spectroscopy measurements in terms of energy resolution and charge collection efficiency using a 0.1 μCi $^{241}$Am radiation source. Defect characterization of the epitaxial layers was conducted by deep level transient spectroscopy (DLTS) to thoroughly investigate the defect levels in the active region induced from radio frequency (RF)
sputtering and rapid thermal annealing of the Ru Schottky contacts in the 4H-SiC epilayers.

5.2 DETECTOR FABRICATION WITH RUTHENIUM SCHOTTKY CONTACT

Device operation in high performance applications requires the formation of metal contacts with excellent thermodynamic stability and high Schottky barriers for good current rectification. The challenge in producing consistently good Schottky contacts depends on the surface preparation of the semiconductor and how easily the contact metal diffuses into the semiconductor at the temperatures in which the device will operate. For this purpose, Ru has been explored in this study for Schottky barrier radiation detectors on the 4H-SiC epilayer. Ru is a high work function (υ_{Ru} = 4.71 eV) rare earth transition metal with excellent physical, chemical, and electrical properties [83]. A schematic of the cross-section of the Ru/4H-SiC/Ni planar detector structure is shown in Figure 5.1.

![Schematic diagram of Ru/4H-SiC/Ni planar detector structure](image)

Figure 5.1 Cross-sectional view of a fabricated Ru/4H-SiC n-type epitaxial Schottky barrier detectors.

In this study, 10×10 mm² samples diced from n-type 4H-SiC 50 μm thick epitaxial layer grown on 4H-SiC (0001) substrate wafer highly doped with nitrogen and
$8^\circ$ off-cut towards the [11$ar{2}$0] direction were used for the fabrication of radiation detectors. The epilayers were grown by a hot wall chemical vapor deposition (CVD) process using dichlorosilane (SiH$_2$Cl$_2$) and propane (C$_3$H$_8$) as the precursors and hydrogen as the carrier gas [78]. The epilayer surface was prepared using a modified Radio Corporation of America (RCA) cleaning process [80]. The cleaning process includes organic solvents (TCE, Acetone, and Methanol), solution #1 ($\text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{NH}_4\text{OH}$ with ratio 5:1:1), piranha solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ with ratio 2.5:1), and the final dip of the substrates into diluted HF followed by DI water rinse prior to metal contact deposition.

A photolithography process in a class 1000 clean room was carried out on the substrates (10 x 10 mm$^2$) to center align the detector windows on the epilayer with respect to the edges. A Karl Suss MA6 mask aligner was used with a pre-designed quartz photo-mask with 3.9 mm diameter circular patch for alignment and exposure of the photoresist coated samples. After UV exposure, the substrates were submersed in a proper developer followed by Ru and Ni deposition for the top and bottom contact. Ru was deposited on the epilayer surface by radio frequency (RF) sputtering using a metal shadow mask (115 μm thick) to form a 3.8 mm diameter circular Schottky contact with a thickness of $\sim$12 nm. Following Ru metal deposition, rapid thermal annealing (RTA) was carried out at 950°C for 5 minutes in argon (Ar). 12 nm Ru contacts were used to minimize energy loss from Coulomb interaction between the alpha particle and the detector window [6]. A depletion width of 20 μm was obtained at a reverse bias as low as 75 V which is a sufficient depth for stopping most alpha particles resulting from n,α nuclear reactions. Ni bottom contact ($\sim$100 nm thick) was deposited by DC sputtering.
5.3 **Electrical Characterization of Ru/4H-SiC Schottky Detector**

I-V measurements were carried out at room temperature (300 K) under dark using a Keithley 237 High Voltage Source Measure Unit. Detectors were loaded into an aluminum EMI shielded box during measurements. As shown in Figure 5.2, the fabricated Ru/4H-SiC Schottky barrier diode exhibits excellent current rectification with less than 0.57 pA at an applied bias voltage as high as −180 V. Using a thermionic emission model, the barrier height of 1.70 eV with a diode ideality factor of 1.2 were determined from the forward I-V measurement. Series resistances due to the presence of an oxide layer at the metal-semiconductor interface or inhomogeneity in the Schottky barrier height due to surface states causes deviations from an ideality factor of 1.

![Figure 5.2 I-V characteristics obtained at room temperature for fabricated Ru/4H-SiC n-type epilayer Schottky barrier radiation detector](image)

High frequency (1MHz) C-V measurements were performed on the detectors inside an EMI shielded box at 300 K under dark. The effective doping concentrations of the detectors were determined from the measured Mott-Schottky plots presented in
Figure 5.3 and calculated to be of $4.34 \times 10^{14} \text{ cm}^{-3}$. The calculated barrier height from C-V measurements was determined to be 1.81 eV which is slightly higher than that calculated from I-V measurements and is normally observed to deviate higher [78]. This can be attributed to the presence of a thin oxide layer at the metal-semiconductor interface. Inhomogeneity of the Schottky barriers also results in the current flow through lower barrier height areas across the metal contact area which dominates the barrier height obtained from the forward I-V characteristics [44], [78], [85]. It should also be noted that the barrier height obtained from C-V measurements is an average value over the entire diode area.

![Figure 5.3 Mott-Schottky plot (1/C^2 vs. V) for Ru/4H-SiC epilayer Schottky barrier diode measured under dark at 300 K. Inset shows C-V characteristics.](image)

5.4 **Electronic Noise Measurements and Alpha Spectroscopy for Detector Evaluation**

Electronic noise measurements were carried out by using a calibrated capacitor installed in a BNC connector feed-through between the precision pulser output and the
preamplifier input and pulse-height spectra were obtained [6]. The width of the pulser peak expressed in terms of Coulomb rms was used as the equivalent noise charge (ENC). ENC measurements with the detector connected were carried out by feeding the pulser output through the pre-amplifier test input. Figure 5.4 (a) shows the variation of ENC and other noise contributions as a function of shaping time without the detector connected; the minimum attainable noise in the system is at a shaping time of 3 µs. After the detector is connected to the system and reverse biased at 90 V, the minimum ENC is higher and found to be between a shaping time of 6 µs and 10 µs as shown in Figure 5.4 (b). An increase in both the white series and pink noise were expected following connection of the detector due to an increase in device capacitance, however, the pink noise increased while the white series noise decreased. This would lend itself to a decrease in the thermal noise of the FET channel. White parallel noise was observed to have increased following connection of the detector due to the additional current from the detector.

Alpha spectroscopy was performed on the fabricated detectors irradiated with a 0.1 μCi ²⁴¹Am broad window alpha source using a standard spectrometer at 300 K. The detector sample and source were both loaded into an EMI shielded box and sealed. A rotary pump was attached and the measurement chamber was constantly evacuated during measurements to minimize the scattering of alpha particles due to air molecules. A CoolFET A250CF charge sensitive pre-amplifier was used to collect the detector signals. An Ortec 671 spectroscopy amplifier was used to shape the incoming charge pulses. The amplified and shaped signals were collected using a Canberra Multiport II ADC-MCA unit to obtain the pulse-height spectra. The energy resolution was expressed in terms of full width at half maximum (FWHM) and percentage resolution for the relevant peaks.
Figure 5.4 Variation of equivalent noise charge as a function of shaping time: (a) with the pulser peak fed to the pre-amp using a calibrated capacitor and (b) with a Ru/4H-SiC detector connected to the pre-amplifier; detector was biased to -90 V and exposed to the $^{241}$Am source during the measurements. Contributions from white series noise, white parallel noise, and pink noise are shown.

The penetration depth of $^{241}$Am alpha particles in 4H-SiC is about 18 µm. Although 20 µm is sufficient depth for stopping alpha particles of that energy, a bias of approximately -450 V would be needed to fully deplete the 50 µm epilayer. It was determined from observing the electronic noise during alpha pulse-height measurements that a reverse bias of -450 V would not be possible without reaching breakdown for this detector.
The pulse-height spectra were collected with an $^{241}$Am source. As observed in Figure 5.5, the resulting peak is broad, and the three characteristic major alpha particle energies, viz. 5.388 MeV, 5.443 MeV, and 5.486 MeV from the $^{241}$Am source could not be precisely resolved. Furthermore since the 50 µm epilayer could not be fully depleted, incomplete charge collection results. At a 180 V reverse bias, roughly 36% of the epilayer remains un-depleted. As Coulomb interaction occurs the generated charge carriers are swept out under the influence of the electric field (drift), however, the electrons must then traverse the remaining un-depleted section of the epilayer by diffusion in which case a higher chance of recombination occurs leading to incomplete charge collection thereby shifting the peak centroid to lower energies and overall broadening of the peak as seen in Figure 5.5. The energy resolution was calculated to be ~0.75% at a 180 V reverse bias.

![Pulse-height spectra collected at a bias of -180 V obtained from the Ru/4H-SiC Schottky barrier radiation detector fabricated on 50 µm epilayers.](image)

Figure 5.5 Pulse-height spectra collected at a bias of -180 V obtained from the Ru/4H-SiC Schottky barrier radiation detector fabricated on 50 µm epilayers.
5.5 **Deep Level Transient Spectroscopy (DLTS)**

A DLTS study was conducted to investigate deep level defects within the active region of the detector. DLTS measurements were performed using a SULA DDS-12 DLTS system as explained in details earlier. The system contained a 1 MHz oscillator used for capacitance measurements and built-in preamplifier module. The sample was mounted in a Janis VPF-800 LN$_2$ cryostat for low to high temperature measurements (80 K to 800 K) under dark controlled by a Lakeshore LS335 temperature controller. A steady-state reverse bias of −12 V was applied to the Schottky diode under test, and the equilibrium carrier concentration (in this case electrons) was perturbed by a trap filling pulse to 0 V with a pulse width of 1 ms over a 1 s period. The steady-state reverse bias is reapplied at the termination of the trap filling pulse and relaxation to equilibrium is achieved through thermally emitting trapped charges resulting in capacitance transients.

Representative DLTS spectra (1 of 4 correlator rate windows shown) are presented in Figure 5.6 and Figure 5.7. The significance of using the largest and smallest correlator initial delays enable areas of interest in the DLTS spectra to be observed which may have been partially outside of a given rate window as was the case for peak 2 in Figure 5.7. The DLTS measurements are consistent with our previous work on 50 μm n-type 4H-SiC epilayers [26], [78], with the exceptions of peaks 4 and 7 which involve new findings. In total, seven distinct peaks were observed in this study which corresponds to particular defect levels. The defect parameters were calculated from Arrhenius plots shown in Figure 5.8 which include their activation energies, capture cross-sections, and trap concentrations, and are summarized in Table 5.1.
Figure 5.6 DLTS scan with smallest correlator initial delays (1 ms) of Ru/4H-SiC epilayer Schottky diode from 85 K to 800 K.

Figure 5.7 DLTS scan with largest correlator initial delays (100 ms) of Ru/4H-SiC epilayer Schottky diode from 85 K to 800 K.
Figure 5.8 Arrhenius plots of emission rate $T^2/e_n$ vs. $10^3/T$ for DLTS scans with (a) smallest correlator initial delays shown in Figure 5.6, and (b) largest correlator initial delays shown in Figure 5.7.

The defect levels corresponding to peak 1 and peak 2 were located at $E_C - 0.12$ eV and $E_C - 0.17$ eV, respectively. Both of these peaks have been identified as titanium (Ti) substitutional impurities. Dalibor et al. have observed similar defect levels in Ti$^+$ implanted 4H-SiC and attributed them to Ti$^{3+}$ residing at hexagonal and cubic Si lattice sites [65]. The presence of Ti impurity is likely introduced during the growth processes from the Ti parts in the growth reactors. The activation energy corresponding to peak 3 was found to be located at 0.65 eV below the conduction band edge. This defect level, $Z_{1/2}$, is usually observed in DLTS studies on n-type 4H-SiC [26], [53], [78] and carbon related vacancies is suggested as the origin [68].
Table 5.1 Summary of defect parameters obtained from DLTS measurements of Ru/4H-SiC/Ni detector structure.

<table>
<thead>
<tr>
<th>Peak #</th>
<th>$\sigma_n$ cm$^2$</th>
<th>$\Delta E$ eV</th>
<th>$N_t$ cm$^{-3}$</th>
<th>Possible trap identity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak 1</td>
<td>$7.18 \times 10^{-16}$</td>
<td>$E_c - 0.12$</td>
<td>$3.66 \times 10^{10}$</td>
<td>Ti(h)</td>
</tr>
<tr>
<td>Peak 2</td>
<td>$4.39 \times 10^{-15}$</td>
<td>$E_c - 0.17$</td>
<td>$1.17 \times 10^{11}$</td>
<td>Ti(c)</td>
</tr>
<tr>
<td>Peak 3</td>
<td>$1.58 \times 10^{-15}$</td>
<td>$E_c - 0.65$</td>
<td>$6.36 \times 10^{10}$</td>
<td>$Z_{1/2}$</td>
</tr>
<tr>
<td>Peak 4</td>
<td>$1.69 \times 10^{-16}$</td>
<td>$E_c - 0.89$</td>
<td>$8.41 \times 10^{10}$</td>
<td>Ru-related</td>
</tr>
<tr>
<td>Peak 5</td>
<td>$6.10 \times 10^{-13}$</td>
<td>$E_c - 1.26$</td>
<td>$7.98 \times 10^{10}$</td>
<td>Ci1</td>
</tr>
<tr>
<td>Peak 6</td>
<td>$2.35 \times 10^{-15}$</td>
<td>$E_c - 1.53$</td>
<td>$3.87 \times 10^{10}$</td>
<td>EH$_{6/7}$</td>
</tr>
<tr>
<td>Peak 7</td>
<td>$3.10 \times 10^{-14}$</td>
<td>$E_c - 1.98$</td>
<td>$2.31 \times 10^{10}$</td>
<td>Unidentified</td>
</tr>
</tbody>
</table>

Peak 4 corresponds to a defect level located at $E_C - 0.89$ eV which is similar to activation energies reported for the RD$_{1/2}$ defect level observed in irradiation studies by Castaldini et al [86]. Stuchlikova et al. has observed a similar defect level in RuO$_2$/4H-SiC Schottky diodes and suggested the emergence of this defect level may be related to defects induced by Ru impurities in the interfacial region of the epilayer [87]. Munthali et al. conducted a study on the solid-state reaction of Ru with SiC and observed an intermixed layer of Ru and Si at the interface following annealing at 600°C and formation of Ru$_2$Si$_3$ and graphite observed at 700°C from their Rutherford Backscattering Spectrometry (RBS) data [88]. The defect level $E_C - 0.89$ eV observed in this study is similar with that of a study conducted by Stuchlikova et al. [87]. Further Ru/4H-SiC Schottky diode was fabricated by annealing at the solid-state reaction temperature of Ru with SiC observed by Munthali et al. [88]. Hence, there is strong indication that this defect level is related to this solid-state reaction between Ru and Si at the interface and
resulting carbon release. The activation energy of the defect level corresponding to peak 5 was determined to be $E_C - 1.26 \text{ eV}$. Alfieri et al. has observed a similar defect level for chlorine implanted n-type 4H-SiC [61]. The defect level located at $E_C - 1.53 \text{ eV}$ has been attributed to carbon vacancies and is believed to be another charge state of $Z_{1/2}$ and has been labeled as EH$_{6/7}$ [89], [90]. The defect level with activation energy $E_C - 1.98 \text{ eV}$ corresponding to peak 7 remains unidentified and no similar defect level has been reported in the literature to the best of our knowledge.

**5.6 CONCLUSION**

High Schottky barrier (barrier height of 1.703 eV) Ru/4H-SiC 50 µm epilayer detectors were fabricated to produce large active volume alpha particle detectors, however, electrical and alpha pulse-height characterization results revealed a detector performance dependency on various electrically active defects in the active region of the device. An energy resolution of 0.75% was achieved but was limited by leakage current from the defects preventing higher bias levels to fully deplete the epilayer. Incomplete charge collection occurred due to charge carriers traversing the medium by diffusion which is dependent upon the gradient of the carrier concentration rather than under the influence of an electric field as in the case of drift leading to a higher probability of recombination.

Inhomogeneity of the Schottky barrier heights observed may be due to interfacial defects resulting from a solid-state reaction involving Ru, Si, and C. The defect levels of the detectors were investigated by DLTS and seven defect centers were observed in a temperature range of 80 K to 750 K. Comparing with activation energies reported in literature, five of those peaks with activation energies $E_C - 0.12 \text{ eV}$ (Ti(h)), $E_C - 0.17 \text{ eV}$
(Ti(c)), $E_C - 0.65$ eV ($Z_{1/2}$), $E_C - 1.26$ eV (Ci1), and $E_C - 1.53$ eV (EH$_{6/7}$), were identified. The microscopic structure of the defect level which was indicated to be induced by Ru with activation energy $E_C - 0.89$ eV is unknown though it shows similar activation energy with the radiation induced defect $RD_{1/2}$ which involves a carbon and silicon vacancy complex $V_C + V_{Si}$. The origin of the defect level with activation energy of $E_C - 1.98$ eV is not known at this time and further investigations are necessary and in progress in the group.
CHAPTER 6: CONCLUSION, DISSEMINATION OF WORK, AND SUGGESTIONS FOR FUTURE WORK

6.1 CONCLUSION

Radiation detectors are important tools for accounting of radioactive materials and have widespread applications in national security, in nuclear power plants, nuclear waste management, in medical imaging such as x-ray, mammography, CT scan, and in high energy astronomy for NASA space exploration. The currently available detection systems are limited by their detection efficiency, stability of response, speed of operation, and physical size due to requirement of cryogenic cooling. To address this, in this dissertation n-type 4H-SiC epitaxial layer was explored for “all solid-state”, “direct read-out” radiation detector that can operate at room temperature and above. Silicon Carbide (SiC) is an indirect wide bandgap semiconductor (3.27 eV at 300 K) with high thermal conductivity allowing detector operation well above room temperature (~773 K) as required for nuclear fuel processing environment in nuclear power plants. SiC allows very low leakage currents (low noise) at high operating bias and has high displacement energies of the constituent elements, making it available for detectors that are deployed in harsh environments such as high radiation field found in nuclear power plants and in upper atmosphere and/or outer space.

Schottky barrier radiation detectors were fabricated on nitrogen doped n-type 4H-SiC epitaxial layers (on silicon face of crystal lattice). The epitaxial layers were grown by a hot wall chemical vapor deposition (CVD) process on n-type 4H-SiC (0001) substrates.
with 4-8° off-cut towards the [11\bar{2}0] direction. The junction properties of the fabricated detectors were characterized by current-voltage (I-V) and capacitance-voltage (C-V) measurements using thermionic emission model. Deep level transient spectroscopy (DLTS) was used to investigate electrically active defects in the epitaxial layer. Defect parameters such as activation energy, capture cross-section, and density were calculated from the Arrhenius plots obtained from the DLTS spectra. Detector performances were evaluated in terms of the energy resolution at full-width-half-maximum (FWHM) using alpha pulse-height spectroscopy measurements with a 0.1 µCi \(^{241}\)Am source.

Ni/4H-SiC Schottky barrier detectors on 12 µm, 20 µm, and 50 µm thick n-type 4H-SiC epitaxial layers were investigated. This work was the first reported study when a 12 µm n type 4H-SiC epilayer was investigated for radiation detectors. For 12 µm Ni/4H-SiC epilayer detector, reverse I-V measurements revealed a very low leakage current of 0.18 nA at 250 V bias, representing low noise thereby better detector performance. However, the barrier height of 1.10 eV and an ideality factor of 1.29 were determined by forward I-V measurement, indicating inhomogeneity at the metal-semiconductor interface causing non-uniform Schottky barrier height. From C-V measurements, effective carrier concentration was calculated to be \(1.03 \times 10^{15} \text{ cm}^{-3}\). DLTS analysis revealed presences of shallow level defect related to titanium impurities, electrically active lifetime killer \(Z_{1/2}\) defect level, and deep level defects assigned as \(EH_{6/7}\) which are related to carbon or carbon-silicon divacancies. Among these defect levels \(Z_{1/2}\) is the most detrimental to detector performance. The concentration and capture cross-section of \(Z_{1/2}\) defect level were determined to be \(1.6 \times 10^{12} \text{ cm}^{-3}\) and \(9.12 \times 10^{-16} \text{ cm}^{2}\), respectively. The trap density was orders in magnitude lower compared to other 4H-SiC
detectors. Detectors on 20 μm thick epilayer were used for front-end readout electronics noise analysis and assessment of edge terminating passivation layer employed to improve detector resolution.

New edge termination has been developed using surface passivating layers of silicon dioxide (SiO$_2$) and silicon nitride (Si$_3$N$_4$). Ni/4H-SiC epitaxial Schottky barrier detector with edge termination exhibited a significant improvement in electrical and defect characteristics, thereby detector performance. The edge terminating layers served to collimate alpha particles incident on the detector window which allowed energy of more alpha particles to be deposited in the active region of the device. High surface barrier height (1.7 eV) and Schottky diode ideality factor of 1.07 which is very close to ideal value of ‘unity’ indicating better surface uniformity that enhances charge collection efficiency. Following edge termination, reverse leakage current was decreased by two orders of magnitude, leading to increased signal-to-noise ratio. The C-V measurements showed a doping concentration of 2.4 x10$^{14}$ cm$^{-3}$ which ensured a fully depleted (~20 μm) detector at bias voltage as low as ~70 V. DLTS analysis showed a reduction in carrier-lifetime reducing Z$_{1/2}$ defect level densities in detectors with edge termination. Also, performance-limiting electrically active EH$_{6/7}$ deep-levels were not detectable in edge terminated detectors. Reduction in electrically active defects also contributed to more complete charge collection thereby improved detector resolution. Edge termination thus had multiple positive effects toward improving detector energy resolution. Alpha spectroscopy measurements revealed an improved energy resolution from ~0.7% to ~0.4% for 5.48 MeV alpha particles after edge termination with SiO$_2$ and Si$_3$N$_4$. 

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4H-SiC detector with ruthenium (Ru) Schottky barrier was developed for operation in harsh environment. Ruthenium with abrasion and fatigue resistance, high melting point (2334 °C), and chemical stability is a better choice for metal contact for such environment. Ru/4H-SiC detectors were fabricated on 50 μm n-type 4H-SiC epitaxial layers grown on 360 μm SiC substrates by depositing ~12 nm thick Ru contact by RF sputtering, followed by annealing. Room temperature I-V measurement revealed a very low leakage current of ~ 0.57 pA at 180 V reverse bias. The barrier height for Ru/4H-SiC Schottky contact was found to be of ~1.7 eV with a diode ideality factor of 1.2. Inhomogeneity in the Schottky barrier height due to surface states causes deviations from an ideality factor of 1. From front-end electronics noise analysis, the minimum equivalent noise charge was found to be between a shaping time of 6 μs and 10 μs when detector is connected to the system. To completely deplete 50 μm epilayer, a bias of approximately -450 V would be needed, which was not possible without reaching breakdown for this detector. Thus energy resolution for alpha particle detection using a $^{241}$Am alpha source was obtained at 180 V reverse bias, and an energy resolution was ~0.75% obtained. At a 180 V reverse bias, roughly 36% of the epilayer remains undepleted. Capacitance mode deep level transient spectroscopy (DLTS) revealed the presence of seven defect levels. Five of them were identified and related to titanium impurities (Ti(h) and Ti(c)), carbon related vacancies $Z_{1/2}$, chlorine implantation related defect Cil, and carbon related vacancy $EH_{6/7}$ believed to be another charge state of $Z_{1/2}$. DLTS revealed two new defect levels. Defect data reported in the literature suggests the new defect level of $E_C - 0.89$ eV may be correlated to Ru-induced defect formation in 4H-SiC epitaxial layer. The defect level observed at $E_C - 1.98$ eV has been observed for
the first time in 50 μm 4H-SiC epilayer following Ru Schottky barrier formation and subsequent rapid thermal annealing (RTA) and this deep level has not been reported.

6.2 DISSEMINATION OF WORK


6.3 **SUGGESTIONS FOR FUTURE WORK**

- We have studied Ni and Ru for Schottky barrier contact structures. Different contact structures with other metals with varying work function could be studied
to reduce the leakage current of detector by controlling carrier transport inside the devices for better detection signals and energy resolution.

- In SiC, the electron mobility is much higher than hole mobility. In order to compensate for poor hole transport properties, specialized detector geometries, such as co-planar, Frisch grid, or multiple small pixel detector structure, can be applied and performance evaluation could be compared to the planar detector studied in this dissertation.

- A future research could be undertaken to focus on lowering detector capacitance without reducing the detector active area by increasing the detector active thickness, i.e. by using a thicker epitaxial layer. An electronic noise analysis of the detection system will reveal the possibility of achieving better energy resolution by lowering the detector capacitance. This will reveal the white series noise due to the total input capacitance which may have substantial effects on detector performance.

- The nature and type of crystallographic defects could also be investigated through defect delineating KOH etching and the results will be analyzed and correlated to observe the impact of deep lying point and/or extended defects in the active region, i.e., in the width of the depletion region.
REFERENCES


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