Silicon and silicon carbide radiation detectors for alpha and neutron detection at elevated temperatures

by

Yusuf Musa Abubakar

under the supervision of

Dr Annika Lohstroh

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Department of Physics
Faculty of Engineering and Physical Sciences
University of Surrey

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Dedicated to Zahra, Husna and Hamida
Abstract

Harsh radiation environments are characterised by high temperature, high radiation fluence, high pressure and sometimes vibration. These conditions are present in nuclear reactor cores and oil and gas prospecting. Because of the limited supply of $^3$He, new materials are needed for neutron porosity measurements in order to reduce the cost of nuclear well logging and reduce dependence on $^3$He.

High radiation fluence affects material’s performance for radiation detection applications. For decades silicon has been an important material for radiation monitoring because of its cost effectiveness and relative availability. In recent years, the level of radiation produced in high energy physics (HEP) experiments has drastically increased, which compels searching for special materials that can withstand high radiation fluence. On the other hand, silicon carbide (SiC) devices have also been receiving considerable attention because of their properties that makes them excellent candidates for harsh radiation media applications. The main aim of this work was to investigate the thermal neutron detection ability of SiC diodes covered with standard LiF neutron converter layers at elevated temperatures up to 500 K for extended periods of time - up to 100 hours. For this purpose, a range of 4H- silicon carbide(SiC) devices, based on semi-insulating bulk material and epitaxial diodes has been characterised in terms of current voltage, capacitance voltage and alpha spectroscopy performance as a function of temperature to underpin the neutron tests with an understanding of changes in Schottky Barrier Height, conductivity and effective doping with temperature before proceeding to neutron tests. In parallel, a series of Silicon (Si) Schottky diodes was also studied with various levels of proton induced radiation damage as part of an international collaboration. LiF coated non-damaged devices have served as a benchmark for the SiC tests at room temperature.

It was found that all epitaxial SiC devices tested exhibited an increased effective dopant
density due to increased activation of dopants with increasing temperature, leading to some reduction in depletion width, and hence active thickness as well as to an increase in leakage current. Nevertheless, leakage currents only compromised the energy resolution of alpha particle spectroscopy (within the limitations of the set-up) at the highest applied bias voltages. Long term stability tests indicate that the devices respond in a stable manner within 8 hours of operation or less and, maintain performance for at least 24 hours. Stability is reached faster at higher temperatures. So far, the silicon devices show attractive response to alpha radiation and confirm their possible applications for hard radiation detection at room temperature including the large hadron colliders (LHC). The alpha particle detection and its stability at high temperature demonstrated by SiC samples is an indication of their suitability in harsh radiation detection applications. In addition, their neutron detection and its stability imply future application in oil and gas prospecting and nuclear reactor monitoring.
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Chapter 1

Introduction


Detector technology is a vast area of research that has gained a lot of attention from nuclear and related industries. Apart from detecting radiation at room temperatures, another challenge is the use of the detectors in harsh environments. Harsh radiation media are characterised by high temperature, high radiation fluence, high pressure and sometimes vibration. These conditions are present in nuclear reactor cores and oil and gas prospecting. The magnitude of these parameters differs from one environment to another depending on the application [1].

Due to their wide band gap (3.2 eV for 4H-SiC at room temperature) [2], limited leakage current even at elevated temperatures (in excess of 200°C) [3], SiC devices are ideal for harsh environmental applications. In addition, the devices are chemically and mechanically stable [3, 4, 5]. They are widely used for nuclear radiation detection [6, 7, 8, 9, 10, 11, 12]. Other potential areas requiring High Temperature Electronics (HTE) include nuclear well logging,
aerospace and space craft as well as automobile industries. The well logging sector is one of the largest areas that operates at high temperatures. The temperature ranges from 75 °C up to 312 °C, for logging that takes longer periods [13, 14]. This increase in required operating temperature is expected to continue to match the corresponding increase in the desired depth of investigation. In the auto-mobile industry high temperature electronics (HTE) are required to reduce engine exhaust emissions and improve fuel efficiency while in aerospace and space craft it is necessary for weight and size reduction as well as efficiency. In addition to the high temperature exposure, sometimes sensors are exposed to vibrations and shocks [15]. Therefore, demand for new materials and technology that can cope with harsh radiation environments is one of the central challenges in nuclear radiation detection applications. With respect to space research, a group at the University of Leicester is currently looking at the possibility of developing an X-ray SiC detector for space activities [16].

Nuclear well logging utilizes radiation of a nuclear radiation source which will interact with the formation; the final signal detected by the detector gives information about the qualitative and quantitative properties of the formation [17]. The concern has always been about the performance of the material used for the detection under harsh radiation conditions.

Schottky diodes (or metal semiconductor devices) were considered in this project because of their numerous advantages such as being majority carrier devices, which enable them to have zero reverse recovery charge. They are also fast switching devices. Although some of these properties are seemed to be more applicable to electronic devices, fast response is still advantageous to radiation detection applications.

The characteristics of the metal-semiconductor interface are as important as the charge transport properties of the bulk material because the devices are subjected to harsh environments. The two important parameters are Schottky barrier height (SBH) and ideality
factor (IF). Ha and Kim [18] observed spatial non-uniformity in barrier height for the 4H-SiC/Ni interface with increasing temperature. They believed that the cause of this anomaly arises from potential fluctuations caused by crystal defects, inhomogeneous doping and other related defects. On the other hand, the model suggested by Tung [19] predicts the presence of non-uniform regions or patches locally, with relatively low and high barrier height. Since IF and SBH are strongly dependent on temperature, a Gaussian distribution suggested by Werner and Güttler [20] can successfully explain the I-V(T) characteristics of semiconductor devices [21].

One of the earliest works on the suitability of using SiC for high temperature applications was conducted by Babcock and Chang [22]. The study involves exposing a SiC (p–n junction) detector to alpha radiation at high temperatures; extending up to 800 °C. They observed that the magnitude of capacitance increases as the temperature is raised, implying that more carriers become ionized. Because they expected too much noise generation as a result of thermal agitation, they recorded the alpha spectra at high temperatures at zero bias voltage. This restriction of bias voltage would deny the chance of knowing the effect of temperature on the voltage dependent charge collection efficiency (CCE). In addition, a single cycle (heating and cooling once) was used for the high temperature measurement, limiting the understanding of structural changes in the devices as a result of heating and cooling. Garcia et al. [23] demonstrated the possibility of operating the devices for more than 10 cycles; however they suggested further investigation for extended periods of exposure, using devices with increased thickness of Schottky contacts (i.e. 200 nm). In a similar development, Ha and Kim [24] investigated the thermal dependence of properties of p-n-n–type SiC detector for application in harsh environment. Their measurement of alpha spectra above room temperature was also carried out at zero bias. The reduction in CCE was relatively small but, there was significant broadening of the peak beyond 12 Volts bias. In an attempt to use a UV SiC detector for radiation detection in harsh
media, Metzger et al. [25] exposed 6H-SiC UV detector to Gamma-rays, proton and neutron radiation at high temperature. The response (induced current versus dose rate) of the device was linear with respect to Gamma radiation at high temperatures up to 200 °C. With 1000 kGy (Air) dose of Gamma-rays, $8.5 \times 10^{12}$ cm$^{-2}$ fluence of 32 MeV protons and $4.1 \times 10^{12}$ cm$^{-2}$ fluence of 14 MeV neutrons, the device performance did not change significantly. Beyond 200 °C, the response of the device was significantly affected.

Other studies conducted at high temperature involve the use of Schottky SiC devices operated at about 100 °C. Ruddy et al. [26] compared the alpha detection properties of Schottky and p-n junction of 4H-SiC devices between 22 and 89 °C and discovered that the Schottky devices have better energy resolution than the p-n junction ones. Additionally no temperature effect was noticed in the devices. This was supported by Nava et al. [27], who used 4H-SiC Schottky devices for alpha and beta detection at increased temperatures up to 94 °C. In the most recent investigation, Kalinina et al. [28] demonstrated that an Al-ion implanted p-n junction (SiC) can be operated effectively in the temperature range of 20 – 500 °C. They also established that CCE increases with raising temperature and the overall noise of the system reduces at higher temperatures. The former was attributed to an increase in hole diffusion length with temperature, while the latter was due to annealing of some radiation defects.

Although temperature effects are important in studying the performance of these devices, high radiation fluence (precisely protons and neutrons) has a greater effect on their detection ability. Nava et al. [29] have presented a comprehensive review in this regard. In the paper, particular emphasis was given to the CCE and resolution degradation of the devices under high radiation fluence.

In most of the studies published for charge particle radiation detection at high temperature using SiC devices, the observations are either limited to 100 °C, or the spectral acquisition was carried out at single bias voltage. In some cases no voltage was applied at
high temperature. Also, the long term stability of the devices at high temperatures has not been reported yet.

Many semiconducting elements are currently in use with respect to radiation detection. These materials include Germanium, Silicon and Diamond. Others are compound semiconductors, such as, Silicon Carbide, Gallium Arsenide, Gallium Nitride, Cadmium Telluride and Cadmium Zinc Telluride [30]. As temperature is a limiting factor on the radiation detection performance of semiconductors with small band gap, wide band gap (WBG) semiconductors such as SiC will play a key role in this regard.

In this work, the SiC suitability for alpha and neutron detection at high temperature and its stability at high temperature was studied. Under different temperatures, electronic and spectroscopic properties of the material were investigated.

International Atomic Energy Agency (IAEA) Coordinated Research Project (CRP) No. 17087, is a collaboration that focuses on the utilization of ion accelerators for studying and modelling of radiation induced defects in semiconductors and insulators and apply the new method to SiC and diamond. The responsibility for the Surrey group (as part of the collaboration) is to investigate changes in defects present in silicon devices resulting from ion irradiation. These changes are to be correlated to change in charge transport of the devices. In order to achieve this, silicon devices were characterised for current conduction and alpha particle detection. Other silicon materials that were damaged by proton irradiation were also characterized. As a proof of concept, some of the undamaged silicon detectors were used for lithium fluoride (LiF) deposition in preparation for thermal neutron detection.
1.1 Nuclear well logging

The current challenges posed by an increased number of high pressure high temperature (HPHT) wells in many areas of the world such as the North sea, the Gulf of Mexico and China (see Figure 1.1) are clear indications of the need for alternative technology [15, 31, 32]. Wells with temperatures above 150 °C are classified as high temperature, while those with pressures greater than 69 MPa are considered as high pressure. A special case where the temperature and pressure are more than 211 °C and 103 MPa respectively is called ultra-HPHT [31]

![Figure 1.1: Spread of HPHT oil wells world wide. Wells with temperatures above 150 °C are classified as high temperature while those with pressures greater than 69 MPa are considered as high pressure. A special case where the temperatures and pressure are more than 211 °C and 103 MPa respectively is called ultra-HPHT. Image courtesy of Schlumberger Oilfield Review, Summer 1998 [31]](image-url)

Deep water wells are needed to explore the reservoirs which in turn, require more sophisticated technology in evaluating the formation. Temperatures expected are in excess of 175 °C in most drilling wells [14]. The common drilling tool utilizes natural gamma-gamma density (measurement that uses a source of gamma rays and a detector of gamma rays)
and neutron porosity measurements [33]. These current challenges highlighted in figure 1.1 necessitate the need for alternative detectors that can operate at high temperatures, different vibration levels and shock and sizeable to fit into the space allotted to them in the logging tool.

Logging technology determines parameters such as resistivity, radioactivity and other parameters to measure formation characteristics which include porosity, lithology, permeability and hydrocarbon or water content [17].

In nuclear logging, the measurements utilize either gamma rays or neutrons. It has been realized that laboratory chemical analysis of formation samples is not only time-consuming but also expensive; as such in-situ measurement is more appropriate [34]. When a nucleus of any atom is subjected to excitation, it is capable of emitting $\gamma$-radiation which possesses characteristic energies of the particular atom and may be used to uniquely identify the atom [35]. Bulk density of formation is a major quantity measurable using $\gamma$-ray logging, since the scattering and transmission of the gamma ray is dependent on the formation density [33].

Neutrons on the other hand, interact with geological formation in two ways. Firstly, bulk properties of the formation can influence the transmission and moderation of the neutrons. In principle, neutrons are efficiently scattered by hydrogen rich materials [35]. Secondly, for neutrons with much greater energy, a nucleus may be excited so that it can emit characteristic gamma rays. The neutron measurements may involve either slowing down fast neutrons by the formation and/or $\gamma$-ray production [33].

Measurement may either be based on passive or active techniques. Passive techniques are simpler, where natural $\gamma$-rays (NG) emitted by uranium (U$^{238}$) thorium (Th$^{232}$) and potassium (K$^{40}$) in the formation are measured [33]. The result will either give information about the layers in the hot formation or relative concentration of the radionuclide, depending on the approach. Active technique use a radiation source (e.g. Caesium (Cs$^{137}$
1.1. NUCLEAR WELL LOGGING

\(\gamma\)-source), Calcium (Ca\(^{52}\)), Americium-Beryllium (Am-Be) or a pulse neutron generator (PNG) and \(\gamma\)-rays or neutron detectors which are housed in the logging tool. PNG is a source of neutrons based on Deuterium–Tritium (D–T) or Deuterium–Deuterium (D–D) fusion reaction. This reaction forms an unstable nucleus which decays to form neutron and helium nuclei. Concerns raised by different security and regulatory agencies of possible dangers which could result from using radioisotope-based sources (precisely Am-Be), suggest taking PNG as the preferred neutron source for nuclear well logging applications [36, 37].

The logging is carried out by deploying the logging tool into the borehole using a wire line (WL) or logging while drilling (LWD) technique (Figure 1.2). In the first case, the equipment is lowered into the hole after drilling has been performed and is drawn out of the hole at constant speed [37]. LWD on the other hand, allow in-situ measurement such that the detectors as well as the radiation sources are located inside the drilling tool so that logging and drilling takes place simultaneously [34]. In this method, vibrations and shocks are expected due to the rotation and grinding inside the well [15].

\begin{figure}[h]
\centering
\includegraphics[width=0.7\textwidth]{figure1.png}
\caption{Comparison between wire-line logging (WL) and logging while drilling (LWD) ©2010 IEEE [14]}
\end{figure}

Since this investigation is concerned with measurements based on neutron interaction,
the following sections will focus on neutron applied measurements.

1.1.1 Theoretical consideration

When the neutrons are released from a radiation source, they travel through the formation to the detector. This transport depends on the properties of the medium. The transport of neutrons may be represented by the Boltzmann transport equation (BTE) [34] as

\[
\overline{\Omega} \cdot \nabla \Phi + \sum_l \Phi = \int dE' \int d\Omega' \sum_s \left( E' \rightarrow E \right) \sum \left( \Omega' \rightarrow \Omega \right) \Phi + S
\]

(1.1)

Where \( \Phi \) is the angular flux specifying the number of neutrons crossing a unit surface area per unit time in the direction \( \overline{\Omega} \), while \( E' \rightarrow E \) is the energy interval at any point in space. The first term on the left hand side of equation 1.1 represents the net rate of loss of neutrons out of the volume in the direction \( \overline{\Omega} \), while the second term is the rate of reaction. Also on the right hand side the first term is the scattering cross section integrated over neutrons with energy \( E' > E \) over all directions \( \overline{\Omega} \) and \( S \) represents the source.

The complex nature of equation 1.1 makes it impossible to be solved analytically [33]. However, other forms of approach have been considered based on numerical methods. These include Monte Carlo methods, the discrete coordinate method as well as a diffusion approximation [34].

1.1.2 Neutron porosity logging [34]

The first nuclear tool used in determining appropriate porosity of geologic formation was a neutron logging tool because, the hydrogen index (density of hydrogen relative to that of water) is directly related to porosity. Since hydrogen has a very large scattering cross section, it can effectively slow down fast neutrons (this aspect is discussed in details in section 2.3.1). For a formation that is rich in water or hydrocarbon content, the scattered
1.1. NUCLEAR WELL LOGGING

(slow) neutrons resulting from interaction of fast neutrons with the formation, give an idea about its porosity. By measuring neutron population at two or three different distances from the source, the concentration of hydrogen can be determined. The use of two or more detectors cancel out borehole effects. A number of parameters affect the result of the porosity measurement. These include rock type, shale and pressure of gas in the logging zone.

1.1.3 Thermal neutron capture measurement [34]

In this method, the ability of the formation to adsorb thermal neutrons is the parameter of interest. When the fast neutrons emitted from the PNG interact with the formation through elastic and inelastic scattering, they lose their energy and form a cloud of thermal neutrons which are captured later via capture reactions. These captured neutrons emit gamma rays. The relationship between the decay time of the neutron cloud (estimated by $\gamma$–ray emission) and the proceeding emission of fast neutrons from the PNG gives an estimate of thermal neutron capture cross section which relates to mineralogical properties of that formation. The elements of interest are chlorine, iron and various rare earth elements. However, if energy spectra of the $\gamma$–rays are used, elemental composition of the formation can be inferred.

1.1.4 Environmental conditions and choice of detector

The environment within which logging will be carried out is the first point of consideration, then followed by the type of technique and nature of signal to be detected.

The nuclear well logging environment is often classified as harsh environment, because of high temperature, pressure and/or vibrations/shocks experienced during the exercise [17]. The choice of detecting element depends on its ability to withstand all these conditions mentioned. The environmental temperature ranges from 175 to 250 °C (Table 1.1), while
the down hole pressure is between 10000 and 30000 Psi (approximately 69 to 207Mpa) [14].

<table>
<thead>
<tr>
<th>Environmental parameter</th>
<th>WL</th>
<th>LWD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Environmental temperature</td>
<td>up to 250°C</td>
<td>up to 175°C</td>
</tr>
<tr>
<td>Detector operational temperature</td>
<td>(75–175°C)</td>
<td>(150–175°C)</td>
</tr>
<tr>
<td>Vibration level</td>
<td>N/A</td>
<td>≈30 g RMS</td>
</tr>
<tr>
<td>Shock level</td>
<td>N/A</td>
<td>up to 70 g</td>
</tr>
<tr>
<td>Down-hole pressure</td>
<td>10,000–30,000 psi</td>
<td>&gt;20,000</td>
</tr>
<tr>
<td>Operational time</td>
<td>≤10 hours</td>
<td>several hundred hours</td>
</tr>
<tr>
<td>Detector diameter</td>
<td>≈30 mm, ≈50 mm or ≈85 mm</td>
<td>(depending on tool diameter)</td>
</tr>
<tr>
<td></td>
<td>(depending on tool diameter)</td>
<td>(25–40) mm</td>
</tr>
<tr>
<td>Thickness of detector case</td>
<td>4–20 mm</td>
<td>&gt;15 mm</td>
</tr>
<tr>
<td>Typical logging speed</td>
<td>3–5 m/min depending on</td>
<td>&lt;0.3 m/min depending on drill bit</td>
</tr>
<tr>
<td></td>
<td>pooling speed (constant)</td>
<td>penetration rate (variable)</td>
</tr>
<tr>
<td>Imaging</td>
<td>no</td>
<td>yes</td>
</tr>
<tr>
<td>Tool lifetime</td>
<td>10–15 years</td>
<td>≈1 year</td>
</tr>
</tbody>
</table>

Table 1.1: Environmental properties and how they impose restrictions to WL and LWD techniques [14]

Neutron porosity measurement in HPHT environments, is affected by either increase or decrease in the actual value expected [31]. Higher temperatures cause an average increase in neutron energy which will reduce the neutron cross section while high pressure results in the reverse effect. Both situations can introduce changes in the measurement and therefore require correction. The situation could be avoided by using epithermal neutrons as they are less susceptible to temperature effects [31].

The life time for WL is much longer than that of LWD, because there is more wear and tear in the latter [14]. Due to attenuation and size effects, data obtained from WL have superior quality compared to the one acquired with LWD. On the other hand, low logging speed and rotation of detector as well as the massive tool body are figures of merit in the LWD technique.

Depth of investigation depends on the following factors [33]

- The type of neutron measurement which may be epithermal, thermal or neutron capture as well as the tool geometry
- Hydrogen concentration in the formation determines the depth of penetration of neutrons; which decreases with increasing hydrogen concentration.
1.1. NUCLEAR WELL LOGGING

- detector enclosure used to screen the detector away from direct source radiation can cause non-uniform isotropic efficiency.

The borehole characteristics such as mud-type, hole diameter and tool positioning can also influence the measurement [34].

All the environmental effects discussed make detector selection a herculean task. It is also worth noting that, one important way of successful logging is identifying a convenient detector measurement system.

Currently, there are about six different detectors used for WL and LWD technology. Table 1.2 presents a summary of these detectors in relation to their characteristics and market demand. $\gamma$-ray counters (which utilizes mainly NaI) for NG measurement experiences highest demand from oil and gas industries. From the table, it can be seen that NaI is suitable in principle for all the measurement types except neutron porosity measurement. However, efficiency and thermal instability limit its application suitability [38]. It has also been shown [38] that, its energy resolution fluctuates with temperature (precisely at higher temperatures). The recent introduction of other scintillation materials such as LaBr$_3$:Ce (lathanium halide) and gadolinium-yttrium orthosilicate (Gd$_{1.8}$Y$_{0.2}$SiO$_5$) simply referred to as GYSO has attracted the attention of many industries, but cost related issues have inhibited a large percentage of industries to implement it; they are five times more expensive than NaI[14].
### CHAPTER 1. INTRODUCTION

<table>
<thead>
<tr>
<th>Measurement type</th>
<th>Detector type</th>
<th>Current detector in use</th>
<th>Main optimization parameter</th>
<th>Performance degradation at high temperature</th>
<th>Problems</th>
<th>Industrial annual consumption (WL/LWD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG measurements</td>
<td>γ-ray counter</td>
<td>Majority NaI</td>
<td>Efficiency</td>
<td>High</td>
<td>High efficiency is needed</td>
<td>&lt;1000/&lt;10000</td>
</tr>
<tr>
<td>NG spectroscopy measurements</td>
<td>γ-ray spectrometer (0 to ≈ 3MeV)</td>
<td>NaI</td>
<td>Efficiency</td>
<td>Medium</td>
<td>Higher efficiency is needed</td>
<td>&lt;50/&lt;100</td>
</tr>
<tr>
<td>Gamma-Gamma density</td>
<td>γ-ray spectrometer (0-≈ 0.7MeV)</td>
<td>NaI</td>
<td>Efficiency</td>
<td>Medium</td>
<td>Quality/reliability cost for Li-6 glass</td>
<td>&lt;100/&lt;300</td>
</tr>
<tr>
<td>Neutron porosity</td>
<td>neutron counter for He-3, low quality spectrometer for Li-6</td>
<td>He-3 and Li-6 glass</td>
<td>Efficiency for neutrons; low sensitivity for gamma rays</td>
<td>High for He-3 medium for Li-6</td>
<td>Quality/reliability cost for Li-6 glass</td>
<td>&lt;100/&lt;300</td>
</tr>
<tr>
<td>Thermal neutron capture measurements</td>
<td>γ-ray spectrometer (0-≈8MeV)</td>
<td>NaI and BGO</td>
<td>Efficiency; spectral resolution low</td>
<td>High efficiency at high temperature is desired</td>
<td>Higher efficiency is needed</td>
<td>&lt;50/&lt;50</td>
</tr>
<tr>
<td>Capture and prompt gamma spectroscopy measurements</td>
<td>γ-ray spectrometer (0-≈8MeV)</td>
<td>NaI and BGO</td>
<td>Efficiency; spectral resolution low</td>
<td>High efficiency at high temperature is desired</td>
<td>Higher efficiency is needed</td>
<td>&lt;50/&lt;50</td>
</tr>
</tbody>
</table>

#### Table 1.2: Nuclear radiation detectors currently in use and market requirement for WL and LWD techniques [14]

Apart from the cost related issues, LaBr$_3$:Ce and LaCl$_3$:Ce suffer from internal radioactivity because of La-138 and Ac-227 [39, 40] and their resolution advantage diminishes at energies below $\approx$100 keV [40]. Recently, strontium iodide (SrI$_2$(Eu)) was found to exhibit highest light yield compared to other europium doped halides (e.g. CaI$_2$, SrBr$_2$ and BaI$_2$) but delayed luminescence and intrinsic non-proportionality discourages its usage [41]. Furthermore, the use of these types of detectors may only look promising if careful detector engineering and optimized optics design is put in place.

The light yield dependence on temperature of GYSO is more significant than that of LaBr$_3$:Ce and its instability at temperatures above 100 °C is of great concern [14]. Nevertheless it has potential for room temperature $\gamma$-ray counting because of its high Z advantage. Gadolinium orthosilicate (GSO) the only detector developed solely for nuclear well logging applications suffer from poor light output; as such its technology was not pursued further [42].

Lithium ($^6$Li) glass was introduced in the early 90’s as a promising nuclear porosity measurement detector [43], but general glass scintillator limitations like response to $\gamma$-rays create an unwanted background in addition to its lower reliability (compared to other
detectors such as $^3$He detector) and signal deterioration at high temperatures (at/above 150 °C) does not guarantee that it can substitute $^3$He detectors [14]. The best detector so far for neutron porosity measurement ($^3$He) has been cost prohibitive, because of the global $^3$He shortage [44, 45].

In summary, one can see that there is a need for nuclear radiation detectors that can:

1. Replace $^3$He

2. Detect gamma-rays reliably at high temperatures (175 °C or higher) for hundred hours

3. Operate within harsh environment (high pressure, vibrations and shocks) without device failure for long duration

The prospects of SiC in HTE; to which oil and gas logging is inclusive, have been highlighted. Challenges experienced with current detectors have been discussed. Considering these two important points, this research suggests that SiC can serve as an alternative detector for neutron porosity measurements.

The writer is only aware of low neutron detection efficiency as a limitation in SiC and, more investigations are under-way to improve it (for detail discussion on this refer to section 2.3.1).

1.2 Thesis outline

Having introduced the relevance of SiC in high energy physics applications as well as the general focus of this project, a brief explanation is given in the following about the constituents of each chapter.

Chapter 2: Chapter two consists of a discussion of the relevant semiconductor principles with respect to detecting radiation. Operational characteristics of silicon and silicon carbide
detectors are highlighted. It starts with interaction of radiation with semiconductors. Subsequently, metal semiconductor junction and its relationship with charge carrier generation and transport is presented. The last part of the chapter described some of the defects in semiconductors and how they can influence the performance of devices as well as method of characterising them.

Chapter 3: Chapter three focuses on different experimental methods, equipment and set-up used in this research. It begins by explaining the properties of the samples used followed by their fabrication process and mounting on ceramic printed circuit board (PCB). Each of the set-up used for current and capacitance measurement, alpha spectroscopy and thermal neutron detection was presented.

Chapter 4: Chapter 4 is the first results chapter. Here, Current/Voltage (I/V) and Capacitance/Voltage (C/V) characteristics of silicon samples were discussed. The relevant parameters for the two components were evaluated. These include resistivity, ideality factor barrier height, doping density and depletion thickness. The expected influence of these parameters to the device performance is discussed. Also alpha particle detection using the silicon devices was investigated in terms of charge collection efficiency (CCE) and energy resolution.

Chapter 5: Chapter five is the second result chapter and deals with operational stability of silicon carbide samples at high temperature. I/V and C/V results were presented as well as their relevant parameters at room and elevated temperatures. Alpha particle detection and its stability at high temperature is discussed. Finally, it relates the alpha particle detection efficiency and stability to thermal neutron detection.

Chapter 6: This chapter is the last result chapter. The chapter presents the findings on thermal neutron detection capability of silicon and silicon carbide devices at room and high
1.2. THESIS OUTLINE

temperature. The stability of thermal neutron detection with silicon carbide at elevated temperature was also discussed.

Chapter 7: This is a conclusion chapter which makes a recap of the major findings and their implication to the application of these devices and other devices in the wider community. It also highlighted the future of high temperature semiconductor detectors.
Chapter 2

Principles of semiconductor radiation detector

Basically, whether radiation detector is suitable for a particular application, depends upon its nature of interaction with the radiation of interest. As such, it is necessary to understand how radiation interacts with the detector element and how radiation sources lose their energy within the medium. Many studies are reported in relation to this very wide topic; Evans [46], Elliot [47] and Mudhole [48] are good examples among others.

Owing to the fact that this project is interested in neutron detectors, discussion on radiation interaction of particles other than neutrons will be limited.

Heavy charged particles’ and fast electrons’ interaction with matter is influenced by Coulomb force while uncharged particles (neutrons, X-rays and γ-rays) have to undergo a reaction that is capable of converting the signal into detectable energy.
2.1 Heavy charged particle interactions

Heavy particles such as alpha particles interact with electrons in matter by excitation or ionization. Because they are charged, for a particle of mass \( m \) and energy \( E \) striking an electron of mass \( m_0 \), the maximum energy transferred to the electron is \( 4Em_0/m \) [35]. Since this energy is very small, the particle is expected to make many such interactions with the electrons as it penetrates into the absorber medium. This ionization is capable of creating electron – ion pairs (electron-hole pairs in a semiconductor) whose movement finally creates a detectable current signal.

The particle’s energy loss in the material is often described with the help of rate of energy loss which is the differential of energy loss in the material per unit path length. The Bethe formula \([35, 49, 50]\) gives a relation for specific energy loss of particle given as

\[
-\frac{dE}{dx} = \frac{4\pi e^2 z^2}{m_0 v^2} N Z \left[ \ln \frac{2m_0 v^2}{I} - \ln \left( 1 - \frac{v^2}{c^2} \right) - \frac{v^2}{c^2} - \frac{\delta}{2} \right]
\] (2.1)

Where \( ze \) and \( v \) are charge and velocity of the primary charged particle respectively. The absorber which has atomic number \( Z \) and density \( N \) possesses an average ionization and excitation potential \( I \) which is determined experimentally for each element; while \( e \) is electronic charge. The quantity \( \frac{\delta}{2} \) represents the density effect correction to ionization energy loss; which is important at higher energies (in the GeV range) [50]. The energy loss therefore, depends largely on the \( NZ \) rather than the logarithmic term.

Equation 2.1 is only valid when the charge sharing between the particles and absorbing medium is negligible.

The distance travelled by a particle in a material is dependent on the material. For any detector to be suitable for alpha particle spectroscopy for example, it must have an active thickness that is greater than the range of the alpha particle. Particles with the same initial energy may vary in the depth of penetration in the absorber medium, because
CHAPTER 2. PRINCIPLES OF SEMICONDUCTOR RADIATION DETECTOR

their interaction is subject to the statistical variations in the energy loss process [51]; a property called straggling. The effect of straggling can be easily visualized using the Bragg curves illustrated in figure 2.1

![Bragg curve](image)

**Figure 2.1:** Bragg curve [52].

2.2 Electromagnetic radiation interactions

The interaction of electromagnetic radiation (X rays or gamma rays) with matter is in sharp disparity to that of charged particles. Here the photon may be lost completely or scattered through an angle. Of the many known interactions of photons with matter, the most relevant ones to our discussion are photoelectric absorption (PA), Compton scattering (CS) and pair production (PP). The importance of these interactions with matter depends on the energy of the photon ($E_\gamma$) as well as the atomic number of the absorber [35]. For lower energies PA dominates while the PP dominates at higher energies; whereas for mid-energies CS dominates [53]. Figure 2.2 shows the relevance of each process with increasing energy and atomic number of the absorber.

---

2.2. ELECTROMAGNETIC RADIATION INTERACTIONS

Figure 2.2: Relevance of the major types of gamma rays interaction with matter.

- **Photoelectric absorption:** When an electron in the bound state (mostly K-electron) of an atom gains adequate energy from a gamma ray photon (typically 100 keV or less) it is liberated with an energy given by [53]

\[
E_e = E_\gamma - E_b
\]  

(2.2)

where \(E_\gamma\) is the gamma ray energy and \(E_b\) the binding energy of the photo-electron. The atom from which this electron is removed becomes excited because of the created vacancy. This vacancy is filled by an electron from a shell with higher energy causing release of photons or Auger electrons. The energy of the photon released is the difference between the energies of the shells involved in the process. To estimate whether the interaction of a photon with a medium will result in photoelectric absorption is somehow complex; as such there is no viable analytical solution for it [35]. However, an approximate expression of probability of interaction known as photoelectric cross section \(\tau\) is given by [54]
\[ \tau = aN \frac{Z^n}{E_m^m}[1 - O(Z)] \]  

(2.3)

Where \( m \) and \( n \) are values (between 3 to 5) depending on the \( E_\gamma \) value. The constant \( a \) is independent of atomic number \( Z \) and \( E_\gamma \), while \( N \) is number of atoms/m\(^3\) in the material [53, 54].

The large dependence of PA on \( Z \), reflects the need for high \( Z \) materials (lead for example) to shield gamma rays. In addition, a detector with high \( Z \) constituents may not be good for experiments that require gamma ray insensitivity.

- **Compton scattering:** In CS, the energy of the photon is transferred to an electron that is loosely bound to the atom (most precisely valence electrons). Assuming the electron to be at rest, the photon is deflected through an angle \( \theta \) (Figure 2.3); while the recoil electron having gained a fraction of energy from the photon is deflected through angle \( \phi \).

\[ E' = E_\gamma - E_e \]  

(2.4)

Due to energy conservation, the energy of the scattered photon is given by [53]

For elastic scattering, the principle of momentum conservation can be applied; so that [35]
2.3. NEUTRON INTERACTIONS

\[ E'_\gamma = \frac{E_\gamma}{1 + \frac{E_\gamma(1-\cos\theta)}{m_0c^2}} \]  

(2.5)

Since CS takes place between photons and valence electrons of atoms of the absorbing medium, the scattering cross section per atom varies linearly with atomic number and as such this process is favoured by high Z materials.

- **Pair production:** For large gamma ray energy, an electron–positron pair can be created and the photon disappears in the process. The pair production can only take place if two conditions are satisfied; first, the interactions have to take place within a field controlled by Coulomb force. Second, the energy of the \( \gamma \)-ray photon should be more than twice the electron rest mass energy \( (E_\gamma > 2m_0c^2) \). The necessary condition for energy conservation in this reaction is given by [35]

\[ E_\gamma - E_e - E_p = 2m_0c^2 \]  

(2.6)

The chance of occurrence of PP is directly proportional to the square of atomic number of the absorbing medium [54]. Also from equation 2.6, it is clear that this type of interaction is only relevant at higher energies of \( \gamma \)-ray photons greater than 1.022 MeV.

2.3 Neutron interactions

Like \( \gamma \)-rays, neutrons have no charge and can only interact with the nucleus of constituent atoms of the absorbing medium; giving them the ability to travel far through the material. This interaction may change the energy and/or direction of the incident neutron or completely substitute it with secondary radiations. The secondary radiations are predom-
inantly heavy charged particles. Since the primary interaction of neutrons with absorbing medium does not result in direct ionization, mechanisms of detecting them rely on a medium (converter layer) capable of converting the neutrons into charged particles before they are finally detected within the detector medium.

There are basically two types of neutron interactions; slow and fast neutron interactions respectively. These interactions depend on the energy of the incident neutron as well as the nature of the target material. The most common neutron energies range from 0.025 eV (for thermal neutrons) to 10 MeV (for fast neutrons). Table 2.1 presents the nomenclature of neutrons relating to their energies. These ranges are not strictly separated and there could be other neutrons with energy outside the ranges, but these are the commonly used classifications.

<table>
<thead>
<tr>
<th>Neutron type</th>
<th>Energy range</th>
</tr>
</thead>
<tbody>
<tr>
<td>High energy</td>
<td>&gt;10 MeV</td>
</tr>
<tr>
<td>Fast neutrons</td>
<td>10 keV-10 MeV</td>
</tr>
<tr>
<td>Intermediate</td>
<td>100 eV-10 keV</td>
</tr>
<tr>
<td>Slow</td>
<td>10 eV-100 eV</td>
</tr>
<tr>
<td>Epithermal</td>
<td>0.2 eV-10 eV</td>
</tr>
<tr>
<td>Thermal</td>
<td>0.025 eV</td>
</tr>
</tbody>
</table>

Table 2.1: Classification of neutrons based on their energy [33].

2.3.1 Slow neutron interactions

The most relevant interactions of slow neutrons (which are dominated by thermal neutrons with energy 0.025 eV at room temperature) are neutron-induced reactions; those that result in reaction products that can generate electron-hole pairs in a detector element.

As soon as neutrons react with a target medium, secondary neutrons or other reaction particles such as alpha particles, gamma rays, and nucleons may be released depending on the energy of the neutron [35]. For thermal neutrons, \(^{10}\text{B}(n,\alpha)^7\text{Li}\) and \(^{6}\text{Li}(n,\alpha)^{3}\text{H}\) are the two most common interactions explored for detection purposes [35, 54, 55, 56]. Boron
2.3. NEUTRON INTERACTIONS

has a larger reaction cross section (3480 barns) with alpha energies of 1.47 MeV and 1.78 MeV for excited state and ground state respectively [54]. Lithium on the other hand, has a lower reaction cross section (940 barns) but higher alpha energy (2.05 MeV) [35]. Because of the nature of its reaction, pure lithium is difficult to use for conversion layers [57].

Basically, most semiconductor neutron detectors consist of two layers joint together: a neutron reactive layer and a semiconductor diode [58]; though recent studies have shown that boron carbide/silicon and magnesium boride/silicon hetero-structures are possible neutron detectors with higher efficiencies [59, 60].

When the neutrons are absorbed by the target nucleus, electron–hole (e–h) pairs are generated through the interaction of the reaction products with the semiconductor and their presence detected when they induce charge in the semiconductor detector [58]. The process of conversion takes place in the neutron reactive coating (converter layer); the converter layer can either be direct or indirect. A direct converter layer is always recommended because, indirect converters tend to be limited in efficiency caused by self absorption losses [61, 62]. In direct converter layer, the material for conversion plays the role of electron–hole pair creation in addition to conversion of neutron into detectable charge particle (for example, hetero-junction). On the other hand, if the material only converts the neutron into charge particle it is regarded as indirect converter layer.

One of the extensive works regarding the use of semiconductors for neutron detection was conducted by MCGregor et al. [57]. Although the larger part of the results presented in their study is theoretical, the assumptions made were valid and thus gives insight into the expected efficiency of neutron detection using converter layers. From the result, the estimated thickness for optimum thermal neutron detection for pure lithium ($^6$Li), lithium fluoride ($^6$LiF) and boron ($^{10}$B) are approximately 100 µm (11.5%), 25 µm (4.8%) and 3 µm (%4.8) respectively. Further, a stack configuration of the detectors up to 15 detectors, indicates potential for increase in thermal neutron detection efficiency in all the materials;
with $^6$Li showing greater efficiency.

In spite of growing technology of semiconductor material synthesis and processing which has existed for more than 6 decades, the reported neutron detection efficiencies have been very small compared to that of He-3 gas neutron detectors [57].

When the neutrons come into contact with the reactive film, charged particles are created. These charged particles will travel through any dead layer into its active layer. The charged particles create e-h pairs which are separated by an electric field in the active layer and, the electric field is typically caused by a diode structure which can be increased by biasing the detector. Mobile charges created induce image charge on either contacts of the detector as they move through; which can be measured using a charge or current sensitive preamplifier depending on the parameter of interest [57].

Polycrystalline diamond is a good alternative for neutron detection but, its uneven response to radiation and cost discouraged further attempts to be used for neutron detection applications [63]. However, recently, there is remarkable development in the technology of chemical vapour deposited (CVD) single crystal diamond; which has shown potential for its use as neutron detector even at high temperatures. Notwithstanding, the metal contact integrity is of significant concern [64, 65].

Most of the earlier investigations conducted on the optimization parameters of semiconductor neutron detectors concentrated on the thickness of the converter layer. However, recently Murphy et al. [58] attempted to determine the optimum thickness of the detector in relation to that of the converter layer. The work was based on simulations with MCNP4 and MCNP V2.7.0. For a 2.8 $\mu$m $^{10}$B thickness, he suggested 60 $\mu$m and 86 $\mu$m for diamond and silicon respectively. In the case of LiF with a thickness of 26.5 $\mu$m the optimum thickness was 49 $\mu$m and 74 $\mu$m for diamond and silicon respectively. Furthermore, least response to $\gamma$ background radiation could be achieved for energies of 511 and 1460 keV at thickness of 3 to 10 $\mu$m for both diamond and silicon detectors. These findings assured
that energy losses due to the thickness of the dead layers in all cases may be negligible for thin layers; as such the energy losses are ignored. Nonetheless, there is need for laboratory based findings and/or simulations taking the dead layer into account to confirm the assumptions.

In some of the recent studies, heterostructures formed have demonstrated a greater efficiency in neutron detection. The materials so far fabricated are n-type boron carbide/p-type silicon with efficiency of 50% [59] and n-type magnesium boride/p-type silicon with expected efficiencies >30% for 10 µm enriched 10B [60]. In these types of semiconductor detectors neutron energy is converted and e–h pair creation takes place as well in the semiconducting converter layer.

Practically, LiF thickness of 1.5 and 1.8 µm respectively have been successfully applied on a silicon planar detector and used for thermal neutron detection by Barbagallo et al. [66]. Although in their study, the LiF was deposited on a removable polystyrene to allow test with and without converter layer, they have proven beyond reasonable doubt that the method could be used to realise a low cost neutron counter. Another fabrication option, is implantation of the converter material sandwiched between metal contacts as conducted by Issa et al. [67].

In the current project, thickness of LiF between 3–4 µm was used based on the fact that the deposition is directly on the surface of the metal contact.

2.3.2 Fast neutrons interaction

The possibility of neutron-induced reactions diminishes with increasing neutron energy [68]; as such the most relevant interactions here are scattering reactions. Products of this reaction are recoil nuclei that acquire a measurable amount of energy from the incident neutrons. On the other hand, if the energy of the fast neutrons is very high, inelastic scattering may take place [54]; which leaves the recoil nucleus in an excited state. Upon
de-excitation, γ–rays are produced and a large fraction of energy is lost by the recoil nucleus. This process is not employed frequently as it causes more technical hitches \[35\]. The vast majority of fast neutron detectors operate based on elastic scattering.

If a neutron with non-relativistic energy \(E_n \ll \text{MeV}\) strikes a target and the collision is elastic, the ratio of energy of the recoil nucleus \(E_r\) to that of the incident neutron \(E_n\) in the centre of mass coordinate system is given by \[35\]

\[
\frac{E_r}{E_n} = \frac{4A\cos^2\theta}{(1 + A)^2}
\] \hspace{1cm} (2.7)

Where \(A\) is the mass of the target nucleus and \(\theta\) is the angle of scattering of the recoil nucleus. From equation 2.7 two deductions can be made; at 90° there is little or no deflection of the incident neutron, making the recoil nucleus to move almost perpendicular to the direction of incident neutron resulting in a small value of the recoil energy \(E_r \to 0\). Conversely, if the angle of the recoil nucleus is 0° or the angle of the incident neutron is 180°, a head-on collision takes place and the recoil nucleus moves along the same line as the incident projectile, resulting in maximum recoil energy. In addition, the ratio (i.e \(E_r/E_n\)) can be increased with lower atomic number atoms. Hydrogen has the highest value of recoil energy in comparison to other elements such as helium, carbon, oxygen and silicon \[35\]. For this reason, low Z materials are attractive candidates for fast neutron detection.

2.4 Intrinsic and extrinsic semiconductors

An intrinsic semiconductor is an undoped semiconductor. Though in reality it is impossible to have a 100 percent pure semiconductor; due to impurities and other defects that may not be avoided during the production process.

The process of introducing impurities intentionally into a semiconductor is called doping; which can improve the properties of some semiconductors depending on the intended
applications. For radiation detection applications, physical (electrical and thermal) and chemical (active or inactive) parameters are most relevant [15]. In the proceeding discussion, the focus will be on radiation detection properties of semiconductors and their applications in harsh radiation environments.

Because of periodicity in the semiconductor lattice, some energy bands are established; which confine electrons to certain energy levels. A forbidden gap (otherwise known as band gap of a semiconductor) is located between the upper band (conduction band) and the lower band (valence band). These bands are very important in determining the electrical properties of a semiconductor [69]. In terms of band gap, semiconductors are divided into two: direct and indirect band gap semiconductors. Figure 2.8 shows the energy momentum (E-k) relationship for Si and GaAs. This momentum energy relationship can be approximated near the band maximum/minimum by

\[ E(k) = \frac{\hbar^2 k^2}{2m^*} \]  

where \( m^* \) is the effective mass. For silicon (Si) the valence band maximum occurs at \( \Gamma = 0 \) (where \( \Gamma \) is momentum) but the minimum in the conduction band occurs at nearly [100] where \( \Gamma \neq 0 \). This implies that a successful transition of electron from the valence to conduction band in Si does not only require change in energy but it also requires a change in momentum [70]. On the contrary, gallium arsenide (GaAs) has both its maximum in the valence band and minimum in conduction band positioned at \( \Gamma = 0 \). Therefore, electron transition from valence to conduction band in GaAs does not require change in its momentum. Because of this peculiar feature, semiconductors with energy-momentum relation similar to Si are called indirect band gap semiconductors while, for those with configuration similar to that of GaAs are called direct semiconductors. It is worth clarifying at this point that, there are many energy levels in a semiconductor detector below the
CHAPTER 2. PRINCIPLES OF SEMICONDUCTOR RADIATION DETECTOR

Figure 2.4: Energy-momentum relationship for (a) silicon and (b) gallium arsenide $E_g$ is the band gap energy [72]. Copyright by The American Physical Society, 1998.

Conduction and valence bands as depicted in the figure; but these two are most important in the current discussion. For an electron to move from valence band to the conduction band, this energy gap has to be overcome [71].

At a very low temperature (0 K) the valence band is completely full while the conduction band is completely empty, so that the material is not conducting [73]. On the other hand if the temperature is greater than 0 K, some electrons in the valence band gain sufficient energy such that the electron with the highest energy will jump to the conduction band leaving behind a vacancy (hole) [74]. The next electron with an energy closer to that of the escaped one, will move to fill the vacancy while creating another vacancy. Since the movement of electrons is opposite to that of the holes, then holes can be seen to act as positive charges. This interaction between electrons and holes establishes a conducting medium [73].

For an intrinsic semiconductor, the number of electrons in the conduction band must be the same as that of the holes in valence band. As mentioned earlier in the beginning of this section, due to some residual impurities this condition is not perfectly achievable [75]. For the doped semiconductors the situation is different in that, the electrical conductivity can depend on the type and concentration of the dopant. Silicon for example, has four (4)
valence electrons and each atom forms covalent bonds with the other adjacent four silicon atoms in its pure state (Figure 2.5). As shown in the diagram, each line represent electron sharing between two silicon atoms.

When silicon is doped with a group five (5) element (P, Sb and Sn), some atoms are substituted by the dopant. Since the dopant has 5 valence electrons, one electron (that is not bound to a specific atom) is left to move freely within the material; thus forming a cloud of free electrons. Because these electrons possess greater energy (thus weakly bond to the dopant atom), they form an energy level (donor level) just below the conduction band. The electrons can be excited at moderate temperature into the conduction band. However, the whole crystal remains neutral because the excess charges on the group 5 atoms will compensate for the free electrons. This process leaves the material with excess electrons and the doped semiconductor is called an n-type semiconductor [69].

Similarly, the semiconductor (silicon) may be doped with group 3 elements (B, Ga and Al) which are popularly called acceptors. When the substitution is accomplished, one electron of the silicon atom will be left unsaturated. This unsaturation creates a level called acceptor level just above the valence band. At room temperature, there will be sufficient energy to excite electrons from the valence band to the acceptor level. For every acceptor impurity atom a hole is generated [69]; thus making holes the majority carriers in the material. This type of material is called p-type semiconductor.
Therefore, at room temperature a donor atom will always create a free electron in the conduction band while each acceptor atom forms a hole in the valence band. Thus, each donor atom is positively ionized and the acceptor atoms are negatively ionized. The donor or acceptor atom requires an energy to be ionized which is called ionization energy.

2.5 Diode characteristics

Having discussed the intrinsic and extrinsic features of semiconductors, it is worth noting that one of the most important electronic systems is the p-n junction; which is achieved by intentionally changing the doping properties of some portion of a p or n-type substrate; one side of the new material will have electrons as the majority carriers (n-type) while the majority carriers in the other part are holes (p-type). This type of structure is commonly referred to as diode. Diodes exhibit the property of conducting current through one direction and blocking most of it from the other, as depicted in figure 2.6.

![Diode characteristics diagram](image)

**Figure 2.6:** Typical Current-Voltage characteristics of a diode. Although the magnitude of Current and Voltage may differ from one type of material to another, the blocking of current in one direction and conducting it in the other, is the most important feature.
2.5. DIODE CHARACTERISTICS

If it is assumed hypothetically that the two types of extrinsic semiconductors (p and n-types) are brought together, without applying any external field, the majority carriers from the p-side will drift into the n-side while the electrons from the n-side will drift into the p-side of the diode. At equilibrium, a region of electric field is created between the two sides. The electric field in this region sweeps electrons and holes away from the junction, thereby creating a space charge region. This is what happens to the p-n junction in the absence of an external field. If the p-side is heavily doped compared to the n-side (i.e. \( N_A \gg N_D \)), the thickness of the depletion layer \( d \), is given by [76]

\[
    d \approx \sqrt{\frac{2\varepsilon_s \varepsilon_0}{qN_D}} V_{bi}
\]

where \( \varepsilon_0 \) is permittivity of free space \((8.85 \times 10^{-14} \text{ F cm}^{-1})\), \( \varepsilon_s \) is the relative permittivity of the material \((11.9 \text{ for silicon and } 6.5 \text{ for SiC} \ [77])\), \( q \) is the electronic charge \((1.6 \times 10^{-19} \text{ C})\), \( V_{bi} \) is inbuilt potential and \( N_D \) is the doping density. Upon application of bias, the thermal equilibrium attained will be distorted. If a forward bias is applied, majority carriers on the p-side will be attracted towards the n-side and vice-versa; causing a reduction in thickness of the space charge region. On the other hand, if the bias applied is reversed, minority carriers will be attracted instead, and this will increase the thickness of the depletion layer. Similarly, the potential at the junction increases from \( V_{bi} \) to \( V_{bi} + V \), where \( V \) is the applied bias. Based on the earlier assumption of a high doping level on the p-side, \( V_{bi} \) can be neglected [76]. The relationship between depletion thickness \( w \) and the junction capacitance \( C \) is given by [71]

\[
    w = \frac{\varepsilon_0 \varepsilon_s A}{C}
\]

where \( A \) is the diode active area. Also, the relation for the doping density is given by [71]
CHAPTER 2. PRINCIPLES OF SEMICONDUCTOR RADIATION DETECTOR

\[ N_D = \frac{2}{q\varepsilon_0\varepsilon_s A^2 \frac{dV}{dV}} \] (2.11)

The depletion thickness can be used to estimate the magnitude of the full depletion voltage of a device, while the doping profile gives insight into the doping level in the material as well as its uniformity through its entire volume.

2.6 Metal-semiconductor junctions

Schottky diodes are metal semiconductor (MS) junctions created by establishing contact between a semiconductor wafer and a pure metal. When the thermal equilibrium is established (taking n–type material as an example), electrons flow from the semiconductor to the metal thus creating a depletion region below the metal contact which is devoid of free electrons [78]. One advantage of a metal semiconductor junction over the p–n junction is the possibility of controlling the barrier height (potential energy between semiconductor and metal contact) by careful selection of metal and semiconductor doping [79]. Since most of the dopant atoms in the semiconductor have lost some of their electrons, they become positively charged; which makes the metal negative with respect to the semiconductor. All the equations stated in section 2.5 are also applicable to MS junction devices.

The third situation considered, is when the diode under reverse bias is exposed to nuclear radiation capable of inducing ionisation in the material; this is discussed in detail in section 2.9.
2.6 Metal-semiconductor Junctions

2.6.1 Schottky barrier formation

Earlier theories such as the popular Schottky-Mott theories \cite{80, 81}, proposed that the Schottky barrier height (SBH) $\phi_B$, can be represented by

$$\phi_B = \phi_m - \chi_s$$  \hspace{1cm} (2.12)

where $\phi_m$ is the work function of the metal which denotes the energy difference between its vacuum and Fermi level and $\chi_s$ is the electron affinity representing the potential difference between the bottom of the conduction band and the vacuum level at the semiconductor surface (see figure 2.7).

![Energy-band diagram of Schottky barrier formation between a metal and semiconductor at thermal equilibrium \cite{82}](image)

**Figure 2.7:** Energy-band diagram of Schottky barrier formation between a metal and semiconductor at thermal equilibrium \cite{82}. $E_{FM}$: Fermi level of metal, $E_C$: conduction band, $E_F$: Fermi level of semiconductor, $E_V$: valence band, $\psi_{bi}$: built in potential $q\phi_m$: energy difference between Fermi level and conduction band of semiconductor, $\phi_{Bn}$: barrier height. Metal-Semiconductor Schottky Barrier Junctions and Their Applications, Physics of Schottky Barrier Junctions, 1984, pp. 1–60, Tyagi, M.S., (No part of this book may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission from the publisher) "with permission of Springer".

Equation 2.12 implies a perfect dependence of SBH on the work function and electron affinity. However, practical situations suggest other factors playing key roles in determining the SBH such as imperfect semiconductor surface, thin oxide layer at the interface and other
types of defects which give rise to surface states [83]. Barrier height engineering can become difficult due to its weak or no dependence on the electron affinity and work function. This behaviour of the barrier height make it somewhat constant commonly referred to as Fermi level (FL) pinning. Fortunately, there is strong dependence of barrier width on the doping of the semiconductor, which provides an alternative of producing an ohmic or Schottky contact by varying the doping level of the semiconductor [84]. Using this idea, one can form two different contacts (i.e. ohmic and Schottky) on a single semiconductor material by varying its doping density.

2.6.2 Influence of electric field on Schottky barrier

The equilibrium condition achieved between metal and semiconductor as depicted in figure 2.7 serves as boundary condition to the solution of Poisson’s equation of the semiconductor; which provides the relationship between electric field $\varepsilon$ and doping density $N_D$ given by

$$\frac{d\varepsilon}{dx} = \frac{qN_D}{\varepsilon_s} \ldots 0 \leq x \leq w$$

(2.13)

where $x$ is the depletion thickness with a maximum value of $w$. On applying a bias, either forward or reverse, the equilibrium state is distorted and the alignment of Fermi levels (FL) that existed between the two materials is changed. Under forward bias, the FL of metal is shifted below the FL of the semiconductor so that more electrons will move into the metal because they experience a barrier reduction (figure 2.8).
2.6. METAL-SEMICONDUCTOR JUNCTIONS

Figure 2.8: Energy-band diagram of Schottky barrier under forward bias [82]. Metal-Semiconductor Schottky Barrier Junctions and Their Applications, Physics of Schottky Barrier Junctions, 1984, pp. 1–60, Tyagi, M.S., (No part of this book may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission from the publisher) "with permission of Springer".

This will lead to a rapid rise of current exponentially obeying the normal diode equation discussed in section 2.6.4. If on the other hand the bias is reversed the FL of the metal will be shifted up so that the barrier height is increased. This will prevent electrons from moving into the metal from semiconductor (figure 2.9).
This creates a depletion region analogous to the p–n junction; consequently, the depletion width and doping density relations of the p–n junction diode are also applicable here.

### 2.6.3 Formation of Schottky and Ohmic contacts

Although there is a difference in work function between different metals which suggests variable interactions with semiconductor surfaces, successful Schottky junctions are formed by variation of doping density in most of the group III–V semiconductors. In silicon for example, an epitaxial layer grown on the surface of a bulk (conducting) substrate is doped lightly, so that the metal contact deposited on it will have a Schottky behaviour. Aluminium is one of the commonly sputtered metal in silicon devices.

A number of researchers such as Ruddy et al. [85], Cunningham et al. [8], Kazauskas et al. [86] and Mandal et al. [87] have reported Schottky contact formation on one face of semi insulating (SI) SiC and Ohmic contact on the other surface. Cunningham et al.
and Mandal et al. used nickel contact while, Kazauskas et al. used titanium. Ruddy et al. combined three different metals together (titanium/platinum/gold) forming three different layers one on top of another. However, it was not obvious to identify which side of the contacts is Schottky or Ohmic from the I/V curves.

A more successful Schottky contact formation on SiC is achieved by sputtering metal on the surface of an epitaxial layer (which is lightly doped) as reported by Nava et al. [88], Bruzzi et al. [7] and Lees et al. [89]. Nava et al. and Bruzzi et al. used gold but Lees et al. used a combination of titanium and nickel. Interestingly, I/V curves from these fabricated devices indicate clear diode characteristics; making it easier to differentiate Ohmic from Schottky contact.

Because of the requirement of this project (i.e. high temperature application), metals selected for sputtering had to satisfy certain criteria; such as non-reaction with atmosphere when heated and non-absorption by the semiconductor in order to avoid distorting the Schottky behaviour during long time operation.

### 2.6.4 Current conduction in the Schottky junction

The current transport in a Schottky barrier is dominated by majority carriers, unlike p-n junction devices. This is why Schottky devices are referred to as majority carrier devices. Based on the thermionic emission theory developed by Bethe [90], the current density from the semiconductor into metal is given by

\[
J_{s\rightarrow m} = A^* T^2 \exp \left( -\frac{q \phi_B}{kT} \right) \exp \left( \frac{qV}{nkT} \right)
\]  

(2.14)
where $A^*$ is the Richardson constant, $\phi_B$ barrier height, $k$ Boltzmann’s constant and $n$ is the ideality factor. The current flowing into the semiconductor is given by

$$J_{m \rightarrow s} = -A^*T^2 \exp\left(\frac{-q\phi_B}{kT}\right)$$  \hspace{1cm} (2.15)

The total current density is obtained by summing equations 2.14 and 2.15 which gives

$$J_n = J_s \left[ \exp\left(\frac{qV}{nkT}\right) - 1 \right]$$  \hspace{1cm} (2.16)

where the saturation current $J_s$ is given by

$$J_s = A^*T^2 \exp\left(\frac{-q\phi_B}{kT}\right)$$  \hspace{1cm} (2.17)

In this derivation, the following assumptions were made:

- The barrier height is far larger than $kT$
- The two current densities can be combined, one from metal to semiconductor and the other from semiconductor to metal, and do not interfere with each other.

When an applied bias is greater than $3kT/q$, the last term in equation 2.16 can be neglected and $J_n$ will only depend on $\exp(qV/nkT)$ [91]. The ideality factor is a constant with a value of 1 in the ideal situation. In practice, this value changes as a result of a number of factors. The barrier height may change when a bias is applied due to image force or interfacial layer. The image force is due to build up of charge carriers in the metal electrodes as the carriers approach metal-semiconductor interface [92]. Thus when a forward bias is applied, the barrier height tends to increase slightly, thereby causing increase in the value of $n$ from unity. Another factor that strongly influence both barrier height and ideality factor is temperature. When the device is subjected to elevated temperature, depending
on the nature of the device (in terms of surface states and deep levels), its contact and interface, it can increase or decrease the ideality factor and/or barrier height.

2.7 Charge transport in semiconductors

When a charged particle (α-particle for example) strikes the surface of a semiconductor, energy is deposited inside the semiconductor and e–h pairs are created. The number of carriers generated is proportional to the magnitude of the energy deposited. If the detector system has a configuration as the one in figure 2.14, under the influence of electric field the electrons move towards the anode and holes towards the cathode. This movement of e–h pairs created, generates a detectable signal in form of quantity of charge $Q_0$ given by

$$Q_0 = \frac{Ee}{W}$$ (2.18)

where $E$ is the absorbed energy and $e$ electronic charge. For semiconductor detectors, the absorbed energy must be greater than the band gap for e–h pairs to be created. In silicon for example, whose energy gap is 1.12 eV, an average $W$-value of about 3.6 eV is required to create an e–h pair. An elaborate discussion on this aspect of charge creation is presented in section 2.9. Figure 2.10 illustrates the relationship between energy gap and $W$-value for different materials.
In order to establish the electric field, the detector is usually biased through the electrodes; so that charge carriers are accelerated. At equilibrium, the drift velocity of the carriers depends on the strength of the local electric field. The velocity $\vec{v}$ is given by

$$\vec{v} = \mu \vec{E}$$  \hspace{1cm} (2.19)

where $\mu$ is the mobility. To achieve a signal with very low noise, a material with low conductivity is used (i.e. high resistivity) so that very low leakage current is realized. In order to achieve this, the material is sometimes doped to provide compensation of the unintended impurities in the substrate.

When a reverse bias is applied to the detector, the space charge region (SCR) becomes widened and depleted of mobile charges; building electric field in the region. Electrons and holes traverse the full depletion thickness of the detector (a process known as charge collection) at full depletion voltage. If we assume the full detector thickness to be depleted, the time taken for the charge to be collected $t_c$, is given by
2.7. CHARGE TRANSPORT IN SEMICONDUCTORS

\[ t_c = \frac{d^2}{\mu v} \]  \hspace{1cm} (2.20)

where \( d \) is the detector thickness, and the induced charge \( Q \), is given by

\[ dQ = \frac{qdx}{d} \]  \hspace{1cm} (2.21)

Equation 2.21 is for a single charge carrier. A more realistic situation is to consider a number of \( e^-h \) pairs being created within the detector volume; in which case equation 2.21 will transform to

\[ Q = \frac{qN_0}{d} [\lambda_e + \lambda_h] \]  \hspace{1cm} (2.22)

where \( \lambda_e \) and \( \lambda_h \) are the mean free paths of electrons and holes respectively; while \( N_0 \) is the number of \( e^-h \) pairs created. Equation 2.22 (which is essentially the simplified Hecht equation) is only valid if \([\lambda_e + \lambda_h] \ll d\). Thus the charges are created sufficiently far away from the electrodes. The charge collection efficiency (CCE) may be defined as the ratio of amount of charge collected to that created; expressed mathematically as

\[ CCE = \frac{Q}{Q_0} \]  \hspace{1cm} (2.23)

The CCE in equation 2.23 indicates a 100% charge collection by the detector. This is not the case with many detectors, as often there is charge trapping; which limits the distance a charge carrier can move within the detector. In that case, the CCE (which is dependent on the mean free path of charge carriers and the point where they are created) is below 100% depending on the purity of the material. This charge trapping is caused by imperfections in the semiconductor during growth, fabrication or as a result of radiation damage [76]. These defects are capable of introducing trap centres that can cause significant
change in the material properties even if they have very small concentration. The survival
time for an electron in a detector after being created before it is trapped or recombine with
a hole is its lifetime. Alternatively, the trapped carrier can be emitted later by thermal
excitation or applied bias.

Consider a charge $Q_0$ created in the detector where trapping is expected, the charge
will decay exponentially with time as

$$Q(t) = Q_0 \exp \left( \frac{-t}{\tau} \right)$$  \hspace{1cm} (2.24)

where $\tau$ is the carrier lifetime. If an external voltage is applied, a field is created causing
the drift of charge carriers with a velocity given by equation 2.19. Considering a planar
detector (see figure 2.11), the time taken to pass through a distance $x$ is

$$t = \frac{x}{v} = \frac{x}{\mu E}$$  \hspace{1cm} (2.25)

Figure 2.11: A planar detector biased with positive voltage and also irradiated from the top
electrode (anode)

After the e–h pairs are created, the time taken for the electrons to move towards the
anode is
\[ t_e = \frac{d - y}{\mu E} \]  
(2.26)

while the time taken for the holes to be collected at the cathode is
\[ t_h = \frac{y}{\mu E} \]  
(2.27)

The total charge collected may be expressed as

\[ Q = -Q_0 \int_0^{t_e} \exp \left( \frac{-t}{\tau_e} \right) \frac{\mu_e E}{d} dt - Q_0 \int_0^{t_h} \exp \left( \frac{-t}{\tau_h} \right) \frac{\mu_h E}{d} dt \]  
(2.28)

so that
\[ \frac{Q}{Q_0} = \frac{\mu_e \tau_e E}{d} \left[ 1 - \exp \left( \frac{d - y}{\mu_e \tau_e E} \right) \right] + \frac{\mu_h \tau_h E}{d} \left[ 1 - \exp \left( \frac{y}{\mu_h \tau_h E} \right) \right] \]  
(2.29)

Equation 2.29 is popularly known as Hecht [94] equation. This equation is simple and robust; it offers many options in determining transport properties of a semiconductor detector. However, the Hecht equation suffers from a number of limitations [95]

- It assumes uniformity of electric field in the detector
- The equation considers permanent carrier trapping (i.e. no detrapping)
- It assumes uniform trap distribution and hence neglects surface recombination effects

The Hecht equation may be further simplified if carriers are considered to be created very close to the irradiated surface (electrode); such that the overall charge transport depends on a single carrier (either electron or hole). One can choose the surface to be irradiated depending on the carrier of interest. Alpha particles for example, will generate carriers very close to the irradiation surface so that the depth of interaction is very small compared to mean free path of the carriers as well as detector thickness as depicted by
equation 2.22. If this condition is achieved, each carrier can be treated independently and equation 2.29 will be split into two components as follows

\[ CCE = \frac{\mu_e \tau_e E}{d} \left[ 1 - \exp \left( \frac{-d}{\mu_e \tau_e E} \right) \right] \] (2.30)

for electrons and

\[ CCE = \frac{\mu_h \tau_h E}{d} \left[ 1 - \exp \left( \frac{-d}{\mu_h \tau_h E} \right) \right] \] (2.31)

for holes. Equations 2.30 and 2.31 which are a more elaborate form of equation 2.22, can be modified to suit any type of semiconductor detector material depending on its properties. Veale et al. [96] modified the Hecht equation to include a voltage offset because of internal voltage of opposite polarity caused by polarization effects in cadmium zinc telluride (CZT). In the same vein, it is also possible to determine the thickness of depletion layer by performing C/V measurements; so that the detector thickness can be replaced with the depletion thickness.

The limitations mentioned are still valid and one has to be very careful when applying Hecht equation in detector characterization; as often modifications have to be made to have a good curve fit.

2.8 Carrier transport mechanism in semiconductors

At room temperature, electrons in a semiconductor (n-type to be precise) can wander randomly because of their average thermal energy. They can also move in three-dimensional space; thus having three degrees of freedom. When an electric field is introduced across the semiconductor, carriers move in opposite directions; holes in the direction of the electric field and electrons in the opposite direction. Acceleration of these carriers is affected by scattering arising from collision with ionised dopant and lattice atoms. The mobility of
electrons $\mu_n$; which implies the ease with which carriers move through semiconductor, is given by [70]

$$\mu_n = \frac{e\tau_c}{m_n}$$ (2.32)

where $\tau_c$ is mean free time and $m_n$ is effective mass of an electron. Equation 2.19 will only hold for low and medium applied electric fields. At very large applied fields (which depends on the material) the velocity saturates and does not depend on the electric field any more. Drift velocity in this condition is given by [97]

$$v_d = \frac{\mu_0 \varepsilon}{\left[1 + \left(\frac{\mu_0 \varepsilon}{v_{sat}}\right)^\beta\right]^{\frac{1}{\beta}}}$$ (2.33)

where $\beta$ is a fitting parameter with values of 1 and 2 for holes and electrons respectively [98]. From equation 2.33, $v_d = \mu_0 \varepsilon$ as $\varepsilon \to 0$ and $v_d = v_{sat}$ as $\varepsilon \to \infty$.

As stated earlier in this section, the mobility of carriers in a doped semiconductor can be affected by scattering mechanisms, the most prominent processes are lattice and ionised impurity scatterings. At room temperature and above, the lattice periodic potential is distorted allowing transfer of energy between the carriers and lattice. This energy transfer increases with increasing temperature. Thus at high temperatures, lattice scattering is favoured. As a result of coulomb interaction, a charge carrier may be deflected as it passes an ionised dopant impurity. But at higher temperatures, the carriers experience large kinetic energy, such that they can move faster and escape deflection. Using the Mattheissen rule [99], it is possible to show that

$$\mu = \left(\frac{1}{\mu_l} + \frac{1}{\mu_i}\right)^{-1}$$ (2.34)

Equation 2.34 represents the mobility due to the two scattering effects. i.e. $\mu_l$ and $\mu_i$ for lattice and ionisation scattering respectively. However, it is worth to note that impurity
concentration is the limiting value for impurity scattering dependence on temperature. Figure 2.12 summarises the effect of temperature on the two scattering processes.

![Figure 2.12: Mobility as a function of temperature (theoretical framework) adapted from Ikeda et al.[100].](image)

From the figure, it can be seen that at a temperature midway between highest and lowest values, the two scattering phenomena could nullify each other in some cases out. This will lead to dependence of mobility only on ionisation impurity scattering. Therefore, this temperature is the equilibrium temperature.

### 2.9 Shockley-Ramo Theory

When the surface of a planar semiconductor detector is irradiated, e-h pairs are created. Depending on the polarity of the applied bias either electrons or holes are attracted towards the anode. The movement of these charge carriers which is influenced by electric field induces charge or current on either sides of the detector (see figure 2.13).
The number of e-h pairs created depends on the specific average energy required in that material to generate e-h pairs also known as W-value; which is an inherent property of the material as depicted in figure 2.10.

The signal formation in a semiconductor detector under bias is best explained by employing the theory developed in the late 1930’s by Shockley [101] and Ramo [102]. The theory which was formulated originally for vacuum tubes has been adopted for semiconductor radiation detectors [103, 104, 35]. A detailed discussion on the Shockley-Ramo theorem has been presented by He [105]. The theorem states that a moving point charge induces charge $Q$ and current $i$ given by

\[
Q = -q\psi_0(x) \tag{2.35}
\]

\[
i = q\vec{v} \cdot \vec{E}_0(x) \tag{2.36}
\]
where $q$ is the charge moving with instantaneous velocity $\vec{v}$. $\psi_0(x)$ and $\vec{E}_0(x)$ are the weighting potential and field respectively at point $x$ of the moving charge $q$. The weighting field is defined by the Laplace equation as

$$\vec{E}_0(x) = \frac{\partial \psi_0(x)}{\partial x} \tag{2.37}$$

This weighting potential is different from the actual electric potential in the detector which determines the trajectory of charge $q$, but it makes calculation of induced charge $Q$ more convenient [105]. From the Laplace equation which was used to arrive at equation 2.37, the voltage on the electrode of interest is taken as unity and the voltages on all other electrodes are taken as zero.

A simple illustration is a planar detector made from an intrinsic material placed between two electrodes (Figure 2.14) (one at the top and the other at the bottom). Since the potential is linear from 0 (at the cathode) to 1 V (at the anode) assuming the thickness of the detector is much smaller than its lateral dimension, the charge $Q$ induced on the anode due to the generation of charge carriers at point $Z$ (see Figure 2.15) can be determined from equation 2.35; so that

$$Q = -q\Delta \psi_0(x) \tag{2.38}$$

$$= -me \left[ \psi_0(0) - \psi_0(z) \right]_{holes} - m(-e) [1 - \psi_0(Z)]_{electrons} \tag{2.39}$$

$$= me \tag{2.40}$$

where $e$ is the electronic charge and $m$ is the number of charge carriers.
2.9. SHOCKLEY-RAMO THEORY


Figure 2.15: Weighting potential at different zones through the volume of the detector [105]. Reprinted from Nuclear Instruments and Methods in Physics Research A, 463, He, Z., Review of the Shockley–Ramo theorem and its application in semiconductor gamma-ray detectors, 250–267, ©2001, with permission from Elsevier.

On the other hand, if the mean free path of holes is much smaller than that of electrons (as is the case in CdZnTe), only a fraction of the holes will be collected and this will result in values of induced charge ranging from $me$ to zero as shown in equation 2.41.

$$Q \approx me \left[ 1 - \psi_0(z) \right]$$  \hspace{1cm} (2.41)
CHAPTER 2. PRINCIPLES OF SEMICONDUCTOR RADIATION DETECTOR

Having discussed the essential general characteristics of the semiconductor detectors from charge creation to signal production, in the proceeding sections specific properties of silicon and silicon carbide detectors are presented.

2.10 Silicon

Silicon is a non-metallic group four element with atomic number 14 and relative atomic mass 28. It is a major part of clay, granite, quartz and sand. It is produced commercially by reacting SiO$_2$ and carbon at about 2200 °C [69]. It has a cubic diamond-like structure with energy band gap of 1.12 eV and can be easily doped in to p-type (B, Ga acceptors) or n-type (P, As, Sb donors) [106]. It is a face centred cubic structure crystal [107].

2.10.1 Synthesis

To date many techniques have been developed for the production of Silicon detectors. Previous studies have based their choice of technique on residual oxygen concentration, purity, resistivity and the magnitude of depletion voltage [108]. In most recent studies, Silicon is produced basically in two different ways; Float zone (Fz) and Czochralski (Cz) techniques [106]. The Cz-method is commonly employed in the production of highly integrated low power devices, while Fz is for high power devices [109]. It appears that no single method can provide a material that will satisfy all the needs of radiation detector, because each method has its advantages and disadvantages. With the Fz-technique, high purity and high resistivity material can be produced but, low oxygen concentration is a disadvantage because it has been proven [110, 111] that radiation hardness of silicon detector can be improved by increasing its oxygen concentration. On the other hand, with Cz-technique a high oxygen concentration and high substrate diameter is possible, while compromising the quality [108]. In the following discussion a summary of each technique is presented.
Czochralski technique

In this method a high purity material (PPM impurity levels) is melted in a crucible (mostly made of quartz). A seed crystal is used to touch the molten silicon and the ingot is pulled by withdrawing the seed in a slow rate. To have a homogeneous material, the crucible is rotated at a speed of about 2 to 50 rpm (rotations per minute). Adjustments are made in the temperature gradient to enable crystal growth on the surface of the seed. In this process, crystals with 50 cm diameter can be obtained (Figure 2.16). The most important impurity in this process is interstitial oxygen whose concentration increases at the end of the process [73]. Material uniformity is largely affected by the thermal gradients inside the crucible melt [111]; which in the long run reduces the crystal perfections. Similarly, because of large concentration of dopants in the silicon melt, the resistivity of the material is reduced by increasing the length of the ingot. Summing up all these factors, it can be seen that the major limitation of material grown based on this technique is resistivity.

Figure 2.16: Schematic diagram of Cz-Si crystal puller used for large crystal growth [109]. Reprinted from Materials Science and Engineering: B, 73/1–3, Zulehner, W., Historical overview of silicon crystal pulling development, 7–15, ©2000, with permission from Elsevier.
Float zone technique

In the Fz method, a contact is created between the molten silicon and (solid) crystal silicon which allows diffusion of impurity atoms by segregation. Since the impurities are more concentrated in the liquid phase, a small boule of silicon is allowed to pass through a very narrow part of the furnace in a slow rate. This slow movement restricts the quantity of boule to be melted at any time. Since the impurities are in the liquid phase, they will migrate as the boule is moved towards the heat zone. This narrow part is heated by a coil which forms a narrow molten zone. Displacement of the molten zone is achieved by moving the coil upward and it is then followed by solidification (Figure 2.17). As soon as this is actualized, a single crystal ingot is obtained [75].

![Schematic diagram of a modern Fz-Si crystal puller used for large crystal growth](https://example.com/figure217.png)

**Figure 2.17:** Schematic diagram of a modern Fz-Si crystal puller used for large crystal growth [109]. Reprinted from Materials Science and Engineering: B, 73/1–3, Zulehner, W., Historical overview of silicon crystal pulling development, 7–15, ©2000, with permission from Elsevier.

It is worth noting that, Silicon produced using this method is limited by size. However, looking at the limitations of both techniques, two other techniques were established as improvements to the Cz-method, the magnetic Czochralski (MCz) method and electromagnetic Czochralski (EMCz) method. The MCz-technique is in principle similar to Cz-technique, but in the MCz-technique the growth of the ingot takes place under the influ-
ence of a strong magnetic field. The magnetic field assists in controlling the concentration and distribution of oxygen by reducing the swaying in the melt.

Harkonen et al. [108] compared I/V and C/V characteristics of MCz-Si and FZ-Si, both having area of 32.5 cm$^2$. In his result, both I/V and C/V characteristics of 380 $\mu$m MCz-Si and 500 $\mu$m Fz-Si measured at room temperature were comparable. The leakage current observed from MCz-silicon at 900 V is 3 $\mu$A and the full depletion voltage is 420 V. As for the Fz-silicon, the leakage current is 1.2 $\mu$A at 1000 V and 320 to 340 V full depletion voltage. This indicates an improvement in MCz-Si detector, which is primarily due to the reduction in impurity concentration. This result is in agreement with that of Messineo [112] who observed that, leakage current density shows no significant difference between MCz and Fz silicon materials.

A more advanced method (electromagnetic Czochralski technique) was developed by Watanabe et al., [113]. In this method, a higher concentration of oxygen and even distribution within the sample is achieved by the use of an electromagnetic field (EMF). The principle used was based on Fleming’s left hand rule so that an electromagnetic force is created(Figure 2.18). The oxygen concentration and distribution in the EMCz was found to be more uniform when compared to the substrates produced by MCz-method. This is because, the EMCz has two components of fields which control the concentration and distribution of oxygen in the vertical and horizontal directions; thus having a crystal with quality better than that of MCz-silicon.
2.10.2 Radiation Hardness

A large number of research works have been carried out on silicon radiation hardness and this report does not intend to capture all but the most relevant findings. Radiation hardness for radiation detectors implies the ability of the material to give a good (reasonable) signal after it has been exposed to a large dose of ionizing radiation. This radiation hardness can be affected by surface and/or bulk damage.

In the case of surface damage in silicon, the radiation causes positive electric charge to be trapped in the passivation layer; while in silicon devices electron hole pair recombination or migration may take place [114]. The probability of immediate recombination is very high especially in the absence of an electric field, and the trapping causes an increase in the oxide space charge and introduces surface generation centres that will normally increase the noise and power consumption [115].

Bulk damage on the other hand, causes displacement damage such as vacancies and
interstitials which may come together to form more stable defects [115]. As a result of Coulomb’s interactions, protons and pions produce more point-like and cluster defects than electrons and gamma-rays [114, 115]. Radiation hardness of silicon can be improved by oxygenating the silicon material [111]. But this hardness depends on the nature and/or type of radiation involved.

In trying to investigate the radiation hardness of oxygenated silicon material, Dezillie et al. [116] irradiated silicon materials (wafer thickness 188, 270 and 400 µm and resistivity 1.1 to 5.6 kΩcm) with neutrons, protons and gamma radiations. Full depletion voltage ($V_{fd}$) was independent of $\gamma$–radiation fluence (up to approximately 600 Mrad) for oxygenated silicon, but for standard silicon high dependence of $V_{fd}$ was observed as well as type inversion at a fluence of 250 Mrad. In the same vein, CCE approaches 100 % at 40 V in the former while in the latter the CCE approaches the same value at about 60 V. In the case of proton irradiated samples, type inversion (change in the sign of effective doping density) was observed in both samples at approximately $9 \times 10^{13}$ p/cm$^2$. However, the slope ($V_{fd}$ against proton fluence) for oxygenated Silicon was 2.3 times less steep than that of the standard silicon. As for neutron irradiated samples, no significant difference was observed. This result is not sufficient, in that only one sample (5.6 Ωcm) was used in the investigation of full depletion layer inversion. Also in the study of proton irradiation, a sample with medium resistivity (1.2 to 3 kΩcm) was used. Similar findings were also reported by Moll [117]. The studies conducted by Casse et al. [114] and Lindstrom et al. [118] showed a slight improvement in the slope of $V_{fd}$ against proton fluence (i.e. 3 times less steep than that of standard Silicon) for proton irradiation.

More conclusive research was carried out by Moll et al. [119] and the ROSE collaboration of the CERN RD 48 project. The research utilized samples of silicon materials that cut across a wider range of properties; in terms of production mechanism, oxygen concentration and resistivity values which aided in having a more comprehensive understanding
of the effect of oxygen concentration on silicon radiation hardness. Oxygenated Silicon showed high radiation tolerance from Gamma irradiation compared to standard silicon, while type inversion was still observed in all the samples irradiated with protons. The information regarding correlation between microscopic (point) and macroscopic detector properties is still pending.

2.11 Silicon carbide

Due to the envisaged improvement in technology at the Large Hadrons Collider (LHC) to very high luminosity colliders with expected hadron fluences of about $10^{16}$ cm$^{-2}$ or greater, CERN approved the RD50 project [120]. The project focused on three major areas which include material engineering, device engineering and variation of detector operational conditions. Furthermore, it was realised that the high oxygen concentration silicon (Cz-Si) has to be treated with caution to avoid changes in thermally activated donor concentration thereby changing the net donor concentration. The most important achievement of the group was proposing new materials that can replace silicon. The new materials include silicon carbide and semi-insulating gallium nitride.

Silicon carbide (SiC) is one of the very important candidates for radiation detection materials. SiC based devices are capable of operating under extreme conditions at high temperatures, under harsh radiation conditions, or in chemically active media. This is because it has high electric and mechanical strength, thermal and chemical stability as well as radiation resistance [121]. These characteristics show that SiC is a great candidate for radiation detection in harsh environments. SiC forms a protection silicon oxide layer which protects it from extra oxidation at very high temperatures and in oxidizing atmospheres [122].
2.11. SILICON CARBIDE

2.11.1 Crystal structure and poly-types

SiC is formed from silicon and carbon with equal proportions. Therefore, for each silicon atom there exist four carbon atoms and vice versa. This type of configuration is depicted in figure 2.19.

Figure 2.19: Carbon atom (white) at the centre of mass bonded to four silicon atoms (black) in a tetragonal structure.

According to the figure, the distance between each carbon atom and silicon (a) is the same but the height (c) differs from one polytype to another. The structure of SiC crystal could either be zinc-blend (cubic) or Wurtzite depending on its stacking order. This difference in stacking order is what gives birth to different structures called polytypes. To illustrate the atomic ordering of SiC structures, it is possible to have the first layer of atoms arranged in a closed packed structure as seen in figure 2.20.
If the first layer is named as A, the next layer will be where the atoms centred between three atoms, thus forming covalent bond. This will make it possible for each atom to have four nearest neighbours with four directional bonds. The third layer of silicon and carbon can now be placed directly on the first layer. If this pattern is repeated for every other bi-layer, the resulting structure will be Wurtzite [124]. On the contrary, the third layer could be placed in a position different from the first and second resulting to a cubic structure. By giving these bi-layers names A, B and C as shown in figure 2.20, the stacking sequence can be used to distinctly identify each structure based on the combination of the stacking feature as shown in figure 2.21.
2.11. SILICON CARBIDE

As shown, a stacking of ABAB will represent hexagonal (2H) because this stacking feature is repeated just after stacking two bi-layers. For cubic structure (3C), the stacking pattern is repeated after three bi-layers. Also it is worth pointing out that both the three bi-layers were having different placement, thus ABC. Other structures are 4H and 6H polytypes. Although there is the possibility of having many polytypes (more than a hundred) [124], not all of them are stable single crystals; implying that for device manufacture, only a limited number of them are suitable. The only commercially available polytypes are 4H and 6H polytypes [125]. These commercially available structures have two faces perpendicular to the c-axis; Si (0001) and C (000\(_1\)). The Si face is more suitable for epitaxial growth because it is mostly the polished side of the SiC wafer [125].

2.11.2 Properties

SiC exhibits excellent electrical properties which are highly appropriate for radiation detection which include high breakdown electric field, good mobilities and high saturated
electron drift velocity (three times higher than that of Si) [30]. The large band gap (3.0 to 3.5 eV) energy produces very low dark currents and allows operation at high temperatures [30, 126, 127]. In addition SiC has very high thermal conductivity (350 to 490 Wm⁻¹K⁻¹) that is also three times higher than that of Si and seven times higher than GaAs [126, 128]. There is a growing increase in the demand of improved radiation detectors as a result of the urgent need to reduce threat as well as nuclear non-proliferation activities. Conventional semiconductor devices fail to function adequately under high power, high frequency, high temperature, high switching speed and harsh radiation environments [126]. The most common poly-types of SiC are Hexagonal (6H-SiC), Cubic (3C-SiC) and Rhombohedral (15R-SiC). Of all the types mentioned above, only 4H and 6H single crystals are available commercially [129, 130]. 4H is more readily available; it is predominantly supplied by Cree in form of SI 4H-SiC [30]. Table 2.2 gives a summary of the main properties of SiC in comparison to other wide band gap (WBG) semiconductors.

<table>
<thead>
<tr>
<th>Property</th>
<th>Si</th>
<th>GaAs</th>
<th>3C-SiC</th>
<th>4H-SiC</th>
<th>6H-SiC</th>
<th>GaN</th>
<th>Diamond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Band gap, E_g (eV)</td>
<td>1.1</td>
<td>1.4</td>
<td>2.40</td>
<td>3.26</td>
<td>3.02</td>
<td>3.4</td>
<td>5.5</td>
</tr>
<tr>
<td>Dielectric constant, ε_r</td>
<td>11.8</td>
<td>12.8</td>
<td>9.72</td>
<td>9.66</td>
<td>9.66</td>
<td>9</td>
<td>3.5</td>
</tr>
<tr>
<td>Thermal conductivity, λ (W/cmK)</td>
<td>1.5</td>
<td>0.5</td>
<td>3.2</td>
<td>3.7</td>
<td>4.9</td>
<td>1.3</td>
<td>20</td>
</tr>
<tr>
<td>Electron mobility, μ_e(cm²/V-s)</td>
<td>1400</td>
<td>8500</td>
<td>800</td>
<td>1000</td>
<td>400</td>
<td>900</td>
<td>2200</td>
</tr>
<tr>
<td>Hole mobility, μ_h(cm²/V-s)</td>
<td>600</td>
<td>400</td>
<td>40</td>
<td>115</td>
<td>101</td>
<td>150</td>
<td>1600</td>
</tr>
<tr>
<td>Breakdown voltage (MV/cm)</td>
<td>0.3</td>
<td>0.4</td>
<td>1.5</td>
<td>3.2</td>
<td>3.0</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>Electronic maximum operating temperature °C</td>
<td>150</td>
<td>350</td>
<td>700</td>
<td>700</td>
<td>700</td>
<td>&gt;750</td>
<td>1000</td>
</tr>
<tr>
<td>Melting point (K)</td>
<td>1690</td>
<td>1510</td>
<td>3100</td>
<td>3100</td>
<td>3100</td>
<td>2070</td>
<td>&gt;2500</td>
</tr>
<tr>
<td>Physical stability</td>
<td>Good</td>
<td>Fair</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Good</td>
<td>Very good</td>
</tr>
</tbody>
</table>

Table 2.2: Comparison of some important wide band gap (WBG) semiconductor properties for HTE [13].

2.11.3 Synthesis

SiC unlike Silicon, cannot be produced from melt because it sublimes before melting at about 1800 °C and may also dissociate [131]. The first industrially based method of silicon carbide synthesis was developed by Lely in 1955; by using the vapour phase through a sublimation technique [131, 132] which is limited by the high temperature required to
2.11. SILICON CARBIDE

grow the crystal (2600 to 2700 °C), deficiency of control over the nucleation, very small sizes (about 2 to 5 mm) and doping non-uniformity. Tairov and Tsvetkov [132] proposed a better technique which is an improvement over Lely’s method normally called Physical Vapour Transport (PVT). The method was based on a classical scheme of supersaturated vapour condensation which aided in the studies of mass transfer and growth kinetics of SiC. The improved method was able to reduce growth temperature to 1800 °C with further reductions in boron impurity concentration to 1/4 of the initial system. The ingot produced was about 8 mm long and 8 mm in diameter.

Similarly, Schulz et al. [133] reported SiC growth temperatures and pressures of 2010 to 2100 °C and 300 to 5000 Pa respectively. Although open volume defects were observed on the surface of the grown epitaxial layer, it was observed that nitrogen plays a significant role in stabilising 4H-SiC growth. Further improvement in the growth technique produced 6H-SiC bulk crystals with about 35 to 40 mm diameter [134]. In this improved technique the temperature range was 2150 - 2350°C and the growth rate 200 to 250 µm/h. Various growth diameters were also reported by many other authors; 50 mm [135, 136] and 60 mm [137].

Currently the only two ways of SiC production are Bulk and Epitaxial growth with wafer thickness of (100 to 500 µm) and 50 µm respectively [30]. The latter will provide good performance in radiation detection but is limited by commercial availability; while the former has good radiation hardness but suffers from polarisation and crystal defects.

Like other semiconductors, doping is capable of improving radiation detection properties of SiC. Vanadium is one of the major elements used in the doping which can increase the resistivity of SiC [30]. Bickermann et al. [138] investigated the effect of incorporating Nitrogen, Boron and Vanadium as doping impurities during a PVT growth of SiC. Results show a decrease in charge carrier concentration with growth time, while in the Boron doped samples charge carrier concentration increases with volume fraction. The Vanadium doped...
crystals have non-uniformity in doping concentration with growth time. Further research is expected to improve the uniformity of the Vanadium concentration.

The common defects affecting bulk SiC are inclusions, voids and micropipes [30]. There have been attempts to reduce or completely remove micropipes. Research conducted by Shiomi et al. [139] reported the growth of micropipe free crystals of epitaxial 4H-SiC by the use of a conventional sublimation method. The growth conditions were improved (temperature 2100 to 2300 °C and pressure 1.3 to 2.6 kPa) and a high purity seed was used. X-ray topographic images of the grown crystal showed absence of micropipes and stacking faults; which are normally present in conventionally grown samples.

A different attempt was made by Filip et al. [140] to heal the micropipes in a SiC wafer by adding small concentrations of germanium (Ge) utilizing the Liquid Phase Epitaxy (LPE) technique. Growth temperatures and pressures were up to 2300 °C and 200 bar respectively. About 80 % of micropipes with 5 µm were healed while some decomposed into other forms of dislocations. Scanning electron microscopy (SEM) confirmed that the process did not create any distortion in the crystal. Adding Ge in a different passion (considering different conditions) may further heal the micropipes in SiC to a larger extent.

2.11.4 Radiation Hardness

As a particle detector, SiC performance can be assessed by its CCE as a function of reverse bias voltage [129]. The properties expected from an ideal semiconductor detector for application above room temperature and high radiation fluence comprise of large band gap energy, small electron-hole pair generation energy, low dielectric constant, low defect concentration, high atomic displacement energy and high thermal conductivity among others[29], but this cannot be realized in real materials. Most importantly, e–h pair formation energy increases with increasing energy band gap and a trade-off has to be made.

Several authors examined the performance of SiC for radiation detection applications.
The common methods are characterization (electrical and optical) including spectroscopy techniques.

Katulka et al. [141] observed improvement (about 100%) of conductivity of a SiC detector, after being implanted with Ge. As for the annealed and as-implanted samples, no difference was detected; which requires further investigation. Raman spectroscopy revealed a distorted signature plot for the as-implanted SiC in comparison to the annealed one and, fluorescence of the damaged material surface.

With respect to performance of SiC in high radiation fluence, Cunningham et al. [8] reported a strong increase in free carrier concentration when the sample was irradiated with 300 MeV $10^{12}$ pions cm$^{-2}$ and a sharp decrease for irradiations with higher fluencies (i.e. $10^{13}$ and $5 \times 10^{14}$ pions cm$^{-2}$). The same situation was also observed in the resistivity of the material, but the literature could not provide a reason for this anomaly. Certain persistent noise in most of the figures presented, were not accounted for. Choosing other fluencies of different intervals could reveal more information about the abnormality.

However, attempts to improve the radiation detector efficiency, show that a 100% CCE is possible with a 4H-SiC epitaxial layer deposited on a 4H-SiC substrate using a $^{90}$Sr $\beta$-source at 400V; with negligible trapping and de-trapping [7].

A comprehensive investigation was carried out by Nava et al. [142], where electrons (8.2 MeV), protons ($10^{14}$ cm$^{-2}$) and Gamma-rays (0 to 40 Mrad) were used to irradiate 4H-SiC detectors. A collective behaviour of all the diodes was observed in terms of decrease in effective doping concentration ($N_{eff}$) and reverse current with increasing dose. The author believes that the deep centres formed by electron and proton irradiations might have caused the reduction in the $N_{eff}$. This idea is subject to further investigation. Samples irradiated with 5.48, 4.14 and 2.0 MeV $\alpha$-particles exhibit 100% CCE irrespective of their crystallographic quality.
2.12 Defects in semiconductors

No matter how good a material is produced, due to the inherent properties of the precursors or the method of production, imperfections cannot be completely avoided. In fact due to the dangling bonds, the surface of a crystal is considered as a defect itself. The defect in a material at atomic scale can be one dimensional, two dimensional or three dimensional. These defects can affect physical properties of materials such as resistivity of a semiconductor. This resistivity depends largely on atomic impurities present in a semiconductor. The two most important defects considered here are point and surface defects.

2.12.1 Point defects

When an atom is missing in a lattice, it creates a defect known as Schottky defect [143]. This missing atom will move up to the surface of the crystal. Although the material may be in thermal equilibrium, because of the imperfections that increased the entropy, lattice vacancies will always be present. On the other hand, an atom of the material may be transferred from its lattice site to a location not normally occupied by an atom, popularly called interstitial location [144]. This is a self interstitial but the interstitial atom could be different from parent atom; in which case it is called interstitial impurity. There could be the more complex situation in which, the atom moves from its original position in the lattice to a normally vacant site; thereby leaving a vacancy in the initial position. This forms a pair of interstitial – vacancy defect otherwise known as Frenkel defect [145]. Another case may be a substitutional impurity where, an atom of different material substitute the position of an initially occupied position by the parent atom. All these situations are capable of changing the electrical conductivity of a semiconductor material [144]. Sometimes, these changes occur in an uncontrolled manner and they are called simply impurities and defects but, when they are applied in a controlled manner,
they are referred to as dopants. Thermal defects or radiation damage could result to interstitial atoms and, they can also introduce vacancies [146]. Formation of antisites is a possibility in a compound material. If a material is made up of two elements for example, say AB, the atom A can occupy B position due to unequal chemical proportions [147].

2.12.2 Surface defects

As discussed earlier in section 2.12, the finite nature of a crystal is a potential source of surface defects and, these defects could be electronic or vibrational [146]. Due to the difference in configuration of these atoms compared to the ones inside the crystal, their contribution to the properties of a material is different. Scattering of electrons and phonons can take place at a crystal’s surface [148].

The surface defects may be acceptors or donors resulting from dangling bonds at the surface of the semiconductor [149]. In addition, a contaminated surface can give rise to additional surface defects. The properties of the semiconductor could be chemically modified when exposed to atmosphere or other environments and, this change is capable of causing carrier trapping and band deformation [150].

The surface states created by surface defects may act as [150]

- **Traps**: these traps can attract carriers which could result to charging a device

- **Recombination** centres: Effect of these centres is evident from reduction of carrier density in steady state condition.

- **Scattering** centres: the centres are well known for their influence in reducing mobility of carriers in semiconductors.

All the aforementioned defects will in one way or the other affect the performance of a semiconductor device. As a way of mitigating these effects, passivation, barrier formation
and encapsulation are important measures [150]. As for surface engineering, it is not yet clear how it can be beneficial towards improving device performance. Therefore, this area requires more investigation.

2.12.3 Deep and shallow levels

In principle, the band gap of a semiconductor is a region devoid of energy states and thus it clearly differentiates between conduction and valence bands. By intentionally adding foreign atoms in a controlled manner (doping) or unintentionally introducing other atoms (impurity), which can take place during synthesis, one can easily introduce some states within the forbidden band region. Electrically active levels with very low binding energy (typically $10^{-2}$eV) are considered to be shallow levels [151]. On the other hand, if the binding energy is large compared to the band gap, it is classified as deep level.

2.12.4 Emission and capture of carriers from defect centres

Due to traps present in a semiconductor, carriers can either be captured or released from the traps. This capture and release depends on a number of factors, which include the electric field, temperature and nature of the trap itself. A detail discussion on the kinetics of emission and capture of carriers has been presented by Shockley [152], Hall [153] and Bourgoni [154]. Here, the case of a degenerate semiconductor with two charge states is presented. This approach is identical to that of Bourgoni [154]. Consider the states to be labelled A and B with A having an excess electron, four situations are possible [155]

- emission of electrons to conduction band with rate given as $e_n$
- emission of holes to valence band with rate given by $e_p$
- capture of electrons or holes from conduction or valence bands respectively with capture rates represented by $C_n$ and $C_p$
These processes take place in competition with one another. For simplicity, it is assumed that the electrons do not have to cross any barrier, for an electron to be captured by state B, the capture probability $k$ is given by

$$k_n = \sigma_n v_{th,n} n$$  \hspace{1cm} (2.42)

Similarly, the probability for holes is

$$k_p = \sigma_p v_{th,p} p$$  \hspace{1cm} (2.43)

where $\sigma_{n,p}$ and $(n,p)$ are the capture cross-sections of defects and density of carriers respectively. $v_{th,n,p}$ is the thermal velocity for carriers. For electrons, the thermal velocity is given by \[99\]

$$v_{th,n} = \left[ \frac{8kT}{\pi m^*} \right]^{1/2}$$  \hspace{1cm} (2.44)

The emission rates $e_n$ and $e_p$ will both be independent of the carrier concentration if and only if the carrier concentration is far less than the density of states in both conduction and valence bands. Let $a$ and $b$ represent defect concentrations in A and B states respectively, so that the rate of emission and capture of electrons is

$$k_na = C_n na \Rightarrow electron - capture$$  \hspace{1cm} (2.45)

$$e_nb \Rightarrow electron - emission$$  \hspace{1cm} (2.46)

and that of holes can be

$$k_pb = C_p pb \Rightarrow hole - capture$$  \hspace{1cm} (2.47)

$$e_pa \Rightarrow hole - emission$$  \hspace{1cm} (2.48)
Equations 2.45 and 2.46 are equivalent at thermal equilibrium. The same argument goes for equations 2.47 and 2.48. This gives

\[ e_n = \sigma_n v_{th,n} n' \frac{a'}{b'} \quad (2.49) \]
\[ e_p = \sigma_p v_{th,p} p' \frac{b'}{a'} \quad (2.50) \]

Also, the ratio of concentration of the states is given by

\[ \frac{a'}{b'} = \gamma \exp\left[\frac{E_t - E_F}{kT}\right] \quad (2.51) \]

where \( \gamma \) is the degeneracy factor. The carrier densities at equilibrium are represented by

\[ n' = N_c \exp\left[\frac{E_t - E_c}{kT}\right] \quad (2.52) \]
\[ p' = N_v \exp\left[\frac{E_v - E_F}{kT}\right] \quad (2.53) \]

By concentrating on electrons only and using equations 2.51, 2.49 and 2.52, it is possible to show that

\[ e_n = \frac{2}{\pi^2 \hbar^3} \sigma_n m^* k^2 T^2 \frac{E_t - E_c}{\gamma} \exp\left[\frac{E_t - E_c}{kT}\right] \quad (2.54) \]

Therefore, an Arrhenius plot of \( e_n / T^2 \) as a function of \( 1/T \) should produce a linear relation from which the activation energy \( E_t - E_c \) of the trap and its cross section can be evaluated.

### 2.13 DLTS: Deep Level Transient Spectroscopy

DLTS is a method invented by Lang [156]; which is used to assess defects in a semiconductor material. As the name implies, it is used to determine electrical properties of deep level defects. A number of methods have been used to study deep levels in semiconductors. Such
techniques include thermally stimulated capacitance and thermally stimulated current. These methods are largely applied to dielectric and large resistivity materials. These techniques depend strongly on how precise the rate of change of temperature is controlled. Also, the sensitivity is very low [157]. DLTS is one of the oldest techniques used mostly for detection of deep level traps. Its sensitivity to detect traps with $10^{-3}$ cm$^{-3}$ or lower impurity concentration is of great importance and does not depend on the emission time constant [84]. The magnitude of its spectroscopic peaks could be easily used to differentiate minority from majority carriers [157]. However, this method cannot be used to characterise high resistivity materials [84].

2.13.1 DLTS technique

The technique utilises capacitance transients produced as a result of thermal excitation of carriers trapped within the depletion layer of a reverse bias diode, Schottky barrier or metal oxide semiconductor [157]. The proceeding discussion will concentrate on an n-type Schottky device. A sensitive and fast capacitance meter is used to measure the capacitance of the sample. A change in capacitance of the sample is caused by emission of carriers which is measured according to [158]

$$N(t) = N_T e^{-e_{nt}}$$ (2.55)

where $N_T$ and $N$ are concentrations of defect level and occupied defects respectively. A typical DLTS cycle starts with a small forward bias (filling pulse) applied across the semiconductor; which will reduce the thickness of its depletion layer and increase the capacitance. This reduction in depletion fills up the traps to a length $x$ close to the depletion thickness below the semiconductor surface (see figure 2.22B)
When the traps are filled, reverse bias is applied which will increase the depletion thickness, so that the device will return to a condition similar to the initial stage in (A) as shown in figure 2.22C. By implication, the capacitance will be reduced by a small amount. However, since some traps in the depletion region are filled up, the charge density in this state is a bit lower than in the initial state.

DLTS signal is usually recorded in pF. While the forward and reverse bias are applied, they are accompanied with temperature variation via a temperature controller. The signal can be represented as a function of capacitance as follows

\[
DLTS = a [C(t_2) - C(t_1)]
\]  \hspace{1cm} (2.56)

According to equation 2.56, if the temperature applied (in the cause of ramping up) is very low that at time \( t_2 \) no significant emission has occurred, the result is zero. Similarly, if the temperature is too high that the emission has already taken place at time \( t_1 \) no signal will be recorded. DLTS peaks will appear only if the emission rate falls within a rate window defined by \( t_1 \) and \( t_2 \). The condition that has to be satisfied for a peak to appear is given.
by

\[ e_n = \frac{\ln \frac{t_2}{t_1}}{t_2 - t_1} \quad (2.57) \]

From equation 2.54, an Arrhenius plot of \( e_n/T^2 \) against \( 1/T \) will give an estimate of the trap energy and its associated cross section.
Chapter 3

Experimental techniques and equipment

3.1 Introduction

Oxygen has been shown to improve radiation tolerance of silicon detectors [111]. Magnetic Chzochralski (Cz)-Silicon naturally contains oxygen considering its process of production which is different from standard Float zone (Fz)-Silicon (this has been discussed in section 2.10). The CERN RD48 collaboration has considered MCz-silicon for high radiation fluence application in large hadron collider (LHC) experiments but, unfortunately at that time Cz-material with sufficient resistivity was not available [110]. The samples under investigation have been produced by utilizing the oxygen diffusion method. It is thus expected that these devices will be more radiation resistant than non-oxidized ones. On the other hand, silicon carbide (SiC) has been considered as a substitute for silicon because of its hardness and large band gap. The former feature makes it suitable for radiation hard detection while, the latter imply its application for high temperature environments.
3.2 Materials and devices

3.2.1 Float zone (Fz) and Magnetic Chzochralski (MCz) silicon

Silicon detectors used in this project were provided by the IAEA-CRP\textsuperscript{1} collaboration which involves other groups around Europe, Asia, Australia and America. The information received from the manufacturers of the 11 devices is presented in table 3.1, consisting of 2 n-MCz-silicon, 2 n-Fz-silicon and 7 p-Fz-silicon (n- and p- denote the doping type) devices. The range of operating voltage for the n-MCz-silicon diodes is $\approx 300\text{V}$ while for the Fz-silicon one is $\approx 50\text{V}$. The devices have an active area of 0.7 by 0.7 cm\textsuperscript{2} and a thickness of 652 $\mu\text{m}$.

<table>
<thead>
<tr>
<th>Surrey no.</th>
<th>Wafer label</th>
<th>IAEA no.</th>
<th>Nominal Depletion Voltage $V_{fd}$ (V)</th>
<th>Leakage current $I_{lc}$ (nA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>n-MCz Si-2009</td>
<td>14</td>
<td>N/k</td>
<td>N/K</td>
</tr>
<tr>
<td>2</td>
<td>n-MCz Si-2009</td>
<td>N/K</td>
<td>$\approx 350$</td>
<td>$\approx 0.9$</td>
</tr>
<tr>
<td>3</td>
<td>n-FzSi-2012 Wafer 1</td>
<td>10</td>
<td>N/K</td>
<td>N/K</td>
</tr>
<tr>
<td>4</td>
<td>n-FzSi-2012 Wafer 2</td>
<td>39</td>
<td>N/K</td>
<td>N/K</td>
</tr>
<tr>
<td>5</td>
<td>p-FzSi-Diode2011C1</td>
<td>8</td>
<td>$\approx 51$</td>
<td>$\approx 7.9$</td>
</tr>
<tr>
<td>6</td>
<td>p-FzSi-Diode2011C1</td>
<td>9</td>
<td>$\approx 51$</td>
<td>$\approx 7.1$</td>
</tr>
<tr>
<td>7</td>
<td>p-FzSi-Diode2011C1</td>
<td>10</td>
<td>$\approx 48$</td>
<td>$\approx 6.4$</td>
</tr>
<tr>
<td>8</td>
<td>p-FzSi-Diode2011C1</td>
<td>11</td>
<td>$\approx 45$</td>
<td>$\approx 6.6$</td>
</tr>
<tr>
<td>9</td>
<td>p-FzSi-Diode2011C1</td>
<td>12</td>
<td>$\approx 48$</td>
<td>$\approx 6.6$</td>
</tr>
<tr>
<td>10</td>
<td>p-FzSi-Diode2011C1</td>
<td>14</td>
<td>$\approx 51$</td>
<td>$\approx 6.5$</td>
</tr>
<tr>
<td>11</td>
<td>p-FzSi-Diode2011C1</td>
<td>15</td>
<td>$\approx 51$</td>
<td>$\approx 6.3$</td>
</tr>
</tbody>
</table>

Table 3.1: Summary of IAEA silicon samples received in summer, 2012. $V_{fd}$ and $I_{lc}$ information were supplied by the manufacturers.

The silicon devices were produced by Okmetic Ltd and Topsil [160, 161] and later processed at the centre for Micro- and Nanotechnology of Helsinki University of Technology. The resistivity of MCz and Fz devices are given as 1.1 kΩ and 1.2 kΩ respectively. Full details of their fabrication is available at [162].

\textsuperscript{1}International Atomic Energy Agency Coordinated Research Programme, No. 17087 "Utilization of ion accelerators for studying and modelling of radiation induced defects in semiconductors and insulators"
The top surface of the material is Schottky with mirror-like color and a small opening for light transmittance and the other face is the backside; which is an ohmic contact. Both electrodes are aluminum (i.e., for the top and back surface). Figure 3.1 presents a schematic view of the devices. In between these electrodes are two lightly doped epitaxial layers grown on heavily doped substrate.

![Figure 3.1: Schematic diagram of IAEA n-type Si detector [163].](image)

Additionally, similar samples consisting of four (4) p-type Fz-silicon (p-Fz-Si) diodes irradiated at Centro Nacional De Aceleradores (CNA) at the University of Sevilla, Spain have been studied. The irradiation was carried out using 17 MeV protons in air at room temperature under zero bias with approximately 5 mm beam diameter; so that a large part of each detector was damaged. Fluences of the proton irradiations used range from $0.5 \times 10^{13}$ to $1 \times 10^{13}$ p cm$^{-2}$. Table 3.2 presents the information for each sample and its level of irradiation. The diodes have similar physical characteristics to the ones characterized earlier at Surrey University in terms of their doping profile, thickness and area [164]. In addition, they were diced from the same processed wafer.
3.2. MATERIALS AND DEVICES

<table>
<thead>
<tr>
<th>IAEA no.</th>
<th>Fluence ($1 \times 10^{13} \text{ p/cm}^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>33</td>
<td>0.5</td>
</tr>
<tr>
<td>36</td>
<td>1</td>
</tr>
<tr>
<td>34</td>
<td>5</td>
</tr>
<tr>
<td>76</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 3.2: Information about irradiated p–Fz-Si received from University of Sevilla.

These similarities provide the basis for comparing the results from these samples to the un-irradiated ones available at Surrey previously.

3.2.2 Silicon carbide

Silicon carbide samples used in this study are of two categories: epitaxial and semi-insulating (SI) devices. Some of the epitaxial devices were obtained from the RD50 collaboration while the remaining ones were supplied by the IAEA-CRP collaboration. The semi-insulating devices were fabricated using wafers purchased from Cree Incorporation (sample No. W4TRE0R-0200), U.S.A [125]. In the following discussion, details of characteristics of each sample are given. All devices are 4H-SiC polytypes.

RD50 sample

This SiC Schottky device was processed by Alenia Marconi Systems (AMS) from 50 µm thick n-type 4H-SiC epitaxial layer [$N_D - N_A = 5 \times 10^{14} \text{ cm}^{-3}$] grown on $n^+$-type 4HSiC wafer (16-30 micropipes/cm²) manufactured by Cree Inc. (see Fig. 3.2). Details of the fabrication processes are available in [165]. The diode of 10 mm × 10 mm area was diced from a 2” substrate.

EPI and SBD samples

These materials have identical properties because, they were diced from the same wafer both with area of 10 mm by 10 mm. However, the devices differ in their mode of fabrication.
The average thickness of their epitaxial layer is 47 $\mu$m and doping density $(4.85 \pm 0.25) \times 10^{14} cm^{-3}$ [163]. EPI is an epitaxial Schottky device fabricated at Surrey University (details of its fabrication are presented in section 3.3). SBD was fabricated by Ohshima [163] (a member of the IAEA-CRP collaboration). The top contact of this sample is Schottky, made up of an array of squares of dimension 1mm by 1mm Nickel contact with average thickness of 80 nm (see figure 3.3). The bottom surface of the sample is an ohmic contact formed by deposition of 100 nm Nickel and subsequently sintered at 950 °C.
3.3. FABRICATION PROCESS

Semi-Insulating (SI)SiC

The SI (high purity) SiC purchased from Cree Inc. has an average resistivity of $1 \times 10^9 \Omega$-cm and thickness $350 \pm 25 \mu$m [125]. The SI sample used for this research has an area of 10mm by 10 mm. The device was fabricated at University of Surrey (details of fabrication are presented in section 3.3).

3.3 Fabrication process

3.3.1 Wafer preparation and surface cleaning

All the wafers fabricated in this project were given thorough surface cleaning using acetone, Iso-propanol (IPA), methanol and de-ionized water to remove any surface contamination prior to metallisation. At the end of each cleaning process, nitrogen gas was used to dry the wafer surfaces.

3.3.2 Sputtering

After cleaning the surfaces, metal contacts (electrodes) were deposited on the two surfaces (top and bottom). A JLS sputterer was used to deposit top and bottom electrodes (Details of this equipment are presented in section 3.6). In each case the ohmic contact was formed first because, rapid thermal annealing (RTA) is required to improve the interaction between the metal and semiconductor to form the ohmic junction. At the final stage, Schottky contacts are formed (without further annealing). A number of different targets and thickness were adopted in this work based on the information provided in previous works; such as the work of Ruddy et al. [85]. Information on targets chosen and the electrode thickness for each sample is presented in table 3.3. The settings of the sputtering in each case are presented in appendix B.
Table 3.3: Targets and their thickness used for sputtering on SiC samples.

Two different shadow masks were designed and constructed for the top/Schottky contact formation. A square of 8 mm × 8 mm area for EPI and an 8 mm by 8 mm square with 0.5 mm recess and 4 circular holes of 3mm diameter around the centre. A schematic diagram of the shadow masks is presented in figure 3.4.

![Figure 3.4](image-url)

**Figure 3.4:** Pictorial representation of two shadow masks designed for metal deposition on (a) top surface of EPI and (b) top surface of SI silicon carbide substrates.

### 3.3.3 Annealing

Before forming the top contact (Schottky in this case), the samples have been annealed using a rapid thermal annealer. The ramp up temperature was 25 °C/s up to 400 °C. There was a hold at 400 °C for 1 minute and, then it was ramped up at the same rate (25 °C/s) up to 950 °C. The sample remained at this temperature for approximately 5 - 10 minutes. At the end of the 5-10 minutes the sample was allowed to cool down naturally (no forced cooling was applied). This annealing process was carried out by NANO-RF group.
3.4. MOUNTING AND WIRE BONDING

at Foundation for Research and Technology Hellas (FORTH) in Greece through Kostis Michelakis; an ATI staff who forwarded the samples.

3.3.4 Converter layer deposition

A lithium fluoride (LiF) powder enriched with 95 % atom of $^6$Li (with natural abundance of 7.59 %) was deposited on the top surface of the devices by the use of an Edwards E306 vacuum evaporator. The LiF powder was placed on the boats inside the vacuum chamber. After establishing vacuum (excess pressure of $2 \times 10^{-5}$ torr), voltage is applied to the boats which in turn heated the LiF salt to vapour state and finally deposited to the surface of the sample. Various thickness (of the order of $\mu$m) of LiF were deposited on glass slides followed by silicon and silicon carbide samples. This is to assist in deciding the best thickness for efficient thermal neutron detection without losing substantial amount of alpha energy. A thickness monitor was used to estimate the amount of deposited LiF.

3.4 Mounting and wire bonding

Samples were cleaned using acetone and carefully mounted on ceramic printed circuit boards (PCB) using a conducting silver paint (electrodag 1415 with maximum operating temperature of $\approx 100$ °C) with the back surface on the metal track. The ceramic PCB’s can operate up to 150 °C for several hundred cycles. Additional PCB’s rated for up to 400 °C were procured for SiC characterisation. A thin wire was glued to the top surface by applying the silver conducting paint (Figure 3.5). The thin wire was soldered together with a wire on the track of the PCB, while another wire was soldered on the metal tract to which the base was mounted on. A permanent marker was used to mark each sample on the PCB and stored in a plastic box.
A similar approach was followed for most of the SiC samples except for SBD which is a pixelated sample; a gold wire bonder (model 4124) manufactured by Kulicke and Soffa was used to connect 5 by 6 pixels of the device making a system of array of 30 devices (see figure 3.6).

Earlier, some pixels were characterised individually and compared to ascertain the uniformity of their response.
3.5 Equipment

3.5.1 Low temperature Cryostat

The low temperature cryostat is an existing system in the department of physics purposely built for low temperature related measurements. The cryostat is manufactured by Oxford Instruments Ltd. consisting of Microstat N (operating temperature 77–300 K) [166] connected to a rotary pump connected to the cryostat to evacuate the excess air in the cryostat (typical pressure during operation is 0.5 mbar) and TTC 502 temperature controller. The temperature controller is connected to a thermocouple and a copper sample holder (cold finger). There is an inlet and outlet pipe connected to the nitrogen dewar to allow flow of liquid nitrogen to cool down the temperature of cold finger.

3.5.2 High temperature Cryostat

The high temperature cryostat was commissioned for this project. This N2 Oxford Instrument’s cryostat is connected to a Mercury ITC temperature controller (with range 120–500 ± 0.5 K)[166]. The temperature stabilizes within 15 minutes while, sample change requires 30 minutes. A vacuum pump connected to the cryostat was used to evacuate the cryostat leaving excess pressure of 0.5 mbar. However, this cryostat was optimised for high temperature applications. After assembling the system, it was tested to make sure that its calibration is valid. A cryostat extension was designed and fabricated. Micro probes and their holders were also designed and fitted on the hot plate. Figure 3.7 shows the important parts of the cryostat.
This high temperature cryostat is very similar to the low temperature one. The basic difference between the two is that, the low temperature cryostat cannot be used at high temperature.

3.5.3 DLTS spectrometer

The DLTS spectrometer is an equipment that can be used to perform defect analysis of a semiconductor material (details of the processes and principles relating to its application are presented in section 2.13). The DLTS spectrometer used in this work is DL4600 manufactured by Polaron equipment, a Division of Bio-Rad laboratories. It is located in the Surrey ion beam centre. Training on the use of this equipment was provided by a staff with the ion beam centre. It consists of a cryostat (Comprising of microprobes)
3.6 Sputterer

The sputterer used is an MPS 500 manufactured by JLS limited. It provides an option of four (4) different 6 inch diameter RF/DC magnetron in the sputterer down configuration (figure 3.8).

![Figure 3.8: Pictorial representation of MPS 500 JLS sputterer located in the Advanced Technology Institute.](image)

It consists of load-lock and can be pumped with a turbo molecular pump or cryo pump. The equipment is operated via a PLC controller.

3.7 Measurement set-up

In order to characterize a material for radiation detection applications, electrical and optical techniques are normally employed. The former can provide information about leakage current, doping density and charge transport properties; the latter provides details of
structural properties of material [167].

This section is aimed at explaining different electrical methods employed in the investigation. Current-voltage and capacitance-voltage characterizations as well as alpha spectroscopy techniques are discussed.

3.7.1 Current–Voltage (I/V) characterization

The current signal as a function of voltage applied to the top surface of the samples was measured in air at room temperature with a Keithley 487 picoammeter, integrated with a voltage source that was used as bias supply. The samples were placed inside a purpose built die-cast box for I/V characterization of detector samples. The device under test (DUT) was connected in series with a 10 kΩ resistor (see figure 3.9). The use of the 10 kΩ resistor slows down the breakdown effect by increasing the time constant to the circuit.

![Set-up for I/V measurement.](image)

The box is light tight to avoid interference of light with the sample response and provide electronic shielding. Bias voltage applied to the top contact of the devices was in two stages; small voltage steps of typically 0.1 V step size from -1 to +1 V (or -2 to +2 V) to estimate bulk resistivity of the samples; at these voltages series and surface resistances can be neglected [168], and larger voltage steps typically 1 V step size up to expected
detector full depletion voltage are used to determine the magnitude of leakage current of the detector under typical operating bias values. For each voltage applied, one second was allowed to pass before the current was measured. This permitted measuring a steady current from the DUT. Bulk resistivity of the samples was determined from equation 3.1.

\[ I = \frac{A V}{\rho d} \]  

(3.1)

Where \( A \) is the diode area, \( I \) observed leakage current, \( d \) diode thickness and \( V \) the applied voltage. Using equation 3.1, bulk resistivity of the devices was estimated from forward current curves (I-V curves).

Additional leakage current was measured in vacuum from 300 ± 0.1 K to 500 ± 0.1 K (see figure 3.10). The temperature was controlled by means of an Oxford Instruments’ Microstat which comprises a cryostat, temperature controller connected to the hot finger in the cryostat and vacuum pump (as explained in section 3.5.2).

![Figure 3.10: Experimental set up for I/V measurement at elevated temperatures.](image)

Because of the voltage drop at the 10 kΩ resistor caused by relatively large leakage current in the forward bias, to extract the voltage drop across the DUT compared to the
applied bias a correction was made such that the voltage reaching the DUT is given by

\[ V = V_a - V_d \] (3.2)

where \( V_a \) is the applied voltage and \( V_d \) is the voltage drop across the series resistor. The ideality factor \( n \), and barrier height \( \Phi_B \), were determined from thermionic emission theory equations 3.3 and 3.4 [70]

\[ J = J_s \left( e^{\frac{qV}{nkT}} - 1 \right) \] (3.3)

\[ J_s = A^*T^2 e^{\frac{-q\Phi_B}{kT}} \] (3.4)

where \( A^* \) is the Richardson constant taken as 146 \( cm^{-2}K^{-2} \) for SiC [169], \( V \) the forward voltage, \( q \) electronic charge, \( k \) Boltzmann’s constant and \( T \) the temperature. Current stability was measured by subjecting the devices to high temperatures for 20 minutes. Resistivity values extracted from the high temperature forward I/V curves were used to estimate the activation energy from equation 3.5 [170]

\[ \ln(\rho) = \ln(\rho_0) + \frac{E_a}{kT} \] (3.5)

where \( E_a \) is the activation energy.

### 3.7.2 Capacitance–Voltage (C/V) characterization

Capacitance of the devices was measured in air at room temperature without light interference by applying bias voltage to the top contact of the devices. An LCR meter (Agilent 4284A) was connected to the detector as shown in figure 3.11 where it acts as voltage source while it measures the capacitance (i.e. from 0 to +40V or -40V to 0 as the case
3.7. MEASUREMENT SET-UP

may be). The capacitance was measured by changing the voltage range on a dedicated LabVIEW program. An elapse time of one second was allowed to pass for each applied voltage before the capacitance was measured.

![Diagram of measurement setup](image)

**Figure 3.11:** Standard circuit for C/V measurement of semiconductor materials; where $L_C$: low current, $L_P$: low potential, $H_P$: high potential and $H_C$: high current.

A small ac voltage ($\approx 0.03$ mV(rms)) was superimposed on the dc voltage at a frequency of 1 MHz. This frequency was chosen after a series of C/V measurements have been carried out on a silicon PIN diode at frequencies of 10 kHz, 100 kHz and 1 MHz respectively (see figure 3.12). Although the highest frequency was used in this measurement, the difference in the results as a function frequency is only observed at lower voltages (i.e. 1.2 V and below).
The high energy physics community has chosen 10 kHz as their frequency for the testing signal [171]; which may be for the purpose of comparison, figure 3.12 shows that it is also possible to perform the measurement at higher frequencies. On the other hand, the CRP has not adopted any unique frequency for C/V measurement; which may be helpful in comparing results, since the capacitance reading depends on the frequency even for non-irradiated samples [171]. Campbell et al. [172] seems to have overlooked the variation of C/V values in non-irradiated samples and considered only the irradiated ones.

An alternative configuration using the Keithley 487 as the source of voltage (figure 3.13) is in principle capable of providing a higher voltage range of the C/V measurements. However, in this configuration electrical length correction has not been possible and as such a noisy and unstable curve was observed (see figure 3.14).
Figure 3.13: Alternative set-up for C/V measurement. \( R_L: 226 \, k\Omega \), \( R_F: 100 \, k\Omega \), \( R_H: 10 \, k\Omega \), \( C_F: 1 \, \mu F \), \( C_L: 0.1 \, \mu F \), \( C_H = 10 \, \mu F \), \( L_C: \text{low current} \), \( L_P: \text{low potential} \), \( H_P: \text{high potential} \) and \( H_C: \text{high current} \) [172].

An Agilent external bias fixture (AG16065A) capable of providing up to +/- 200 V was used instead, but no significant improvement was observed in the C/V result. For the sake of the current investigation, the second configuration has no significant contribution. Due to the limitation of the LCR meter (+/- 40 V internal bias supply), measurements of capacitance up to the expected depletion voltage for most of the detectors could not be achieved.

Figure 3.14: C-V measurement based on figure 3.13 at 1MHz frequency.

Additional measurements of capacitance were carried out at different frequencies with the Agilent 4284A LCR meter. Capacitance variation with temperature from low temper-
ature to room temperature were investigated using the DLTS spectrometer while, similar studies from room temperature to high temperature were conducted with a 4200-SCS Keithley semiconductor parameter analyser. Access to this instrument was provided by the ATI at the University of Surrey with additional support from their staff. It is worth to note that room and low temperature measurements were carried out on silicon devices while, for silicon carbide devices high and room temperature were applied. Doping density \( N_D \) and depletion thickness \( w \) were obtained from equations 3.6 and 3.7 respectively [173]

\[
N_D = \frac{2}{qwA C \frac{d(C/V)}{dV}} \tag{3.6}
\]

\[
w = \frac{\epsilon_0 \epsilon_{4H-SiC} A}{C} \tag{3.7}
\]

where \( A \) is active area of the device , \( \epsilon_0 \) permittivity of free space, \( \epsilon_{4H-SiC} \) material’s relative permittivity (6.7 for 4H-SiC and 11.9 for Si [77]) and \( C \) the capacitance.

### 3.7.3 Alpha spectroscopy

The set-up for alpha spectroscopy is shown in figure 3.15 consisting of a multichannel analyser (MCA), shaping amplifier (ORTEC 570), preamplifier (ORTEC 142 A), pulser and bias supply; all fixed in a nuclear instrumentation module (NIM) rack. The detector is mounted on the base of a cryostat which is connected to a vacuum pump to reduce energy loss due to air pressure (remaining air pressure is less than 0.05 mbar).
Two different alpha sources were used; alpha dumbbell source for Si devices and disc source for SiC devices. Due to safety reasons, these are the sources that could only be used for the two extreme temperatures. The dumbbell source (which is rated for low temperature only) is an uncollimated radioisotope ($^{241}$Am $\alpha$-source) which has an average emission energy of 4.95 MeV and approximate activity of 180 kBq. It was placed at about 5 mm above the Si detector inside the cryostat. The encapsulated disc source (rated for high temperature only) is an uncollimated ($^{241}$Am $\alpha$-source) radioisotope with average emission energy of 3.76 MeV and approximate activity of 60 kBq. The source was placed at about 20 mm above the SiC detector.

Both $^{241}$Am sources used in this study suffer from energy degradation due to the nature of their encapsulation. The sources are sealed within a disk of thin silver foil. This is in accordance with American National Standards Institute classification for high/low temperature rated sources. Spectral responses acquired from $^{241}$Am alpha emitting sources using a Si-pin diode under the same condition including those used in this study, have shown clearly the shift and broadening in the spectra of the emission energy of the sources.

Figure 3.15: Set-up used for the high temperature alpha spectroscopy.
in comparison to other sources. Fig. 3.16 is a comparison between the single alpha sources used in this study ($^{241}$Am) and a triple alpha source ($^{239}$Pu, $^{241}$Am and $^{244}$Cm).

![Energy emission spectra comparison](image)

Figure 3.16: *Comparison of energy emission spectra between $^{241}$Am and $^{239}$Pu, $^{241}$Am and $^{244}$Cm triple alpha radiation sources.*

The detectors are normally biased via an ORTEC (142A) charge sensitive preamplifier with their output signal connected (with a BNC cable) to the spectroscopy amplifier for signal integration. Finally, the multi channel analyser converts the signal amplitude from the shaping amplifier into digital form which is displayed on a computer using MCA software.

Spectral responses were acquired from Silicon detectors at room temperature for a number of bias voltages for about 8 to 10 minutes using MCA software. For some detectors low level discrimination (LLD) was used to discard noise. Alpha spectroscopy of the irradiated silicon samples was carried out under cryogenic temperature (at approximately 100 K) using liquid nitrogen in order to reduce leakage current, which inhibited room temperature characterisation. The detectors were placed in the low temperature cryostat (details of this cryostat are presented in section 3.5.1).

Silicon carbide alpha spectra were recorded for various applied bias voltages at different temperatures; ranging from 300 to 500 K at intervals of 50 K and 100 K in some occasions.
The radiation source and detector were placed inside a temperature controlled cryostat. In addition, $^{241}\text{Am}$ and pulser signal peaks were collected simultaneously by injecting a signal from a 480 ORTEC pulser into the test input of the 142A ORTEC pre-amplifier. The broadening of the pulser peak recorded in this manner can be used to assess collectively the effect of detector leakage current and that of the noise form the front-end electronics to the overall resolution of the detector [174]. Further, stability of the signal was studied by fixing the values of temperature and bias voltage for longer periods up to 24 hours. Spectra were recorded at intervals of 20 minutes. In this work, we consider stability to be reached when the signal variations with time have dropped below 0.5% of the signal value.

A 2.31 pF standard capacitor was connected to the pulser and preamplifier to calibrate the channels as suggested by Chaudhuri et al. [174]. The voltage from the pulser is measured in mV on the oscilloscope. For each pulser signal, peak to peak voltage is measured against the peak centroid (channel number) from the MCA. 5 to 40 mV pulser signals were applied to the capacitor with intervals of 5mV and, the corresponding values of centroid were recorded. The pulser signal recorded in mV is converted in to energy in keV using equation 3.8 [174]

$$E_{\text{pulser}}(\text{keV}) = V_{\text{pulser}}(\text{mV}) \times C(\text{pF}) \times w(\text{eV}/\text{ehp}) \times 6.25$$

(3.8)

Where $E_{\text{pulser}}$ is the pulser energy in keV, $V_{\text{pulser}}$ the pulser voltage in mV, $C$ is capacitance in pF and $w$ is the energy per electron hole pair. Values of $w$ used For silicon and silicon carbide detectors are 3.6 eV/ehp and 7.28 ev/ehp respectively, while the value of $C$ is 2.31 pF. The pulser energy was plotted as a function of peak centroid and the fitted equation was used to estimate the energy at each applied voltage.

Determination of mobility-lifetime product from equations 2.23 and 2.30 was not possible, because the Hecht equation assumes that the internal electric field is uniform.
inside the detector; which is not true for the devices used in this project. More details about suitability of using equation 2.30 in this experiment and other related experiments that require determination of mobility-lifetime values has been discussed in section 2.7.

### 3.7.4 Neutron spectroscopy

Thermal neutron detection was carried out using two AmBe standard neutron sources located inside a water tank. The two sources with average activities of 11.0 and 18.5 GBq respectively are separated by a horizontal distance of 30 cm. The expected flux from these sources is $6.72 \times 10^5$ and $1.2 \times 10^6$ neutrons/s for the weak and strong sources respectively. The distance between the surface of the sources to the bottom of a transparent plastic pipe (which is closed at the bottom) is $10 \pm 2$ cm; which provides thermalisation of the fast neutrons. The distance between the frame and the bottom of the plastic pipe is $41 \pm 0.5$ cm. Figure 3.17 depicts the experimental set up for neutron test.

![Figure 3.17: Set up for neutron measurement. The dimensions in this figure are in cm.](image)

An initial characterisation carried out using a $^3$He gas detector with and without cadmium shielding, suggests 10 to 12 cm to be sufficient for slowing down the fast neutrons without undermining the thermal neutron flux. Due to replacement of the neutron sources, the weaker one was used for silicon devices while the stronger for silicon carbide detectors.
3.7. MEASUREMENT SET-UP

The high temperature cryostat was used for high temperature measurements and an aluminium box for room temperature tests. As a result of low number of counts experienced during the measurement, each spectrum was recorded after 20 hours. Finally, spectra were acquired for 100 hours with interval of 20 hours at different temperatures ranging from 300 to 500 K using the RD-50 silicon carbide device. Cadmium shielding was used in each case in order to differentiate between events from fast neutrons and those from slow neutrons.
Chapter 4

Charge transport and defect analysis of silicon detectors

This chapter presents current and capacitance variation with voltage. Some of the important parameters extracted such as bulk resistivity, depletion thickness and doping density are discussed. In addition, defects characterisation and alpha spectroscopy are studied. The implication of the magnitude of each parameter in relation to detector performance is considered.

4.1 I/V characteristics

All diodes show good blocking characteristics (see figures 4.1b and 4.3b). Generally, there is an offset at 0 bias voltage in all the devices; indicating the presence of an inbuilt potential in the devices. Significant difference in magnitude of leakage current was observed in samples 1 and 2 which were fabricated using the MCz-method. Sample 2 has a leakage current density of \( \approx 33 \, \text{nAcm}^{-2} \) at -300 V while, sample 1 shows a leakage current density of \( \approx 42 \, \mu\text{Acm}^{-2} \) at -200 V as the device approaches its breakdown limit (Figure 4.1).
Figure 4.1: (a) Leakage current density as a function of reverse bias for MCz-2009-Si samples 1 and 2. Sample 1 approaches breakdown at \( \sim -200 \) V (b) Current density for n-type silicon samples 1, 2 and 4 indicating their Schottky behaviour.

Though samples 4 and 8 are of different types (i.e. n-type and p-type respectively), they were fabricated using the same method (Fz-technique). The samples have similar magnitudes of leakage current density of the order of nAcm\(^{-2}\) (Figure 4.2).

Figure 4.2: Leakage current density for n-Fz-wafer-2012 (sample 4) and p-Fz Diode-2012 (sample 8).

The remaining p-type samples (sample 5 to 7 and 9 to 11) can be categorised in to three;
with each group representing two similar diodes. Current density from these samples ranges from 65 nAcm$^{-2}$ to 0.62 µAcm$^{-2}$. Samples 10 and 11 show the least current density while, the highest current density was observed in samples 6 and 9 (Figure 4.3).

The resistivity of these devices estimated from forward I/V curves ranges form $1.3 \times 10^5$ to $2.2 \times 10^7$Ωcm. The full list of resistivity for all 11 devices, is presented in table 4.1.

Figure 4.4 shows leakage current from the irradiated silicon devices compared to a non-irradiated sample.
As presented in figure 4.4, it is observed that the leakage current measured at room temperature is of the order of µA. The leakage current in sample 76 is the highest and the lowest was observed in sample 33. This is expected owing to the fact that sample 76 was irradiated with higher fluence compared to samples 33 and 34. This value of current is 33% higher than the one recorded for unirradiated samples (see sample 7 for example in the figure). Similar increase in leakage current after irradiation have also been reported by Vizkelethy [175] after irradiating comparable samples with a fluence of $10^{15}$cm$^{-2}$ of 1 MeV electrons. However, due to the nature of interaction in terms of depth and charge on particles, greater damage with proton irradiation is expected [76].

4.2 C/V characteristics

The plot of $1/C^2$ against voltage is linear; indicating a uniform doping density in the devices. As shown in figure 4.5, samples 1 and 2 behave in a similar manner in terms of the magnitude of their doping density and depletion width; which appears to be lower than that observed in sample 4. In addition, sample 4 is fully depleted at $\simeq 30$ V while,
samples 1 and 2 require larger voltage to achieve full depletion.

**Figure 4.5:** Doping profile and depletion thickness of n-type IAEA silicon samples. Sample 4 reached full depletion at 30 V.

Furthermore, the depletion thickness of sample 4 is twice that of samples 1 and 2.

The doping density of samples 5 to 11 range from $1.1 \times 10^{12}$ to $7.8 \times 10^{12}$ cm$^{-3}$ (Figure 4.6a) and their depletion thickness is similar except for samples 7 and 11 which have lower values of doping density and depletion thickness (Figure 4.6b). The observable steps in sample 7 for doping density and depletion thickness curves, which occur between $\approx 7.5$ to $27.5$ bias voltage are believed to be the artefact of the sample. A strange peak was observed in the capacitance of the p-type samples at very low bias (< 2.0 V). Figure 4.6c is an enlargement of a C/V curve exposing the peak. This is in agreement with the results obtained by Vizkelethy [175] a member of IAEA-CRP collaboration on similar devices. The origin of this strange peak has been investigated by varying the frequency and temperature of measurement. It has been established that this feature is dependent on both temperature and frequency. A detailed discussion on this will be presented in section 4.3.
4.2. C/V CHARACTERISTICS

![Graphs showing C/V characteristics](image)

Figure 4.6: Doping density (a) and depletion thickness (b) for p-type IAEA silicon samples. Peaks are observable towards the left side of the graph at voltages lower than 2 Volts for some devices. Enlarged part of a C/V curve showing the area where the peak appeared is shown in (c).

SRIM [176] calculation performed, reveal that the alpha penetration depth in silicon material is about 26 µm when irradiated with 5 MeV alpha particles (similar to the energy of the source used for silicon spectroscopy in this work); which implies that most of the charge created is expected to be collected by the detector (figure 4.7).
The doping density value and depletion thickness obtained from the measurements agree well with the result obtained by Vizkelethy [175] and data provided by sample manufacturers. Doping density of each device is presented in table 4.1.

<table>
<thead>
<tr>
<th>Surrey no.</th>
<th>Resistivity (Ωcm)</th>
<th>Doping density(cm⁻³)</th>
<th>Bias Voltage(V [Recommended])</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.3±0.1 × 10⁹</td>
<td>7.8×10¹² ± 1.4×10¹⁰</td>
<td>300</td>
</tr>
<tr>
<td>2</td>
<td>2.1±0.3 × 10⁹</td>
<td>6.4×10¹² ± 1.0×10¹⁰</td>
<td>350</td>
</tr>
<tr>
<td>3</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>1.2±0.2 × 10⁹</td>
<td>1.1×10¹² ± 3.0×10⁹</td>
<td>50</td>
</tr>
<tr>
<td>5</td>
<td>2.2±0.5 × 10⁹</td>
<td>1.3×10¹² ± 4.0×10⁹</td>
<td>50</td>
</tr>
<tr>
<td>6</td>
<td>2.0±0.2 × 10⁹</td>
<td>1.4×10¹² ± 5.7×10⁹</td>
<td>50</td>
</tr>
<tr>
<td>7</td>
<td>5.8±0.5 × 10⁹</td>
<td>2.1×10¹² ± 8.3×10⁹</td>
<td>50</td>
</tr>
<tr>
<td>8</td>
<td>4.7±0.4 × 10⁹</td>
<td>1.1×10¹² ± 4.8×10⁹</td>
<td>20</td>
</tr>
<tr>
<td>9</td>
<td>9.2±2 × 10⁹</td>
<td>1.5×10¹² ± 6.4×10⁹</td>
<td>35</td>
</tr>
<tr>
<td>10</td>
<td>5.4±0.4 × 10⁹</td>
<td>1.3×10¹² ± 4.7×10⁹</td>
<td>50</td>
</tr>
<tr>
<td>11</td>
<td>4.9±1.7 × 10⁹</td>
<td>3.6×10¹² ± 9.8×10⁹</td>
<td>50</td>
</tr>
</tbody>
</table>

Table 4.1: Estimated resistivity values and doping density for IAEA silicon samples. Sample 3 was damaged (i.e. broken into smaller pieces) in the process; as such its data is not presented. Recommended operational voltage of the devices can be seen in the last column of the table.

Considering the leakage current from the devices and depletion thickness in relation to applied bias it is reasonable to suggest an operational voltage for each device as presented in the table. This recommended voltage was arrived at based on the level of leakage current...
from the devices, depletion thickness and centroid position in alpha spectroscopy.

### 4.3 C/V variation with frequency and temperature for silicon based detectors

As earlier discussed in section 4.2, a peak appeared in the C/V curves of the p-type silicon devices at low bias voltages, its variation with temperature and frequency is examined. Figure 4.8a shows the variation of Capacitance of sample 11 (as an example) at different voltages with varying frequency. Compared to sample 4 in figure 4.8b which is an n-type sample.

![Capacitance variation with frequency for different applied voltages](image)

**Figure 4.8**: Capacitance variation with frequency for different applied voltages for (a) Sample 11 (p-type) and (b) Sample 4 (n-type). Prominent peaks are observable in sample 11 at frequencies higher than 400 kHz.

Peaks were prominent in sample 11 from 600 kHz to 1 MHz. At frequencies lower than 600 kHz, the peaks disappear. Comparing this behaviour to sample 4 (which is an n-type device), no peaks were seen at all frequencies. This indicates that the peaks observed during the room temperature C/V measurements, are frequency dependent. An additional feature found in the frequency dependent measurement, is the decrease in capacitance with
increase in frequency which is visible in the p- and n-type devices as reported by Boschini et al. [177]. Although they reported it in their findings, they did not indicate its cause.

Similar results were obtained when the devices were subjected to different temperatures while maintaining 1 MHz measurement frequency (see figure 4.9).

![Figure 4.9: Capacitance variation with temperature for different applied voltages for (a) Sample 11 (p-type) and (b) sample 4 (n-type).](image)

It can be seen from the figure that sample 11 has a peak at room temperature (300 K) which disappears at 250 K and below (figure 4.9a). Contrary to sample 11, sample 4 indicates no presence of peak at all temperatures of measurement (figure 4.9b).

Further investigation into the defects present in these silicon devices using DLTS measurement reveals prominent deep traps in the four devices with similar activation energies. The cross section of the traps is in the order of \(10^{-5}\) cm\(^2\). Figure 4.10 shows a typical DLTS signal acquired from sample 6 as an example. H and C refer to heating and cooling regimes respectively. The values with unit s\(^{-1}\) represent the rate window defined by equation 2.57; which is the inverse of relaxation time. The parameter has been explained in detail in section 2.13.1.
4.3. C/V VARIATION WITH FREQUENCY AND TEMPERATURE FOR SILICON BASED DETECTORS

Figure 4.10: DLTS signal for Si sample 6. The peaks appeared between 200 and 340 K. C and H represent cooling and heating curves respectively.
As illustrated by figure 4.10, all the peaks observed were negative indicating that the traps are for majority carriers (holes for p-type and electrons for n-type devices) [157, 178]. The signals recorded for heating and cooling cycles did not indicate significant thermal delay in the process. The peaks shift from lower to higher temperatures during heating cycle while, the reverse is the case for cooling. Using the DLTS signal peaks for all the four devices as in figure 4.10 and equation 2.54, Arrhenius graphs were plotted as shown in figure 4.11.

**Figure 4.11:** Arrhenius plots for 3 p-type and 1 n-type silicon devices

By evaluating the gradient and intercept of the graphs, estimated activation energy values and cross sections were determined as presented in table 4.2.

<table>
<thead>
<tr>
<th>Surrey no.</th>
<th>Activation energy (eV)</th>
<th>Cross section ($\times 10^{-5} \text{cm}^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.42±0.08</td>
<td>3.33±0.03</td>
</tr>
<tr>
<td>6</td>
<td>0.45±0.02</td>
<td>2.44±0.17</td>
</tr>
<tr>
<td>7</td>
<td>0.46±0.02</td>
<td>5.12±0.40</td>
</tr>
<tr>
<td>11</td>
<td>0.43±0.03</td>
<td>1.20±0.15</td>
</tr>
</tbody>
</table>

**Table 4.2:** Summary of activation energies and cross section of the traps detected in non-irradiated silicon devices
The activation energies of the p-type samples are associated with boron interstitial as reported by [179]. However, the origin of the n-type trap is not fully understood. Values of activation energy are more comparable with each other than the capture cross sections. This is due to the extrapolation method used in estimating the values. The best method is to measure the capture cross section directly using photo electron spectroscopy or other relevant methods [180]. This could not be achieved due to limited facilities. Nevertheless, all values have the same order (i.e. $10^{-5}$).

### 4.4 Alpha spectroscopy

Charge transport of these devices was studied by irradiating the samples with 4.95 MeV alpha particles (Am-241) and the spectra recorded with an MCA as described in section 3.7.3. A significant shift in peak centroid towards higher channel numbers with increasing bias voltage was observed in all samples. This indicates that more charge is collected as the bias increases. Figure 4.12 shows a typical pulse height spectra acquired as a function of bias voltage from sample 7, representative of all non-irradiated samples.

![Figure 4.12: Alpha pulse height spectra from Am-241 for silicon sample 7 measured at room temperature. The peaks tend to broaden as the bias voltage is increased](image-url)

Figure 4.12: Alpha pulse height spectra from Am-241 for silicon sample 7 measured at room temperature. The peaks tend to broaden as the bias voltage is increased.
The change of peak centroid position is more pronounced at low voltages, while it tends to reduce drastically at high bias voltages; showing that the device approaches its full depletion Voltage. The peaks are generally broad due to energy degradation of the radiation source as explained in section 3.7.3. However, the broadening of peaks increases with the applied bias voltage, which is expected because of the increase in leakage current with increasing bias voltage. The variation of leakage current with bias voltage has already been discussed in section 4.1. As the width of the peaks is dominated by the broad emission spectrum of the source rather than the quality of the detector, it may not be rational at this point to conclude on its resolution.

The CCE of the detectors was estimated from equation 2.23. The CCE of all the detectors is greater than 80% at full depletion voltage. Figure 4.13 illustrates the CCE for n-type and p-type silicon samples respectively.

(a) Variation of CCE with bias voltage for n-type samples
(b) Variation of CCE with bias voltage for p-type samples

Figure 4.13: CCE as a function of reverse bias for p-type and n-type non-irradiated silicon samples

As presented in the figure, the devices demonstrated large CCE even at 0 bias voltage; indicating that the devices can operate as efficient detectors even at 0 applied voltages. Sample 8 has a unique feature; achieving about 87% CCE at 20 V bias. Thus sample 8
4.4. ALPHA SPECTROSCOPY

seems to be a good option where source of voltage is an issue.

Because of the high leakage current observed in the irradiated silicon samples as evident from figure 4.4, alpha spectroscopy at room temperature was not practicable. In order to study the charge transport in the devices using alpha spectroscopy, the measurement temperature was reduced by employing liquid nitrogen. Although it is important to reduce the temperature it was obvious that the performance of the irradiated devices can be undermined by subjecting them to very low temperature (for example \(\sim 100\) K) as indicated in figure 4.14.

![Alpha spectra acquired from sample 33 at 20 V bias for different temperatures](image)

**Figure 4.14:** *Alpha spectra acquired from sample 33 at 20 V bias for different temperatures*

From the figure, 148 K is the temperature at which charge collection is highest and thus chosen for the spectroscopy of the damaged samples. This is to avoid freezing out of charge carriers as highlighted by Singh [73].

Figure 4.15 shows the CCE of the three detectors studied under low temperature.
In the figure, sample 76 has lowest CCE (20%) and the highest is observed in sample 33. This is expected because of the variation in the radiation fluence to which the samples were exposed to. The reduction in CCE is normally caused by excessive trapping which results from radiation induced defects [181]. A related study shows that irradiating MCz-silicon detector with $10^{15}$ pcm$^{-2}$ will result in 25% reduction of CCE and $1.6 \times 10^{16}$ pcm$^{-2}$ is capable of reducing the CCE to 25% [181]. In the current study there is a reduction of 61% and more than 70% of CCE as a result of irradiation with $5 \times 10^{13}$ pcm$^{-2}$ and $10 \times 10^{13}$ pcm$^{-2}$ respectively. By comparing these results with the one obtained in figure 4.13b, it can be seen that the CCE of sample 33 is not significantly affected by the proton irradiation. This happened partly because of the oxygen diffusion utilized during production of the samples as anticipated [110]. Although looking at the CCE reduction in MCz-Si and Fz-Si, one can see that the MCz-Si appeared to be more radiation resistant at the expense of resistivity.

4.5 Summary

The focus of studying silicon devices based on the IAEA collaboration as stated in chapter 1 is to identify changes in the characteristics of the devices after being irradiated with
protons. It also seeks to understand the quality of the devices in terms of their level of leakage current and alpha particle detection. So far it has been demonstrated that the leakage current from the unirradiated devices at room temperature is very small (of the order of nAcm$^{-2}$) in most of the samples.

The CCE is excellent with an average value of 80%. The effect of proton irradiation was highlighted as well as the influence of low temperature in reducing the magnitude of leakage current from the irradiated devices. However, caution should be exercised, so that the carriers will not freeze out during spectroscopy.

The capacitance investigation, has levered interesting features of variation of capacitance with temperature and frequency, precisely at low voltages. When compared to the DLTS result, a remarkable correlation was observed. Although the cause of these peaks was not fully clear, it highlights the need for further research to unravel the mystery. With respect to activation energies and cross sections for all the devices in their pristine state, indicates uniformity of the large substrate because of similarity in both the energies and cross sections.
Chapter 5

Operational stability of SiC at high temperature

In this chapter, current and capacitance behaviour of SiC devices at elevated temperature are presented. The parameters evaluated such as barrier height, activation energy and depletion thickness are discussed. CCE dependence on voltage and temperature was examined. The link between the capacitance and current density study was established. The impact of these results and their effect on the operational characteristics of the materials are considered. Also, the application of the devices in light of their response to radiation while being subjected to different environmental conditions are discussed. Some content of this chapter have been published in "Y. M. Abubakar, A. Lohstroh, and P. J. Sellin, ‘Stability of Silicon Carbide Particle Detector Performance at Elevated Temperatures’, IEEE Transactions on Nuclear Science, vol. 62, no. 5, pp. 2360-2366, Oct. 2015’.
5.1 Current conduction at elevated temperature

Current conduction in the devices is very low under reverse bias as expected from Schottky barrier devices. Current density as a function of bias voltage for temperatures from 300 to 500 K is presented in figure 5.1. In figure 5.1a it can be see that, the RD-50 sample has a very low current at room temperature compared to other temperatures above it. In fact, by just increasing the temperature from 300 K to 360 K, current density increases seven fold. Although a different scenario is observed in the EPI sample (figure 5.1c), there is an anomaly in the curves for 400 to 500 K at \( \sim 10 \) and \( \sim 40 \) V. This could be due to interface and edge effects between the electrode and the semiconductor. Other reasons may have to do with fabrication issues. Although there were few variations in the fabrication parameters based on the approaches discussed in section 2.6.3, the device shows blocking of current in one polarity compared to the other.
Figure 5.1: Current density as a function of bias voltage for temperatures from 300 K to 500 K for epitaxial 4H-SiC devices

Much lower current density can be noticed in the SBD sample with highest current density of $\sim 62 \text{nAcm}^{-2}$ at 500 K. This indicates that the pixelated devices are better in terms of low leakage current and, are therefore expected to have large signal-to-noise ratio due to small contact area because of low capacitance [107]. Under reverse bias, current transport is mainly due to tunnelling (popularly called thermionic field emission) because of the large applied voltage [70]. An additional increase is expected as a result of Schottky barrier
5.1. CURRENT CONDUCTION AT ELEVATED TEMPERATURE

lowering; as discussed in section 2.6 [98].

For the SI sample (which is a semi-insulating bulk device), the current density from 300 K to 500 K varies from 4.5 nAcm\(^{-2}\) to 60 \(\mu\)Acm\(^{-2}\) (see figure 5.2).

![Figure 5.2: Current density for SiC SI sample. The colour of each curve relates to the colour of the axis.](image)

Although the method used to fabricate the sample aimed to produce a Schottky contact on the top surface and an ohmic one on the bottom, it is not obvious to infer reverse and forward bias from the shape of the I/V curve. However, the current density in the device is generally low.

The resistivity of the epitaxial devices extracted from forward I/V curves vary linearly with temperature. Figure 5.3 shows the variation of resistivity with temperature for the epitaxial and semi-insulating SiC devices.
CHAPTER 5. OPERATIONAL STABILITY OF SiC AT HIGH TEMPERATURE

Figure 5.3: Resistivity variation with temperature from 300 K to 500 K for 3 epitaxial 4H-SiC devices (a) to (c) and a bulk 4H-SiC device (d). The error bars were derived form errors of the graph used to estimate the resistance in the low voltage I/V curves.

As evident from the figure, the resistivity reduces with increasing temperature in samples RD-50, SBD and SI while, the EPI sample behaves differently with non-linear characteristics. Its resistivity appear to be constant from 300 to 400 K. Beyond 400 K, the resistivity increases. The large value of resistivity in SI-1 is expected because it is a semi-insulating
5.1. CURRENT CONDUCTION AT ELEVATED TEMPERATURE

<table>
<thead>
<tr>
<th>Sample</th>
<th>$E_a$ (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RD-50</td>
<td>$0.00593 \pm 0.00036$</td>
</tr>
<tr>
<td>SBD</td>
<td>$0.15 \pm 0.01$</td>
</tr>
<tr>
<td>SI-1</td>
<td>$0.33 \pm 0.03$</td>
</tr>
</tbody>
</table>

Table 5.1: Activation energies extracted from resistivity-temperature curves for 4H-SiC devices

device. There was a slow rising part in the forward I/V curves of the epitaxial devices resulting from the barrier effect. Activation energies extracted from figure 5.3 are presented in table 5.1.

The ideality factor (IF) and barrier height (BH) estimated from the forward bias curves is similar for SBD and EPI devices and different for RD-50 sample (figure 5.4).

As shown in figure 5.4, IF values for SBD range from 2.38 (at 300 K) to 1.36 (at 500 K); lower than those obtained from EPI which range from 3.38 (at 300 K) to 2.13 (at 500 K). The BH range from 1.14 eV (at 300 K) to 1.72 eV (at 500 K) and 0.95 eV (at 300 K) to 1.63 eV (at 500 K) for SBD and EPI devices respectively. The similarity in response between SBD and EPI is not surprising, considering the fact that they were diced from the same wafer. The only difference between the two is the contact geometry.

Contrary to the SBD and EPI samples, the RD-50 sample shows a consistent and
linear increase of IF with temperature up to 500 K. Within this range the maximum and minimum values of IF are 1.17 and 1.79 respectively (Fig. 5.4). On the other hand, the SBH reduces with temperature increase from 1.33 to 1.11 eV within the range of 300 K to 500 K. The increase in ideality factor with increasing temperature indicates that thermionic emission is dominant and, a similar result has been reported by Lee et al. [182]. As for the reduction in SBH, it is possible that the temperature increase has increased the effective dopant density in the device, which can reduce the SBH so that thermionic field emission could take place [91]. Generally, recombination current (under low forward biases) plays an important role in determining the departure from ideal behaviour of Schottky diodes [91].

Leakage current (monitored for 20 minutes) reaches stable values faster at higher temperatures. As illustrated in Figure 5.5, it can be observed that at 500 K the current stabilizes just after the first two minutes while at lower temperatures it takes up to 8 minutes or more before stability is achieved. Disregarding the magnitude of the leakage current, it is clear that the operation of this device is favoured more at high temperatures. In this work, stability is considered to be reached, when the signal variations with time have dropped below 0.5% of the signal value.
5.1. CURRENT CONDUCTION AT ELEVATED TEMPERATURE

Figure 5.5: Leakage current stability studies of RD-50 sample for 20 minutes at different bias voltages and temperatures. (a) 300 K, 50 V (b) 400 K, 50 V and 300 K, 100 V (c) 400 k, 100 V (d) 500 K, 50 V and 500 K, 100 V.

Compared to RD-50 sample, the leakage current of SI sample appears to stabilize faster at all temperatures (see figure ).
Figure 5.6: Leakage current stability of SI-SiC at different temperatures for approximately 17 hours. Some of the measurements were performed beyond 17 hours.

However, due to time constraints, the leakage current of the SI sample could not be measured beyond 343 K. On the other hand, the RD-50 sample stability for leakage current was only studied for 20 minutes. Although this length of time is incomparable to 17 hours, it is expected to be stable beyond the 20 minutes and possible even achieve better stability.

5.2 Capacitance behaviour at room and high temperatures

The $1/C^2$ versus voltage is linear in all the three devices studied, indicating that the doping concentration is constant throughout the active volume of the devices yielding values of $(16.3 \pm 0.04) \times 10^{14}$ cm$^{-3}$, $(4.81 \pm 0.02) \times 10^{14}$ cm$^{-3}$ and $(5.80 \pm 0.03) \times 10^{14}$ cm$^{-3}$ for RD-50, SBD and EPI samples respectively (Fig. 5.7a).
5.2. CAPACITANCE BEHAVIOUR AT ROOM AND HIGH TEMPERATURES

The doping density estimated for the EPI and SBD samples are in fairly close agreement with each other. This is expected since the devices were diced from the same wafer. On the other hand, RD-50 sample has doping density that almost double the value of EPI device.

The depletion thickness reaches 32.5 $\mu$m at 40V for the RD-50 sample while, lower values were observed in SBD and EPI samples (see figure 5.7b). A SRIM [176] simulation performed for alpha particle penetration depth in SiC is given in figure 5.8. According to the figure, alpha particles with energy similar to the source used have an average range of 10.56 $\mu$m (i.e., for 3.76 MeV alpha particles). Therefore, comparing figures 5.7 and 5.8 shows that, there is full assurance that most of the alpha particles striking the surface of the samples will only cause ionization in the active volume. It is important to note that, although the values of depletion thickness as in SBD and EPI devices are less than the simulated value, it is expected that more depletion width will be achieved at higher voltages. Due to equipment limitation, the depletion thickness could not be estimated for bias voltages greater than 40 V.

Figure 5.7: Relationship between capacitance and voltage at room temperature showing (a) Doping profile and (b) Depletion thickness for epitaxial SiC devices.
Further investigation on the doping density and depletion thickness at elevated temperature reveals variation of these parameters with temperature. Figure 5.9 summaries the doping density and depletion thickness of the epitaxial devices from 350 K to 500 K.
The RD-50 sample displays very small variations in the slope of $1/C^2$ against voltage as well as depletion thickness for different temperatures. Figure 5.9a shows that the effective doping density from 350 K to 500 K increases with just $0.3 \times 10^{14}$ cm$^{-3}$ while the depletion thickness is about the same. Comparing this figure with figures 5.7a and 5.7b, close inspection reveals that the depletion thickness reduces to about 50% of its value obtained at room temperature. However, there was only a very slight change in the doping density.
at all temperatures. As it will be observed in section 5.3, these effects did not cause any noticeable change in CCE of the RD-50 sample.

Interestingly, the remaining two epitaxial devices indicate significant variation in both effective doping and subsequently depletion thickness at elevated temperatures. Figure 5.9b shows an increase in $1/C^2$ of the order of 10 compared to the values obtained in figure 5.7a. At 350 K, it attains a maximum value of $17 \times 10^{19}$ cm$^{-3}$ at -25 V. However, the points tend to get scattered further from the central fitting curve beyond -15 V and, this may have been due to the difference in the equipment used and the nature of contacting. Accordingly the depletion thickness increases of up to 3 times the original value obtained at room temperature using different equipment and contact method. It should be noted that capacitance voltage relationship under high temperature used to estimate the parameters was measured using slightly different contact method and different equipment. Therefore, it is not out of place to have some reasonable disparity in the results.

In the case of the EPI sample, the value of $1/C^2$ against voltage at 350 K is approximately similar to that obtained at 300 K (see figures 5.9c and 5.7a). At temperatures higher than 350 K, the value reduces gradually up to 500 K. Similarly, there is a reasonable agreement in depletion thickness values at 350 and 300 K. Beyond 350 K this thickness gradually reduces to 3.6 µm at -30 V.

Figure 5.9d gives a summary of the doping density of all the epitaxial devices evaluated from the C/V data. Examining the figure, a consistent increase in doping density with increasing temperature is observable up to 500 K. In principle, electrons from donor atoms and valence band excited electrons crossing the band gap into the conduction band affect the average number of carriers in a doped semiconductor; thereby changing its equilibrium number of carriers [98]. This is achieved by increasing the temperature which in-turn frees more electrons that are weakly bond to donor sites.
5.3 Alpha spectroscopy

In order to examine the contribution of leakage current to the overall noise in the system, pulser and $^{241}$Am signals were recorded simultaneously and full width at half maximum (FWHM) was extracted at various applied voltages and temperatures. The FWHM values obtained from the pulser peaks for all the epitaxial devices are presented in ?? . The figure also illustrates the spectra acquired from both sources simultaneously for the SBD SiC device. Although the leakage current in the devices at 500 K is low, the large input resistance of the pre-amplifier (100 MΩ) [183] has caused a significant voltage drop. This voltage drop was corrected using equation 3.2.
As it can be observed from the figure, the pulser spectra acquired during the alpha irradiation indicate low FWHM fluctuation at high operational temperatures in the RD-50 sample (figure 5.10a). The FWHM from the pulser tends to be constant and significantly lower at 450 and 500 K than the room temperature value, even at 80 V bias. In contrast, FWHM increases to large values (more than 3 times of the initial value at room temperat-
5.3. ALPHA SPECTROSCOPY

ure) beyond 50 V from 300 to approximately 400 K. Nevertheless, at 50 V bias and below, the observed pulser signal width is between 19 to 20 Chan. for all temperatures. The most important part of this result is the decrease in FWHM with increasing temperature affirming that there is limited contribution of leakage current to the overall energy resolution of the system. This is consistent with observations of Kalinina et al. [28].

On the other hand, SBD sample pulser values show approximately the same FWHM values from 300 to 450 K at 60 V and below. At 500 K, the FWHM increases slightly from 40 to 50 V. Beyond this point, it increases by an average of 25% (figure 5.10b). It is evident from figure 5.10d that the broadening observed in the $^{241}$ Am source signal is quite large compared to the pulser peaks; indicating that the effect of leakage current to the overall peak broadening is minimal. Although there was generally an increase in FWHM with temperature and bias in figure 5.10b compared to figure 5.10a, beyond 50 V the values in the former are far less than those obtained in the latter for temperatures below 450 K. This behaviour of the SBD sample is connected to its configuration and small contact area as explained in section 5.1.

The third sample (EPI) shows the highest pulser FWHM values for all temperatures as can be seen in figure 5.10c. Due to the very large noise in the signals from the alpha source, simultaneous measurements with pulser beyond 10 V at 350 and 400 K as well as 20 V at 400 and 500 K were not practicable. This could be ascribed to the long term exposure of the sample to high temperature during the stability measurements which damaged the Schottky contact. It is important to note that the time for recording the number of counts in figure 5.10d vary from one bias voltage to another; hence the difference in pulse height of the pulser signal.

It is thus very important to carefully decide on the nature and method of contact formation on a substrate intended for high temperature applications. To date there is no standard metal contact thickness and deposition method for devices intended for high
temperature applications. In this work, the metallization of RD-50 seems to offer an excellent performance at elevated temperatures. If a similar method is applied to a pixel device such as the SBD sample, a superior performance is expected. Recent studies carried out by Bell et al. [184] on Cadmium Zinc Telluride to investigate the variation of fabrication parameters in electroless deposition, raised more questions than providing answers to the current problem. As such, thorough investigation is necessary to demystify the critical factors that can improve or deteriorate the performance of semiconductor detectors at elevated temperatures.

The CCE of the devices approaches 90% for most of the temperatures with the SBD sample achieving the highest CCE value (see Fig. 5.11).
Surprisingly, there is remarkable improvement in resolution at higher temperatures in the RD-50 sample. At about 60 V the resolution is less than 15% (figure 5.11a). Comparing this result with that obtained in figure 5.10a, it can be observed that the levelling up of the FWHM at elevated temperatures influenced the resolution of this sample. The drastic improvement in the resolution between 10 and 20 Volts indicates increase in charge collection as a result of limited leakage current and improved electric field in the depletion

Figure 5.11: Charge collection efficiency (CCE) and resolution for 4H-SiC epitaxial devices from 300 to 500 K for (a) RD-50, (b) SBD and (c) EPI.
layer. Leakage current is known to be an important factor influencing the resolution of semiconductor detectors.

In the case of the other two epitaxial devices (SBD and EPI) CCE values reduce with reducing temperature as shown in figures 5.11b and 5.11c. The resolution in both SBD and EPI samples is approximately the same; having very low values at low voltages. The resolution continues to decrease down to an average of 23% at 60 V and subsequently remains fairly the same for the remaining voltage. The increase in CCE might have been influenced by the reduction in depletion thickness with increasing temperature (already discussed in section 5.2) as reported by Babcock and Chang [22]. Therefore narrower depletion thickness and higher fields could cause increase in CCE and vice-versa. The RD-50 sample shows excellent response to alpha particles in terms of its resolution at all measurement temperatures compare to SBD and EPI samples. On the other hand, the SBD sample displays better CCE due its small capacitance which implies large signal to noise ratio.

5.3.1 Alpha detection Stability at high temperature

Most of the 4H-SiC epitaxial devices studied show stable response in terms of energy, count rate and resolution at various temperatures and bias voltages. Figure 5.12 shows the stability of the RD-50 epitaxial SiC device monitored under high temperature and extended period.
5.3. ALPHA SPECTROSCOPY

(a) Count rate of $^{241}$Am alpha source for RD-50 detector from 300 to 500 K measured at 100 V bias for 24 hours

(b) Energy and resolution of $^{241}$Am alpha source for RD-50 detector from 300 to 500 K measured at 100 V bias for 24 hours

Figure 5.12: Count rate, energy and resolution of RD-50 sample irradiated with $^{241}$Am alpha source from 300 to 500 K for RD-50 sample. The voltages indicated here are not corrected.

As seen in the figure, the detected energy of the $^{241}$Am alpha particles at 100 V and 500 K increases from 3.35 to 3.38 MeV over 8 hours and remains stable afterwards (figure 5.12b). Furthermore, the count rate variations after the first 8 hours is insignificant (with standard deviation of 0.0024 cps) and a similar trend is observable in the resolution. At 450 K and 100 V bias the detected energy reduces from 3.55 to 3.53 MeV over 10 hours and remains stable afterwards. The response of the device at 300 K and 100 V bias was slightly unstable; having an increase in energy after three hours and subsequently started reducing after 12 hours. Under this condition, the sample only reaches a stable value after approximately 17 hours. The difference in the time to reach stability for 300 K, 450 K and 500 K may be a result of charge trapping and de-trapping which could have reached an equilibrium state quicker at 500 K. In addition, the resolution at 500 K and 100 V bias is more stable and far better than that observed at lower temperatures. count rate of the device appears to be stable over the 24 hour period. Therefore, it is evident from this result that the device becomes more stable with increasing temperature.
Response of the SBD sample to $^{241}$Am is illustrated by figure 5.13a. There is excellent stability in the count rate for most of the temperatures and applied voltages. Longer time for stability of the count rate was observed at 400 K and 50 V compared to other temperatures. At this temperature, the sample stabilises just after the first 5 hours with a little increase in the count rate from 84 to 96 cps. Comparing figure 5.12a to 5.13a, it is apparent that count rate stability is better in SBD with very little disparity in the distribution of the points from the average value.

This device demonstrates better stability in the detection of $^{241}$Am alpha source energy at 300 and 500 K than other temperatures. Between these temperatures, the stability is deteriorated as detailed in figure 5.13b. Similarly, the resolution at these temperatures has better stability compared to other temperatures. The approximate resolution value of this device at 500 K and 100 V bias is 40% larger than that obtained in the RD-50 sample.

Figure 5.14b provides the response of the EPI sample as a result of exposure to temperature and $^{241}$Am alpha radiation at different applied voltages. The device was exposed...
to 300 and 400 K only because, at 500 K it was very noisy to such an extent that no resolved peak could be obtained as explained earlier in figure 5.10c. However, at these two temperatures, the count rate from the device is stable both for 50 and 100 V bias as depicted by figure 5.14a.

![Count rate of $^{241}$Am alpha source for EPI sample from 300 to 500 K measured at 100 V bias for 24 hours](image1)

![Energy and resolution of $^{241}$Am alpha source for EPI sample from 300 to 500 K measured at 100 V bias for 24 hours](image2)

**Figure 5.14:** Count rate, energy and resolution of EPI device irradiated with $^{241}$Am alpha source from 300 to 500 K.

Energy stability was excellent at 300 and 400 K for 100 V bias and fairly constant for 50 V bias. The sample’s resolution at 100 V bias reduces initially (with increase in its value from 22.8% to 23.0%) for 3 hours and eventually remain stable for the remaining period. Conversely, at 50 V bias, the resolution is not stable up to approximately 23 hours.

So far, the RD-50 sample displayed an exceptional stability characteristics and excellent resolution compared to the SBD and EPI devices. Therefore, this sample has best potential for high temperature neutron detection using converter layer. Despite the fact that the CCE of SBD device is exceptionally good, it can be a little bit compromised for resolution.
5.4 Summary

Generally speaking, the leakage current from most of the devices characterised is not significant, as it was possible to perform alpha spectroscopy with considerable resolution. In fact as it was shown, the leakage current in RD-50 sample has less influence on its energy resolution. The pixel device had smallest magnitude of leakage current at all temperatures. This is attributable to its configuration. While the resistivity of RD-50 varies slightly with temperature, significant variation was observed in the remaining devices. The contrast in behaviour of IF and BH between RD-50 sample and the remaining two epitaxial samples is of particular interest. But since EPI and SBD were diced from the same wafer, their similarity in IF and BH variation with temperature is expected. In the case of stability of leakage current, the SI sample appears to have faster stability than the RD-50 sample. However, after the the current stability test, the SI sample failed during the alpha stability test. This may be due to variation in fabrication parameters or other processes taking place in the metal-semiconductor interface.

Doping density variation with temperature in RD-50 was approximately the same and that has reflected in its CCE variation with temperature. On the contrary, considerable variation of effective doping with temperature was observed in EPI and SBD which explains the reason behind reduction of their CCE with temperature increase.

Alpha detection stability studied at elevated temperature for 24 hours reveals improvement in stability with increasing in temperature in virtually all the epitaxial devices. The stability was assessed in terms of count rate, peak energy and its resolution. RD-50 has better resolution at 500 K compared to EPI and SBD samples. Also at 500 K the peak energy was more stable than at lower temperatures. Although a similar result could be observed in the EPI and SBD samples, their resolution on the average decreases with increasing temperature and voltage. Nevertheless, this reduction in resolution has not significantly
affected the CCE of the devices.

These results demonstrate the potential of SiC devices for alpha particle detection at high temperature. It also indicates that, they are good candidates for thermal neutron detection at high temperature. Based on the overall result in this chapter, the RD-50 was chosen for thermal neutron detection at high temperature because its CCE does not change significantly with temperature.
Chapter 6

Thermal Neutron detection stability with Si and SiC detectors at elevated temperatures

One of the main areas of intended applications in this project is nuclear well logging as stated in chapter 1. This method utilises the interaction of thermal neutrons with the detector element; through which an equivalent quality and quantity of a formation can be determined (otherwise known as hydrogen index). In the following discussion, results obtained from silicon and silicon carbide detectors are presented and analysed. Also of interest is the stability of the SiC devices to detect the thermal neutrons at elevated temperatures for extended periods. The neutron source used was $^{241}$Am-Be with approximate flux of $1.2 \times 10^6$ n/s and average activity of 18.5 GBq.
6.1 Thermal neutron detection at room temperature

Initially, a silicon detector was used to detect thermal neutrons in order to confirm the expected room temperature response to the neutron source. Although Barbagallo et al. [66] have suggested using removable converters on semiconductor detectors, by depositing the LiF on a thin layer (aluminium foil or polyethylene) and placing it close to the surface of the silicon detector, the temperature requirement of the measurement demands a different approach. In this work the LiF was deposited directly on the surface of the detector. Figure 6.1 shows the spectra recorded from the Si (IAEA sample 8) detector at room temperature. As can be seen in the figure, when the cadmium sheath is placed between the neutron source and the detector, a few counts are recorded. This indicates that the peak at about 2 MeV is indicative of thermal neutrons; resulting from neutron reaction with LiF. The large peak is believed to be for tritons as reported by Barbagallo et al. [66] and the alpha peak is located around 1.5 MeV. The reduction in the energy is due to loss in energy of the particles as they travel through some part of the converter layer.
CHAPTER 6. THERMAL NEUTRON DETECTION STABILITY WITH SI AND SIC DETECTORS AT ELEVATED TEMPERATURES

Figure 6.2: Spectral responses of (a) RD-50 SiC sample and (b) SBD SiC sample to thermal neutrons. The spectra were recorded for 20 hours each under 100 V bias. Due to increase in noise at 500 K, the applied voltage was reduced to 70 V.

and metal contact. Silicon carbide detectors evaporated with LiF were also used for the detection of thermal neutrons at room and elevated temperature. The SBD sample was used at room temperature while the measurement with RD-50 was extended to 500 K. The spectra acquired with both detectors are presented in figure 6.2. Effect of Cd blocking the slow/thermal neutrons can be seen clearly in both figures 6.2a and 6.2b. Because of superior resolution of RD-50 compared to the SBD device as discussed earlier in section 5.3, two distinct peaks are apparent in the figure. On the other hand, the spectra acquired from SBD sample indicates presence of thermal neutrons. However, due to lack of resolution, the peaks are not properly resolved. An attempt to estimate the count rate normalised to the are of each device was performed. The range of the integrated area for Si and SiC samples is 0.8 to 2.3 MeV and 0.9 to 2.1 MeV respectively. A summary of the count rates estimated from the three devices (Si-8, RD-50 and SBD) is presented in table 6.1.
6.1. THERMAL NEUTRON DETECTION AT ROOM TEMPERATURE

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature (K)</th>
<th>Count rate (cph/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si-8</td>
<td>300</td>
<td>70.60 ± 0.12</td>
</tr>
<tr>
<td>RD-50</td>
<td>300</td>
<td>156.89 ± 0.08</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>144.64 ± 0.08</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>153.57 ± 0.08</td>
</tr>
<tr>
<td>SBD</td>
<td>300</td>
<td>34.83 ± 0.17</td>
</tr>
</tbody>
</table>

Table 6.1: Summary of count rate of different semiconductor samples studied at room and elevated temperatures. The count rate was normalised to the active area of each device.

According to the table, count rate from RD-50 SiC is the highest followed by Si-8 and the least is SBD. This is attributable to the thickness of the converter layer. While the thickness used for SBD sample is a little smaller, that of Si-8 is a bit larger with the result that most of the alpha particles are lost. This is because the alpha particles cannot travel through long distances. The SBD sample suffers poor resolution compared to both Si-8 and RD-50. Therefore, a descent peak was not achievable. In this analysis, the a sum of counts per hour within the range of energies stated earlier in this section was determined and used an the relative flux estimate. The average flux of 4 n/s/cm² for a Si device was reported by Barbagallo et al. [66]. However in their work the $^{241}$Am-Be neutron source emits an average $5 \times 10^4$ n/s; which is less than the flux from the source used in this project with about 100 n/s. nevertheless, the thickness of the LiF used was 1.5 $\mu$m removable converter. Thus this indicates that the efficiency of the silicon is a much less than expected. As such the alpha particle energy loss might have contributed to this. Comparing RD-50 with both Si-8 and SBD, it can be observed that the thickness of LiF in this case is about right; showing the expected peaks from alpha and triton in the spectra.
6.2 Thermal neutron detection stability at elevated temperature

Excellent CCE and stability of alpha particle detection at elevated temperatures of RD-50 sample as discussed in chapter 5, informed its selection for thermal neutron stability studies. The spectral responses acquired from this device for temperatures ranging from 300 to 500 K for 100 hours is presented in figure 6.3.
6.2. THERMAL NEUTRON DETECTION STABILITY AT ELEVATED TEMPERATURE

Figure 6.3: Spectral responses of RD-50 4H-SiC sample evaporated with 3.5 \(\mu\)m LiF. The signal was acquired for 100 hours at (a) 300 K (b) 400 K and (c) 500 K. The applied bias for 300 and 400 K was 100 V. Due to increase in noise at 500 K, the applied voltage was reduced to 70 V. Unresolved peak can be seen also at 500 K due to poor signal to noise ratio. The counts in the spectra are cumulative.

According to the figure, two resolved peaks are observable at 300 and 400 K respectively (see figures 6.3a and 6.3b). The first peak is attributable to alphas while, the second one is for tritons in line with [185]. In principle, tritons have a longer range compared to alphas. Therefore, some of the tritons will pass through the detector volume depositing only a
small amount of their energy as compared to alpha particles. But most of the alphas will get detected due to their short penetration range. However, in principle, the energy of tritons from this type of reaction is larger than that of the alphas. The spectra presented here are for gross counts (i.e. without subtracting the final from initial counts otherwise known as stripping). The stripped spectra are shown in figure 6.4.
6.2. THERMAL NEUTRON DETECTION STABILITY AT ELEVATED TEMPERATURE

Figure 6.4: Spectral responses of RD-50 4H-SiC sample evaporated with 3.5 µm LiF. The signal was acquired for 100 hours at (a) 300 K (b) 400 K and (c) 500 K and (d) a summary of counts from 20 to 100 hours at 300 and 400 K. Here the spectra are stripped from their initial value. The prime indicates that, each spectrum represents counts for 20 hours.

As detailed in the figure, the spectra were stripped from their initial values in order to have exact response for 20 hours in each case. Nearly constant counts can be seen in figures 6.4a and 6.4b after respectively. Also, the peak positions remain constant for the 100 hours. The noise component of the signal at 500 K after 80 hours was larger than the signal itself;
with the result that a descent spectrum cannot be recorded. Therefore, it is not included in figure 6.4c. Although poor resolution was demonstrated by this device at 500 K, there exist appreciable consistence in its spectral responses. Due to this same reason, estimation of number of counts at 500 K was not possible. A summary of the net counts from 20 to 100 hours at 300 and 400 K is presented in figure 6.4d. Although the counts at 300 K seems to be higher, it is worth noting that the fitting of the peaks was not perfect so that the absolute counts are subjected to uncertainties from one temperature to another. However, in terms of comparing stability from 20 to 100 hours for a particular temperature, this data is valid. Thus as reported earlier is section 5.3.1, the figure shows that more stability of thermal neutron detection is possible with higher temperature.

6.3 Summary

Thermal neutron detection was demonstrated using silicon (Si) and silicon carbide (SiC) devices. Excellent stability of SiC for thermal neutron detection at elevated temperatures has been demonstrated; with low resolution due to high leakage current at 500 K in air case. The efficiency for detecting thermal neutron from these devices has been one of their major limitation. However, the findings presented highlights the potentials for the use of these as thermal neutron counters.
Chapter 7

Conclusion and Future work

The Si devices studied have shown relatively low leakage current of the order of nA/cm$^2$ at room temperature. The resistivity estimated from silicon devices agrees well with the these values supplied by the manufacturers (see table 3.1). The discrepancy in leakage current in Si detectors may be as a result of some unavoidable surface defects during the process of cleaning, mounting and wire bonding. The peaks observed in the C/V curves of some Si devices, which are dependent on both temperature and frequency, could be related to the defect traps observed in the DLTS signals. Generally, excellent charge transport was observed from all the samples studied with approximately 80% CCE.

The damaged p-type epitaxial silicon samples which have undergone irradiation with high fluence of protons, show drastic reduction of CCE up to an average of 70% for the highest value of fluence used. Although it is convenient to use liquid nitrogen for alpha spectroscopy of damaged semiconductor detectors, care must be taken in choosing the right temperature of measurement so that charge carrier transport in the devices is not undermined by freezing up of carriers.

In the case of SiC samples the current density is order of nA/cm$^2$ at room temperature and few $\mu$A/cm$^2$ at high temperature. The SBD sample has the lowest current density,
even at elevated temperature. Its low current density is believed to be related to its pixel configuration. Therefore, future performance of high temperature SiC devices for alpha particle detection can be improved with well structured pixel devices. Resistivity variation with temperature is smaller in RD-50 compared to EPI and SBD devices and a similar pattern was observed in the effective dopants and depletion thickness. BH and IF have similar trend for EPI and SBD samples while, a contrast was observed in RD-50 samples. It should be noted that EPI and SBD samples were diced from the same wafer. Current stability of the RD-50 sample which is an epitaxial device is a bit slower than in the SI which is a semi-insulating device. However, repeated measurements are possible with the epitaxial device.

An average of 90% CCE was observed in most of the SiC devices. Nevertheless, this value increased a little bit at high temperature in SBD and EPI samples with RD-50 remaining fairly constant. The reduction in CCE with temperature could be explained in the light of variation of resistivity, effective dopants and depletion thickness of these devices when subjected to high temperature.

The thermal neutron detection and its stability with SiC devices was successful even at high temperature. Although the counting observed was limited to 100 hours due to equipment sharing, the first hundred hours indicate stable response at all temperatures. Therefore, these devices have potential for application in areas where temperature is a challenge; such as oil and gas prospecting and nuclear reactor monitoring.

Although it has been established in this work that the SiC devices do not only have the capability of detecting alpha and neutrons at high temperature but, they also prove to be stable for extended periods of application, high temperature stable readout systems are still posing considerable challenges. In order to have a complete solution, there is need for a suitable pre-amplifier and other electronics such as spectroscopy amplifier, that can withstand high temperature for longer period. However, a good attempt have been made
by Hedayati et al. [186] to develop wide temperature range integrated amplifier. In spite of the few short comings, their result is promising.

Further investigation is recommended with various SiC samples fabricated using diverse range of metals for contacting as well different parameters; such as method of deposition, thickness of metal contact and configuration/dimension. A full surface study to demystify the metal contact interaction with semiconductor surface is necessary to realise high temperature SiC detector with optimal performance. Also, modelling the conditions and device modelling will go a long way in reducing time taken in the laboratory.
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Appendix A

List of publications


Appendix B

JLS Sputterer settings

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Table B.1: Settings used for SiC fabrication using the JLS sputterer