Development of a Silicon Carbide Schottky Diode Detector for Use in Determining Actinide Inventories based on Alpha Particle Spectroscopy

THESIS

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Abstract

Deployment of Generation IV nuclear power will require novel materials as well as new techniques for monitoring plant functions. Reprocessing of spent nuclear fuel will play a large role in the future generations of reactors. It would be useful if real-time measurements of actinide concentrations during reprocessing could be achieved through alpha spectroscopy. Devices built upon silicon carbide offer the best hope of surviving the harsh chemical, and high radiation and high temperature environments associated with reprocessing.

In this project 4H-SiC alpha particle detectors were built and tested up to high temperature in order to simulate some of the conditions a detector might encounter in the pyroprocessing environment. The silicon carbide used in this project had a low-doped (5x10^{14} atoms/cm^{3} of nitrogen) 21 micron thick epitaxial layer grown on top of a 300 micron thick bulk substrate with a nitrogen doping concentration of 1x10^{18} atoms/cm^{3}. 100 nm of nickel, 10 nm of titanium, and 10 nm of gold were deposited, in that order, on the epitaxial side and annealed to form a Schottky contact. An ohmic contact was formed by annealing from 100 nm of nickel deposited on the substrate and annealed. The devices were characterized through IV curve measurements and then exposed to Am-241 alpha particles and high temperature while in vacuum.
Mixed results show that these detectors can operate up to 100 degrees Celsius with very little change in peak location, full width half max (FWHM) and energy resolution. The FWHM remained below 100 keV, while the energy resolution was below 1.57%. At 150 degrees Celsius, the FWHM remained below 190 keV while the energy resolution was below 3.46%. Temperatures above 150 degrees Celsius pose significant problems to the current design as the alpha peak moves to lower channels and broadens to have a full width half max between 300 and 500 keV. The current state of literature on SiC Schottky diode detectors shows that no other Schottky diode detectors have been tested up to higher temperatures with a better resolution.
Dedication

This document is dedicated to my family.
Acknowledgments

I would like to thank all of the professors, staff, students and The Ohio State University for their help, support and guidance throughout my time at the university. I would like to especially thank the staff at the Nanotech West Laboratory, specifically Paul Steffen, Aimee Price, Dr. John Carlin and Dr. Robert Davis. Without their help and guidance I would not have any working detectors for this project. Furthermore, I would also like to say thanks to the staff at Edison Welding Institute for their guidance on high temperature joining technologies especially Shankar Srinivasan and Tim Frech.

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Chapter 1 Introduction

Silicon carbide (SiC) has long been thought to be part of the next generation of materials to be used for structural components as well as for radiation detection in the coming generations of reactors. Its mechanical and electrical properties, as well as its wide band gap, make it perfect for use in harsh environments. Structural components and electric devices fabricated from silicon carbide have been shown to be able to withstand high temperatures, chemically harsh environments and operate in high radiation areas. As a member of the semiconductor family, silicon carbide is also said to be radiation hard, which means that it is resistant to damage caused by ionizing radiation. This makes it a very attractive candidate for in-core radiation detectors as well as for use in other highly radioactive, corrosive environments.

The primary goal of this project is to construct a silicon carbide based Schottky diode alpha particle detector. Some of the Generation IV reactors and associated technology currently being designed plan to use online or off site fuel reprocessing as a key component. A detector such as the one developed by this project would be useful to the next generation of reactors as the detector will be able to take real-time actinide inventories in molten fuel based on alpha particle spectrometry.

In order to perform such spectroscopic functions in fuel reprocessing streams, a SiC detector must have stable electrical contacts, be able to withstand high temperature,
chemically aggressive and radiation intense environments and provide good energy
collection efficiency. This project was based on a previous design (Ruddy
2005) and seeks to optimize the results. Contact materials and thicknesses were chosen;
contact patterning was optimized based on numerous trials; and preliminary calibration of
the detectors was preformed. These experimental results were then compared to those
predicted by theoretical calculations.
Chapter 2 Background

As a member of the semiconductor family with a rank of 9 on the Mohs hardness scale, silicon carbide has been utilized in a number of ways in the past. Present since the formation of the universe, this material was probably first synthesized in the mid 1800s. One of its earliest uses was as an abrasive. Today, silicon carbide is a valuable substance in research and industrial applications and is even considered to be better suited for electronics than diamond (Brezeanu 2005).

2.1 Pyroprocessing

Determining actinide inventories for reprocessing is an important part of the advanced fuel cycle. Currently, there are two different methods of reprocessing spent nuclear fuel (SNF): aqueous processing (which includes the PUREX process) and pyrochemical processing. Both methods have benefits and drawbacks, but using a pyrochemical choice allows the process to be more compact and can possibly be done on the same site as the existing nuclear power plant. There is not necessarily a direct separation of plutonium, since low purity products are acceptable for use in fast reactor
fuel. The molten salt in which the SNF would be dissolved is resistant to radiation effects, allowing for a shorter cooling time of spent fuel before reprocessing (Li 2007).

Pyroprocessing has been extensively studied at Idaho National Laboratory (INL).

Figure 1. Basic scheme for the electrorefinement process in pyroprocessing of spent nuclear fuel (Li 2007)

The reprocessing environment is very harsh for any structural or detection related materials, making SiC a good choice for detector components. The light water fuel introduced for reprocessing will be chemically reduced and dissolved into a LiCl-KCl-UCl₃ bath. In the Mark-IV Electrorefiner under development by INL, the salt solution is about 10 wt% UCl₃ and operates around 500 degrees Celsius (Li 2007). Materials are subjected to high heat, corrosive chloride salts, and high-energy radiation from predominantly gammas and betas. A detector placed in this environment must be able to
withstand these conditions while still performing its desired function. In this case, the desired function is to determine actinide inventories, specifically uranium and plutonium.

2.2 Silicon Carbide Devices

As previously discussed, silicon carbide is promising for use in the next generation of electrical devices which can operate in mild or harsh environmental conditions. Semiconductor device technology has evolved greatly over the past few decades and continues to evolve. SiC can now be grown in relatively large wafer sizes with relatively low defect densities. Some of these defects, such as micropipes, severely inhibit the intended functions of the device.

2.2.1 Electrical Properties

Semiconductors by definition are materials that have electrical conductivity greater than insulators but not as large as metals at room temperature. At high temperatures, the conductivity of semiconductors approaches that of metals. SiC is a wide band gap semiconductor material, which makes it better for high temperature applications than its smaller band gap relatives. A smaller band gap device would have more noise associated with it at high temperatures, due to thermal excitations. Insulators have the largest band gaps ranging between 5-8 eV while metals have no band gap with semiconductors in between with gaps ranging from a few tenths of an eV (e.g. InSb) up
to > 3 eV (SiC) and even 6.8 eV (AlN) at room temperature. The smaller the band gap means the easier it is for electrons to be thermally or otherwise excited from the valence band into the conduction band giving rise to mobile carriers and thus current.

The various polytypes of silicon carbide have different band gap energies based on their crystal structures. The 4H SiC polytype, which is used in this project, has a band gap as large as 3.26 eV while 3C SiC has a band gap as small as 2.36 eV (Almaz 2010). A comparison of some of the common materials used in electrical devices for various purposes can be seen in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>4H SiC</th>
<th>GaAs</th>
<th>Si</th>
<th>Ge</th>
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<td>8.00</td>
<td>3.26</td>
<td>1.42</td>
<td>1.12</td>
<td>0.66</td>
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Table 1. Comparison of band gap sizes for common materials used in electrical devices (Almaz 2010)

2.2.2 Diode fundamentals

There are many types of diode detectors on the market but the two main types include pn-junction detectors and Schottky barrier diode detectors. Most semiconductor devices require some sort of voltage biasing in order to operate. Schottky barrier diode detectors must be operated in reverse bias mode. Reverse biasing increases the depletion region thickness. A diagram showing the forward and reverse biasing conditions can be seen in Figure 2 for a pn-junction detector. The depletion region is sustained without any significant current flow unless, ionizing radiation interacts somewhere within the depletion region. Then the electron-hole pairs that are created in the depletion region
follow the applied electric field and are collected by the electronics of the system. If the
applied reverse bias voltage becomes too large, avalanching occurs. Then, the
“breakdown voltage” is said to have been found and current approaches infinity. An I-V
or current-voltage plot can be seen in Figure 3, which shows the relationship between
current and voltage for forward and reverse biasing conditions for a generic diode.

![Diagram of Forward Bias and Reverse Bias](image1)

Figure 2. Sample forward and reverse biasing conditions for p-n junction in
semiconductor detectors (Cao 2010)

![Diagram of I-V Curve](image2)

Figure 3. I-V curve for a diode showing breakdown voltage and reverse and forward
biasing conditions (Michelen 2011)

Schottky diode detectors are very similar to traditional p-n junction diodes.
Instead of having a semiconductor with a p-type and n-type doped regions, Schottky
diodes are formed through the union of an anode (metal) and cathode (n-type
semiconductor material). Schottky diodes are sometimes known as metal-semiconductor junctions or hot carrier diodes. The basic components of a Schottky barrier diode include a metal or Schottky contact, a semiconductor material, a depletion region and an ohmic contact, as seen in Figure 4.

![Figure 4. Basic metal-semiconductor junction (Gavryushin 2011)](image)

The Schottky barrier is formed as a result of differences in the work functions between the metal and the semiconductor material, where the latter needs to have the smaller work function (otherwise, an ohmic contact is formed). The work function is the minimum amount of energy needed to remove an electron from the Fermi level of the material to the vacuum level and has characteristic values for different materials. Metals and semiconductors have different energy bands as seen in Figure 5. \( E_{F,M} \) is the energy level of the Fermi band for the metal whereas \( E_F \) is the Fermi band energy for the semiconductor material. \( E_{\text{vacuum}} \) represents the energy of a free electron; \( E_V \) is the valence band energy and \( E_C \) is the conduction band energy. \( q\Phi_B \) is the Schottky barrier height and the work function of the metal is represented by \( q\Phi_M \) where \( q \) is the charge on an
electron. $q\chi$ is the electron affinity which is the energy difference between the conduction band edge and the vacuum level. The Fermi level and work function of a semiconductor material varies by type and with its doping density, whereas the work function has specific values for different metals.

Figure 5. Energy band diagram of the metal and semiconductor (a) before and (b) after connection (Gavryushin 2011)

In order for a Schottky diode detector to work, the work function of the metal needs to be larger than that of the semiconductor. When the materials are joined, some of the electrons from the semiconductor move into the metal, leaving behind holes or a positive charge. This creates a small, natural reverse bias. Furthermore, this separation of charge creates a strong negative electric field at the boundary of the metal and semiconductor. The volume over which this electric field is exhibited is known as the depletion region.

The behavior of the diode can be modified based on the direction of voltage biasing. An energy band diagram can be seen in Figure 6. It is interesting to note that without any biasing voltage, as in this case, a natural potential or depletion region exists
and is represented by $q\Phi_i$ in this diagram. As evidenced by Figure 7, applying a bias voltage to a metal-semiconductor diode can result in the shrinking or expansion of the depletion region based on the direction of bias. A forward bias shrinks the depletion region based on the size of the applied voltage, while a reverse bias increases the depletion region, again based on the size of the applied voltage. Furthermore, a forward biased diode allows current to flow through the device while a reverse biased diode stops the flow of current, which in combination acts as a rectifier.

![Energy band diagram for a metal-semiconductor connection at thermal equilibrium (Zeghbroeck 2011)](image)

Figure 6. Energy band diagram for a metal-semiconductor connection at thermal equilibrium (Zeghbroeck 2011)
In a reverse biased system where the depletion region has been established, radiation interactions can be detected. This type of device is sensitive to the amount of energy deposited in the depletion region. The incident radiation interacts with the semiconductor material in the depletion region and generates electron-hole pairs. The number of electron-hole pairs generated by the incoming radiation is proportional to the amount of energy the incident radiation has. The electrons and holes can be collected and analyzed by appropriate charge sensitive equipment to determine the energy of the incident radiation. It is imperative that the radiation deposits all of its energy in the depletion region in order to get an accurate measure of the particle’s total incident energy, since only there, a non-zero electric field exists (corresponding to the slope of the energy bands in Fig. 7) that acts on the created carrier pairs and creates a current pulse.
2.3 Previous Work

Radiation effects on silicon carbide have been extensively studied not only at The Ohio State University but also across the country and around the world. Recent work completed by Stephen E. Stone and Jonathan Kulisek at OSU have provided significant background for this experiment. Stone irradiated n-type, 4H SiC at The Ohio State University Research Reactor located on campus. The silicon carbide had two epilayers of 5 microns and 4.8 microns with nitrogen concentrations of $1 \times 10^{18}$ and $1.2 \times 10^{16}$ cm$^{-3}$, respectively. The 5 micron layer was grown directly on top of the substrate followed by the 4.8 micron thick layer. The samples were then irradiated with a 1 MeV equivalent neutron fluence of $3.75 \times 10^{11}$ cm$^{-2}$.

The goal of Stone’s research was to study the carrier concentration, mobility and resistivity of silicon carbide as a function of irradiation and annealing (Almaz 2010). He found that the carrier mobility decreased linearly with increasing 1 MeV equivalent neutron fluence. The carrier removal rate was approximately $48.5 \pm 6.3$ carriers cm$^{-1}$. In addition, he found that the mobility also decreased linearly with increasing 1 MeV equivalent fluence with a damage constant of approximately $(1.49 \pm 0.2) \times 10^{-19}$ V-s. These are important results to the current research because they demonstrate some of the deterioration we may see in our detector operations over time. It is not good enough to build a detector that works for a short period of time. To be useful for the next generation of reactors, these detectors must be functional for extended periods of times and their performance must be characterized.
Stone’s work produced some interesting results after annealing at a low temperature of 175 degrees Celsius. It was discovered that the carrier concentration was able to anneal within 10% of its pre-irradiated measurement. That is to say, the majority of the defects that contributed to free carrier removal annealed out. On the other hand, the mobility continued to worsen or decrease as the samples were annealed. The resistivity did not experience any dramatic changes with annealing and only decreased 8 ± 1% from its pre-irradiated value. The temperature effects on the electrical properties are important as SiC alpha particle detectors must operate in temperatures as high as 600 degrees Celsius in order to function in a pyroprocessing environment. It should also be noted that though the detectors built for this project will operate in high radiation fields and high temperature environments, the radiation will be predominantly gamma and beta and not neutrons as used in Mr. Stone’s study.

Besides an extension of Stone and Kulisek’s work, this project was also motivated by work previously done by Frank Ruddy. Ruddy’s work also concerned silicon carbide based Schottky diode alpha particle detectors (Ruddy 2005). These detectors used a Schottky contact made of titanium (800 angstroms), platinum (1000 angstroms) and gold (9000 angstroms) which was deposited on an 100-micron epitaxial layer with nitrogen doping of 1.2x10^{14} nitrogen cm^{-3} as seen in Figure 8. These detectors required a large bias voltage, over 1000 volts, to fully deplete the epitaxial layer. Furthermore, they were operated primarily at room temperature and were not subjected to the harsh environments associated with pyroprocessing. The detectors had a full width half max (FWHM) measurement of somewhere between 40 and 60 keV depending on the incident alpha particle energy and a leakage current on the order of nanoamps.
Figure 8. Detector designed by Dr. Frank Ruddy (Ruddy 2005)

In addition to the 2005 work by Frank Ruddy, some other works by F. H. Ruddy, A. M. Ivanov, N.B. Strokan and B. V. Kalinina were important for gathering information about the state of silicon carbide alpha particle detector research. Very little high temperature research has been done with silicon carbide Schottky diode detectors for alpha particle spectroscopy. Ruddy (1998) collected data on Schottky diode detectors up to 89 degrees Celsius and achieved energy resolutions of 5.8%. Another 2009 study, which was only done at room temperature, found energy resolutions of 0.37% (Ruddy). Similar results were confirmed by studies conducted in Russia and Sweden. For higher energy alpha particles of 5.1 to 5.5 MeV, an energy resolution of 0.34% was observed and is comparable to that of some silicon based detectors (Strokan 2005).

In addition to Schottky diodes, p-n diodes also based on SiC have been investigated for alpha particle detection. Kalinina (2008) tested p-n junction alpha particle detectors up to 140 degrees Celsius. A more recent article by Kalinina (and Ivanov and Strokan), which was published in 2011, tested similar aluminum ion-
implanted p-n junctions up to 375 degrees Celsius with good results. They found that up to 25 degrees Celsius, the detectors had an energy resolution of less than 2.0%. Between the temperatures of 25 and 300 degrees Celsius, the resolution improved to 1.35%.
Chapter 3 Methodology

3.1 Silicon Carbide Wafer Preparation

Alpha particle spectroscopy places certain constraints on the type of silicon carbide that can be used. For this application, a highly doped 4H silicon carbide wafer with 2.77 micropipes/cm² was purchased from Cree and cut 8 degrees off axis. The wafer has a resistivity measurement, characterized by Cree, of 0.019 ohm-cm. The micropipe density is an important consideration as micropipes are crystallographic defects, which severely impact detector function. The silicon carbide wafer was originally 2 inches in diameter before being diced into smaller pieces for this application. The SiC has a 300 micron thick bulk layer doped with 1x10¹⁸ atoms/cm³ of nitrogen. Grown on top of the bulk is a 0.5 micron thick epitaxial layer, with a doping of 1x10¹⁸ atoms/cm³ of nitrogen. Finally, a second epitaxial layer is grown on top with thickness 21 microns and doping 5x10¹⁴ atoms/cm³ of nitrogen. Based on these considerations, a 200V bias is required to create a fully biased or depleted charge collection region. When the bias is applied, the depletion layer has a thickness of approximately 21 microns or the full thickness of the epitaxial layer. This thickness should be large enough to capture an alpha particle with energy of 5 or 6 MeV.
After receiving the wafer from Cree, it was shipped to Kadco Ceramics in Easton, PA where it was diced into smaller device sized pieces ranging in size from 7mm x 7mm to 11mm x 11mm. The number of dies suitable for detector fabrication was optimized using a MATLAB code, which tried to create a balance between the highest number of usable pieces and the largest size of pieces possible, while avoiding all micropipe defects. A copy of the final, “best” iteration can be seen in Figure 9.

![Figure 9. Optimized SiC wafer die cutting scheme](image)

Unfortunately, the dicing of the silicon carbide was not as straightforward as it originally appeared. Multiple misunderstandings and mistakes led to future difficulties in the fabrication process. More details related to this experience are outlined in Appendix C.
3.2 Silicon Carbide Metallization

A sample holder was fashioned out of aluminum for depositing contacts on the newly cut silicon carbide see Figure 10. It has removable masks, seen in Figure 11, for creating various sized Schottky contacts, including 3 mm, 6 mm and 8 mm in diameter. The sizes were chosen to allow examination of the relationship between the contact area and detector performance.

Figure 10. Aluminum sample holder for metallization of SiC detectors
Prior to metallization, the silicon carbide pieces were washed according to the industry standard processes of RCA cleaning, which is very similar to the SC-1 and SC-2 washes. These processes remove organics, oxides and ionic deposits and growths. A more in-depth description of the wafer washing process can be found in Appendix A. It is imperative that the Schottky contact be deposited directly on the silicon carbide. Any extra thickness caused by oxide growth on the die will reduce the energy of the incoming alpha particles and interfere with the total collection of charge. Furthermore, metal deposited on an insulator can no longer be considered a true Schottky contact. The addition of the high dielectric material will require a greater electric field across the contact in order to perform.

Metal was deposited on both sides of the silicon carbide using a Denton 502A Evaporator (Figure 12) available in the Nanotech West Cleanroom at The Ohio State University. The cleanroom is a class 100 cleanroom and provides the necessary capabilities for complete device fabrication. On the epitaxial face, a Schottky contact was deposited; while on the bulk, an ohmic contact was deposited. Originally, pure nickel was chosen for both contacts. Nickel seemed to be an ideal choice as it has a
relatively low nuclear activation cross section and can easily be made into an ohmic or Schottky contact depending on the annealing temperature. An ohmic contact needs to be annealed at 950 degrees Celsius and a Schottky contact at 650 degrees Celsius. Both contacts were annealed at Nanotech West using the available rapid thermal annealer, Figure 13. Upon further consideration, it was decided that the Schottky contact would be predominantly nickel but have a small layer of titanium and gold on top to reduce oxidation of the contact at elevated temperatures. A more detailed procedure of evaporations and annealing can be found in Appendix B.

Figure 12. Denton 502A evaporator at Nanotech West, located in Bay 4
3.3 Silicon Carbide Detector Construction

3.3.1 Detector Packaging

In order to provide packaging for the detector, alumina substrates were ordered from Ceteck Technologies in Poughkeepsie, NY. They were then metalized and the fabricated silicon carbide detectors were attached to them. Despite its brittleness and ability to experience thermal shock, alumina was a suitable choice for packaging as it provides high thermal conductivity combined with excellent electrical insulation. This is ideal for use with the purchased heater, as the detector needs to be electrically isolated from the heating surface, so as not to short out the heater while still allowing the detector to be heated to 600 degree Celsius.

Using the Denton 502A Evaporator again, 200 nm of pure gold contact pads were deposited on the alumina substrates. The alumina pieces are about three times the size of
the largest detectors at 1.13” x 1.13” x 0.032” or 28 mm x 28 mm x 0.81 mm, which should allow for ample space for the necessary electrical connections. The alumina is still small enough, however, to completely fit on the surface of the heater to provide even heating. A blueprint of the masks used to evaporate the metal contacts onto the alumina substrates and an actual substrate with deposited pads can be seen in Figure 14.

![Figure 14](image)

Figure 14. (L) Blueprint of mask to make alumina pads used for detector packaging and (R) alumina with metal deposited

### 3.3.2 Electrical Connections

In order to have an operational detector, electrical connections needed to be made to the Schottky and ohmic contact. These contacts were to be connected to the metalized alumina pad. From there, other wires would be drawn off and connected to a BNC
connector, which would provide the necessary bias voltage and signal collection capabilities. Originally aluminum silicon braze was chosen as it would allow the detectors to operate up to around 500 degrees Celsius. After many tests, it was found that the AlSi braze would not work and a new solution needed to be found. Finally, nickel paste or nickel cement from Ted Pella, Inc. was discovered and ultimately proved to be successful. According to the specifications, the paste can be used up to 538 degrees Celsius and in vacuum.

![Figure 15. Nickel paste (L) and ceramic alumina adhesive (R) from Ted Pella, Inc.](image)

The nickel paste’s relative ease of use and curing time made it a good choice. The most complicated part of the fabrication process was making extrememly small connections to the Schottky contact. These connections must be as small as possible so as not to interfere with the alpha particle collection area. In order to ensure good, small connections, it turned out to be necessary to work under a magnifying glass with small, 5/0 paintbrushes. The cement can easily be thinned with water, and dried cement that has
not been exposed to temperatures above 260 degrees Celsius can be rehydrated and removed.

First, the ohmic contact on the detector was painted with a thin layer of nickel cement along with a small area in the center gold circle on the alumina substrate. Once both were painted and still wet, the two pieces were put together and the extra cement, if any, was wiped away with the point of an X-acto knife. Slight pressure was applied to the detector after placing a dust free cloth on top to prevent scratching the Schottky contact and the detector was left to set for 5 to 10 minutes.

While the ohmic contact cured, the Schottky electrical connection was made. The Schottky contact was connected with 0.010” diameter Nickel 220 wire. This wire, which has impurities to give it a lower resistance, is a slight modification from pure nickel wire. It is also much more pliable and not as springy as pure nickel wire, making it much easier to use. With all nickel based wires, it is important to have a pair of non or anti magnetic tweezers. The Nickel 220 wire was bent so that it could make a connection between the Schottky contact and the smallest pure gold pad on the alumina. Once the wire was cut and shaped, a small blob of nickel cement was dabbed on each end of the wire using a paintbrush. Then it was quickly and carefully placed on the Schottky contact and then gold pad. Finally, it was left to dry.

Once the connections have air-dried for about 10 minutes or so, a voltmeter can be used to test the connectivity of the set-up. This is an essential step, since the full curing process requires heating at 93 degrees Celsius for two hours. It is better to find out that a detector is not fully connected before annealing than afterward. Furthermore, if
the nickel cement has not dried enough, the circuit will read as open. As the cement begins to dry, it completes the circuit.

Figure 16. Close-up of Detector 13’s connections; detector was not used

Also connected to each of the metal pads were longer wires that will be used to make electrical connection to a BNC connector. The BNC connector will transmit the signal as well as provide the -200V bias voltage needed for operation. To make this connection, Nickel 220 wire was also used in conjunction with the nickel cement. Once these connections were complete, the joints are left to cure in air again for about 10 minutes before placing the whole package in an oven to bake for two hours at 93 degrees Celsius. After curing, the long wires are slipped into very small Kapton tubes to electrically isolate them from one another. Both Kapton coated wires are then slipped into a tinned copper braid, Figure 16. Once fully assembled, the Schottky contact is connected to the center pin of a BNC connector and the ohmic side is connected to the
ground by soldering. The braid is grounded by soldering to the same ground as the signal of the BNC connector in order to provide RF shielding for the detector. Without this shield, the detector leads act as giant antenna. Prior to shielding the detector leads, the noise on a biased detector was on the order of 11 volts; after shielding it, the noise was on the order of tens of millivolts. A completed detector can be seen in Figure 17.

![Detector 13, which was not used, packaged for testing](image)

Figure 17. Detector 13, which was not used, packaged for testing

3.4 Vacuum and Alpha Particle Detection System Setup

While the ultimate goal of this project is to fabricate a detector that can operate in a pyroprocessing environment, testing and calibration must first occur in the lab so detector behavior can be understood.

3.4.1 Bell Jar Description

A bell jar vacuum system was built in Scott Laboratory at The Ohio State University. The base of the bell jar contains five ports, one for each of the following: vacuum pump, BNC for charge collection, heater control and power feedthrough,
pressure gauge and NPT electrical feedthrough. Most of the fittings are KF25 standard unless otherwise noted. While the pressure gauge feedthrough utilizes a thermocouple design, its connection to the bell jar is a non-standard connection. All of the connections, both standard and non-standard, are sealed with Buna-N or Viton O-rings. The L-gasket that surrounds the glass bell top is also made from Viton.

The vacuum pump used for this system is a rotary-vane roughing pump taken from an old glove box. The pump is able to evacuate the system to a pressure of 80-90 mTorr. While this is not a high vacuum environment, it should be sufficient to allow the alpha particles to be collected without a significant loss of energy. Unfortunately, the vacuum system is not good enough to sustain this vacuum pressure on its own. Once all of the valves are closed and the pump is turned off, the bell jar begins to lose pressure immediately.

3.4.2 In-vacuum Detection Set-up

Inside the bell jar, a stand from an old vacuum setup is used to support the heater, detector and alpha source. The stand has a circular base and two platforms separated by ceramic spacers. The ceramic spacers can easily be changed in order to increase or decrease the distance between the platforms. The lower platform supports the heater (described in Section 3.4.3) but is electrically isolated from the heater by two small alumina spacers. These spacers not only electrically isolate the heater but also slow down the process of heat rejection from the heater shroud to the metal plate. The outside
of the heater, its shroud, is grounded through a screw and copper wire which is connected to the stand which supports the experimental components in the bell jar. The higher platform supports a small aluminum holder made to fit a disk alpha source as seen in Figure 18. All of these features can be seen in Figure 19.

The alpha source used is slightly more than an inch in diameter. The two platforms are about 1.5” apart; but due to the height of the heater, the distance from the alpha source to the surface of the heater is about 0.5”. This is not necessarily detrimental as the alpha source has a relatively low activity and the more air that alphas must travel through, the more energy that they lose. The detector sits on top of a grounded copper sheet, which sits atop the heater, and is clamped down to increase thermal contact. This is done to reduce electrical noise from the heater picked up by the detector.

![Figure 18. Two views of alpha holder to be placed in top plate of bell jar stand](image)

Figure 18. Two views of alpha holder to be placed in top plate of bell jar stand
It is very important to consider electrical interference when building radiation detectors. In order to reduce electrical noise in this setup, the BNC connector has an isolated ground. This is beneficial, as the heating system has been grounded to the baseplate of the bell jar. It was eventually discovered that having the signal ground separate from the baseplate ground was not helpful. Thus, the baseplate ground and the signal ground were combined outside the bell jar, eliminating the need for an isolated ground BNC feedthrough. Outside of the bell jar, a small-holed metal cage covered in
aluminum foil was placed over the glass. The cage was also grounded to the baseplate by a copper wire so that it acts like a large Faraday cage and helps reduce the noise within the system.

As stated earlier, the detector sits on a large copper foil atop the heater, Figure 20. This copper foil is slightly larger than 1.6 inches in diameter, the diameter of the heating surface, and less than 0.01 inches thick. It is painted on one side with an alumina paste, Figure 20, so that it will act as an electrical insulator. The center of the heater is electrically isolated from its shroud; the heating surface is “hot” and the shroud is “grounded”. The heating surface cannot be directly connected to ground without shorting out the heater. The copper sheet is also grounded to act as a shield from the electrical noise generated by the heater. Without this sheet, the noise experienced by a biased detector is on the order of 11 volts. The sheet is grounded through a wire connected to a small tab on the shield and connected to the stand in the bell jar. Due to the fact that the alumina packaging of the detector is not directly in contact with the heater, a small thermocouple has also been cemented to the packaging. This thermocouple should give real time readouts of the detector temperature.
3.4.3 Heater Set-up

To have a functional experiment, the detectors must be heated to elevated temperatures to test their operations under extreme conditions. In order to do this effectively, two resistive substrate heaters were purchased from BlueWave Semiconductors in Maryland. One heater will be used for experiments in Scott Laboratory and the other will be used for experiments conducted at The Ohio State University’s Nuclear Reactor Laboratory. Two heaters will be essential in case one becomes too radioactive to leave the reactor lab in the future. The heater is made of Inconel and has a 1.6 inch heating surface with a 2 inch collar or shroud. It can operate under vacuum conditions up to $1 \times 10^{-7}$ Torr and easily heat up to 800 degrees Celsius, which makes it ideal for the proposed high temperature operations. The heater can be set
to its desired temperature by inputting a temperature and automatically allowing the control system to change the power to reach the set point or manually changing the power supplied to the heater. The temperature is accurate to within one degree Celsius of its target. Across the heating surface, there can be a temperature gradient of 3-4% at temperatures above 400 degrees Celsius. Below 400 degrees Celsius, the gradient is 0-2%.

The heater came with its own rack-mountable power supply and control system. The power supply system can be plugged into any generic outlet as the device calls for relatively low power. The heating element requires between 13 and 25 ohms resistance. The maximum voltage that can be applied to the system is 50 Volts. At this voltage, the maximum current can vary between three and five amps depending on the vacuum and/or gas load. An extra probe type thermocouple and readout were also purchased, as it was important to make sure that the alpha particle source, which will be in direct visibility of the heater, and other associated parts do not overheat.

3.4.4 Alpha Particle Collection System

The cable that carries signal between the Schottky diode detector and the Ortec 142B preamp is a one-foot RG62/U cable from Ortec. These cables have an impedance of 93 ohms as required by the preamp manual. Other RG62/U cables are used to view the signal from the E output of the preamp to the oscilloscope or to connect the E output directly to the Canberra DSA2000. When an alpha source is not used to generate a
signal, an Ortec 419 Pulser is used. RG62/U cables and a special adapter found on the back of the pulser are required for this connection into the input of the preamp. The special adapter found on the back of the pulser allows the pulser’s signal to behave like an energy signal from an alpha particle when properly connected. If the adapter cannot be located, the pulser can also be connected to the Test input of the 142B preamp.

After the Ortec 142B preamplifier, the signal is processed through a Canberra DSA2000. This digital signal analyzer shapes the preamplifier pulse into a trapezoid, which can be viewed using an oscilloscope, an RG58/U cable and the Monitor output of the DSA. The shaping of the trapezoidal filter can be modified using built-in controls in the data acquisition software. The rise time, flat top time, gain, pole zero, LLD, etc. can all be appropriately modified. It is very important to have the correct rise and flat top times as this directly affects the charge collection capabilities. Smaller rise times act as noise filters but also run the risk of encountering ballistic deficit. Longer rise times ensure that all energy from an alpha interaction will be collected but also allow more noise to pass, degrading the signal. Too small of a flat top time may mean that not all of the energy is fully collected as the flat top time helps account for slight variations in rise time. A longer flat top time may not be detrimental if the radiation source has a low-count rate. A high-count rate and long flat top time will limit the number of pulses that can be collected despite their rapid occurrence.

The counts and resulting spectrum are collected and recorded using the DSA2000’s Gamma Ray Spectroscopy collection system. Despite the system’s intention to be used as a gamma ray spectroscopic system, it can function quite well as an alpha particle collection system, too. The gain can be tweaked to help differentiate similar
alpha particle energies; however, it is important to keep in mind a known source’s location in a channel or energy bin so that multiple spectra results can be accurately compared. The channels can be calibrated using a well-defined source such as Am-241, which has an alpha peak at 5.485 MeV (84.8%) and a smaller one at 5.442 MeV (13.1%).

The collected alpha particle spectra should fit to a normal distribution and therefore should be able to be represented by a Gaussian fit of the form

$$G(x) = Ne^{-\frac{(x-\mu)^2}{\sigma^2}}$$

(1)

where N is a normalization factor, µ is the mean and σ is the standard deviation.

Assuming that the data fits a normal distribution, the full width at half max (FWHM) is related to σ by the expression

$$FWHM = 2\sigma\sqrt{2 \times \ln 2}.$$  

(2)

In the case of americium-241, there are two alpha particles that are emitted have very close energies. The primary alpha particle (85%) has an energy of 5.845 MeV while its nearest neighbor (13%) has an energy of 5.442 MeV. The closeness in energy can force the alpha peak to be fit to the sum of two Gaussians of the form

$$G(x) = N_1e^{-\frac{(x-\mu_1)^2}{\sigma_1^2}} + N_2e^{-\frac{(x-\mu_2)^2}{\sigma_2^2}}$$

(3)

where each µ and σ correspond to each of the fitting parameters associated with the two Gaussian peaks.

Data will be taken at intervals of 50 degrees Celsius so as to closely track the changes in the alpha particle detector and subsequently fit in MATLAB. At elevated temperatures it may become necessary to cycle the heater on and off in order to reduce noise. If it is necessary to turn off the heater, spectra will be counted for some time.
related to a change in temperature of 10 degrees Celsius of the detector. For example, if the heater needs to be turned off at 300 degrees Celsius then data will be taken between 305 and 295 degrees Celsius. Normally, spectra will be counted for 3000s but in the event of heater cycling, data will only be taken for 1500s due to time constraints. Cycling the heater requires as much as or more time to collect than a traditional 3000s spectrum.
Chapter 4 Results

This section outlines the results achieved in this experiment. The results fall into four main categories: detector fabrication, current-voltage (IV) measurements, theoretical alpha energy spectra and experimental alpha particle detection.

4.1 Detector Fabrication

As previously stated, the process of detector fabrication was a bumpy one. There were many obstacles to overcome but the final results were ultimately successful. After producing 15 total detectors, only five turned out to be done in the correct fashion. The correct detectors had the Schottky contact on the epitaxial layer while the incorrect detectors had the Schottky contact on the substrate. There are still six unused pieces of silicon carbide, with unknown orientations, remaining. The detectors that were successful were samples numbered 01, 05, 08, 13 and 21. Of these, 01, 08 and 21 have 3mm Schottky contacts while 05 and 13 have 8mm contacts. Detector 13 has a pure nickel Schottky contact while the remaining four have nickel, titanium, and gold Schottky contacts. The remaining untouched pieces of SiC are numbered 02, 03, 10, 11, 12, and 17. All other pieces have had contacts incorrectly deposited on them.
Full packaging of the devices was relatively easy. The design of alumina packaging was chosen due to its ease of centering the detector under the alpha source and its cleaner look. Originally, long, unshielded wires were used to make connection from the packaging to the BNC connector. Upon further thought, it became apparent that long, unshielded wires act as large antennas and thus create large amounts of noise in the system. Thus, the wires were electrically shielded from one another with Kapton tubing and placed inside 3/8” tinned copper braid, which was grounded to the signal ground. This combination provided significant shielding and allowed the alpha particle signal to be processed.

The electrical connection between the Schottky contact and its corresponding small gold pad on the alumina was made very precisely and very delicately. It was imperative that the joint connecting the wire to the Schottky contact, which was made with nickel paste, be made as small as possible. A larger connection point on the Schottky contact meant a decrease in the active area of the Schottky contact. Sometimes these nickel paste connection to the Schottky contact popped off due to their delicate nature and had to be reconnected. The new connections were usually made to the same location as the previous connections so as to keep from further reducing the active area. The connections made to the ohmic contact on the bottom of the SiC detector rarely popped off where the detector was connected to the center gold pad on the alumina substrate. It was later discovered that the physical properties of the contacts might actually change with long-term heat and radiation exposure.
4.2 Current-Voltage (IV) Measurements

Current-Voltage measurements were made both in the cleanroom directly after detector fabrication and in Scott Laboratory after packaging the detectors. IV measurements taken in Nanotech West will be referred to as “diode” tests while tests completed in Scott Laboratory after packaging to the alumina substrates will be referred to as “detector” tests. IV measurements of the detectors were also taken before and after heating. Sometimes measurements could not be taken after heating due to the detector falling apart or somehow becoming disconnected. Ideally, measurements would have been taken at various temperatures during the heating process to observe the detector’s response but it was logistically impossible. There was no easy way to stop the alpha particles from irradiating the detector while it was heated in vacuum without venting the system. All of the data collected will be plotted and analyzed using a script written in MATLAB.

Detectors 01 and 08 were fabricated at the same time in Nanotech West cleanroom as were Detector 05 and Detector 21. Both Detector 01 and 08 had 3 mm Schottky contacts while Detector 05 had an 8 mm contact and Detector 21 had a 3 mm one. The diode IV measurements made in Nanotech West were taken up to a reverse bias of 50 V due to limits on the detection set-up. In Scott Laboratory, the same IV curve measurements were performed but up to a reverse bias of 200 V as that is the necessary operating bias voltage for these detectors.
4.2.1 Detector 01

Figure 21, Figure 22, Figure 23 and Figure 24 show detector IV curves for Detector 01 taken before and after heating to 500 degrees Celsius. Before the heating, the leakage current had a relatively slow increase as the bias voltage increased to -200 \ V; it fluctuated around \(-5\times10^{-9}\) Amps as seen in Figure 21.

![IV Curve (Detector 01 Pre Heating)](image)

Figure 21. Zoomed in detector IV curve for Detector 01, pre detector heating
Unfortunately, post heating was a different story. Figure 22 shows that the leakage current started to dramatically increase (or become “more” negative) around -120 or -140 V and finally ended somewhere around $-1 \times 10^{-6}$ Amps at -200 V. Figure 23 and Figure 24 show a comparison of the detector IV curves before and after heating for Detector 01. Upon first looking at Figure 23, it seems as if the detector’s performance has not degraded. Upon further investigation of the higher bias voltages in Figure 24, it becomes apparent that something has changed in the performance of Detector 01 after heating. At the same operating bias voltage, there is now about a three order of magnitude difference in the leakage current.
Figure 23. Detector IV curve for Detector 01 before and after heating to 500C

Figure 24. Zoomed in detector IV curve for Detector 01 before and after heating to 500C
4.2.2 Detector 08

Detector 08 was made in the same run at Nanotech West as Detector 01. The behavior of Detector 08 is similar to that of Detector 01. Before heating, the leakage current of Detector 08, shown in Figure 25 and Figure 26, is somewhere on the order of $-1 \times 10^{-8}$ or $-5 \times 10^{-9}$ Amps. Both Detector 08 and Detector 01 exhibit good diode behavior, as there is a very quick increase in current when the voltage changes from negative to positive. Unfortunately, the packaging housing Detector 08 fell apart before a post heating measurement could be made.

![Figure 25. IV curve Detector 08 pre heating of detector](image-url)
4.2.3 Comparison of Detector 01 and Detector 08

In Figure 27 and Figure 28, the behavior of Detectors 01 and 08 are compared just after deposition. Again, Detectors 01 and 08 were fabricated at the same time in the Nanotech West cleanroom. Both have 3 mm Schottky contacts. Figure 27, a diode IV curve, shows relatively similar behavior for reverse biases up to -50 V, as the graphs for Detector 01 and Detector 08 seem to overlap. The diode IV curves differ in their rectifying behavior. While both samples perform well, Detector 01 performs better than Detector 08. Figure 28 shows that in the high reverse bias voltage region, Detector 01 continues to perform better. Its leakage current is about twice as small as that of Detector 08 at -50 V. While both detectors have a leakage current on the order of $10^{-10}$ Amps at -
50 V, it is interesting that Detector 01 performs slightly better even though both detectors are the same size and have experienced the same deposition and annealing conditions.

Figure 27. Diode IV curves for Detectors 01 and 08 after deposition in the cleanroom; 01 and 08 were made at the same time
4.2.4 Detector 21

Detector 21 was the third 3 mm detector used in this project. It was not fabricated at the same time as Detectors 01 and 08. Unfortunately, the detector IV curve file for Detector 21 before heating was lost so there is only data for the detector IV curve after heating and after repackaging. Data still exists for the diode IV curve. Once the IV curve for post heating was taken, Figure 29, and the detector was removed from the set-up, the detector fell apart (see Figure 80 in section 5.1 Detector Fabrication Analysis). It had to be reattached to its alumina packaging and have the Schottky and ohmic electrical connections remade. A detector IV curve was taken after repackaging and can be seen in Figure 30 and Figure 31. The detector seems to still exhibit good behavior despite

Figure 28. Zoomed in diode IV curves (from Figure 27) for Detectors 01 and 08 after fabrication in cleanroom; 01 and 08 were made at the same time
previously being heated up to 350 degrees Celsius for testing. The zoomed in IV curve in Figure 31 for Detector 21 looks very similar to that of Detector 08 seen in Figure 26.

Figure 29. Detector IV curve for Detector 21 after heating to 350C for testing
Figure 30. Detector IV curve for Detector 21, which was repackaged after heating to 350°C

Figure 31. Zoomed in detector IV curve (from Figure 30) for Detector 21 after heating and repackaging
4.2.5 Comparison of Detector 01, Detector 08 and Detector 21

A comparison of Detectors 01, 08 and 21 was made after contact deposition, after packaging (or repackaging in the case of Detector 21) on alumina substrates and after heating. Figure 32 and Figure 33 show diode IV curves measured in Nanotech West after deposition; Figure 34 and Figure 35 show detector IV curves measured in Scott Laboratory after packaging; and Figure 36 and Figure 37 show the IV curves measured in Scott Laboratory after heating to temperatures of 350 degrees Celsius or higher.

Figure 32. Diode IV curves for Detectors 01, 08 and 21 after fabrication in the cleanroom; all have 3mm Schottky contacts
From Figure 32 and Figure 33, all three detectors are similar after deposition. Detector 21 seems to more closely align with Detector 01 than Detector 08. Detector 08 continues to have worse rectifying behavior and worse leakage current than Detector 21 and Detector 01. Detector 21 has a slightly smaller leakage current than Detector 01 at -50 V. The IV curve for Detector 21, when zoomed in, has a large dip around -10 V. This could be due to slight movement of the IV measurement setup. Knocking the table, rolling on chairs or walking around the set-up while it is running can produce such blips as shown.
Figure 34. Detector IV curve for Detectors 01, 08 and 21 after packaging; tested in Scott Laboratory

Figure 35. Zoomed in detector IV curve (from Figure 34) for Detectors 01, 08 and 21 after packaging; tested in Scott Laboratory
Figure 34 and Figure 35 depict the relationship between the detectors after they were packaged on alumina substrates in order to be heated. The data shown for Detector 21 was taken after it was repackaged since the original data file cannot be found. Detector 21 had previously been heated up to 350 degrees Celsius and subsequently fell apart. It is comforting to see that despite heating and repackaging, the detector IV curve for Detector 21 appears to behave no worse than Detector 08, which had not been heated at this time. Unfortunately, Detector 21 was no longer better than Detector 01 as it had been when both were just diodes. The packaging does not seem to change the IV characteristics of Detectors 01 and 08, as justified by examining both the pre-packaging (diode) and the post-packaging (detector) results seen in Figure 33 and Figure 35, respectively.

![IV Curve (Detector 01 vs Detector 21 Post Heat)](image)

**Figure 36.** Detector IV curve for Detectors 01 and 21 after heating to 350C or greater
The behavior after heating the detectors was also examined. Detector 08 fell apart in the process of heating and cooling and was therefore unavailable for final measurements of its diode characteristics. Figure 36 and Figure 37 show different views of the data collected. Both Detector 01 and Detector 21 behaved very poorly after heating. For Detector 01, the leakage current after heating was on the order of \(1 \times 10^{-6}\) Amps, whereas before heating it was on the order of \(5 \times 10^{-8}\) Amps at \(-200\) V. It is almost 100 times worse post heating. A similar behavior was seen and was even worse for Detector 21. Detector 21 had a leakage current around \(-1.5 \times 10^{-5}\) Amps after heating whereas, after it’s first round of heating and repackaging, the leakage current was about \(-1.5 \times 10^{-8}\) Amps. Detector 21’s performance was three orders of magnitude worse at \(-200\) Volts.
4.2.6 Detector 05

Though only used for a couple of tests, Detector 05, which had an 8 mm Schottky contact, also had detector IV curves taken before and after heating. Detector 05 could only be heated to 90 degrees Celsius before it became very noisy and was unable to be used for further alpha particle detection. Nonetheless, detector IV curves before and after heating were taken and can be seen in Figure 38 and Figure 39. Heating did not seem to create the same problem in Detector 05 as it did in Detectors 01 and 21. While remaining on the same order of magnitude, the leakage current for Detector 05 is about two and a half times better than before it was heated. The improvement in the leakage current and slight shift to the left of the IV curve seems to be at odds with the increased noise observed on the oscilloscope from Detector 05 during alpha particle collection. The increased noise made it impossible to collect an alpha spectrum at temperatures above 90 degrees Celsius.
Figure 38. Detector IV curve for Detector 05 before and after heating to 90C

Figure 39. Zoomed in detector IV curve for Detector 05 before and after heating to 90C
4.3 Theoretical Alpha Energy Spectra

*SRIM 2008* was used as a theoretical tool for calculating the distribution of alpha particle energies emerging from the other side of a pre-defined metal contact as well as the depth to which the alpha particles penetrated into the detector. It is important to understand the spread in alpha particle energies before they enter the active volume of the detector to be collected. It is also important to be aware of the distance an americium-241 alpha particle will travel in silicon carbide because a particle must fully stop in the depletion region in order to have its full energy collected.

To examine the distribution of energies through the Schottky contact, three metal layers composed of 10 nm of gold, 10 nm of titanium and 100 nm of nickel were studied. Alpha energies were mono-energetic and corresponded to the primary alpha emitted from Am-241 (5.485 MeV). All alphas were perpendicularly or normally incident to the contact surface. Their path through the contact material can be viewed in Figure 40. This figure shows the beginning of the spreading of energies of these alpha particles as they traverse through the material. The energy distribution recorded and fit in Figure 41 depicts the spreading of the alpha spectrum due to alpha energy loss within the 4H-SiC alpha particle detectors. The metal, as well as many others factors, contributes to an increase in the FWHM and resulting energy resolution calculation.
Figure 40. SRIM output showing trajectories of primary Am-241 alphas scattering through 10 nm of gold, 10 nm of titanium and 100 nm of nickel.

Figure 41. SRIM calculation of primary alpha from Am-241 scattering through 10 nm of gold, 10 nm of titanium and 100 nm of nickel.
SRIM 2008 was also used to analyze the behavior of the alpha particles in the detector. Again, the primary alpha particle from Am-241 (5.485 MeV) was used and was set to perpendicularly impact the Schottky contact. It was confirmed that americium-241 particles would indeed stop in the active layer of the detector as seen in Figure 42. The depletion region is normally about 20 microns thick at full bias because the low-doped epitaxial layer has a thickness of 20 microns. Figure 43 shows the distributions of distances traveled by the alpha particles. Most particles only traveled about 18.5 microns into the detector. Furthermore, Figure 44 shows the relative number of collisions that occur per angstrom into the detector that an alpha particle travels. The number of collisions significantly increases, as the alpha particle gets deeper into the material.

Figure 42. Am-241 alphas scattering through active volume of SiC
Figure 43. Fitted distribution showing the distances alpha particles travel in SiC after passing through the Schottky contact.

Figure 44. Collision events that have occurred as alpha particle travels through Schottky contact and active SiC region.
4.4 Experimental Alpha Particle Detection

The primary goal of this experiment was to build an alpha particle detector that could operate at room and elevated temperatures. Ultimately, the detector will need to be able to operate in a harsh chemical and high radiation environment. The data that follow shows the results from room and elevated temperature operation with Detectors 01, 05, 08 and 21.

The data will be presented as views of the true, unedited spectra; zoomed in views of sets of spectra at various temperatures; and as single and or double Gaussian, fitted alpha peaks. The alpha peaks were identified and fit to a Gaussian function as identified in Equation (1) and some were fit to a double Gaussian as identified in Equation (3). The corresponding FWHM was calculated based on the formula given in Equation (2) when the peaks were fit with a single Gaussian.

All of the spectra were taken with the following parameters. The bias voltage was set to -200.2 Volts. It was set as close to -200 V as possible based on the constraints given by the DSA. The rise/fall time was set to 1.0 microsecond and the flat top time was set to 0.5 microseconds. The lower level discriminator (LLD) was set to 0.1%. No pole zero correction was made. Some measurements had to be taken with the heater in the off position due to extremely high levels of noise. This occurred any time a temperature is given as a range, i.e. 195-205°C, instead of as a single temperature. An example of the magnitude of the noise can be seen in Figure 89 of Appendix D or Figure 45 of the following section.
4.4.1 Detector 01

Detector 01 had only one series of spectra taken in one long continuous run. Data were taken every 50 degrees Celsius starting from 23 degrees Celsius, 50 degrees Celsius, etc. up to 500 degrees Celsius. After taking data at 500 degrees Celsius, the detector was allowed to cool down over night in vacuum. A room temperature spectrum was taken again the following morning.

![Detector 01 Data, T = 23 – 500C](image)

Figure 45. Combined spectrum for Detector 01 of alpha spectra collected in range of 24-500C in 100C intervals

Figure 45 shows multiple spectra for temperature intervals of 100 degree Celsius. The largest dark blue and green peaks represent room temperature prior to heating and 100 degrees Celsius, respectively. The spectrum taken after the detector was heated and
cooled down is not shown. It was too noisy to be able to discern any features from an alpha peak. When the set-up was brought back up to atmosphere, it was discovered that the detector had split into many piece: the diode, the alumina substrate, the Nickel-220 wire leads connected to the BNC.

Figure 46. Zoomed in spectrum for Detector 01 (from Figure 89) for temperatures between 24-200C

Figure 46 and Figure 47 show subsections of the total number of alpha spectra collected. Figure 46 shows spectra collected between room and 200 degrees Celsius while Figure 47 shows spectra collected between 200 and 500 degrees Celsius. Figure 47 also includes the preheating room temperature spectra as a reference point. It is interesting to note that both sets of peaks in the graphs change with increasing temperature. In Figure 46, the peak shifts slightly to the right with increasing
temperature. There is also a slight change in the magnitude of the peak despite all measurements being taken for 3000 seconds. The total number of counts remained nearly the same at 14913 counts +/- 1.58%, as given in Table 2, for temperatures up to 150 degrees Celsius. The 200 degree Celsius measurement is also strangely low and flat. In Figure 47, the peaks begin to move to the left, or to lower channels, as temperature approaches 500 degrees Celsius. These peaks were expected to be smaller as they were only counted for 1500 seconds unlike the room temperature spectrum, which was counted for 3000 sec. The total number of counts for temperatures between 200 and 450 degrees Celsius averaged 8331 counts +/- 5.42%.

Figure 47. Zoomed in spectrum for Detector 01 for temperatures between 200-500C
Figure 48. Fitted peak for Detector 01 at room temperature (24°C), pre heating

Figure 49. Fitted peak for Detector 01 at T=52°C
Figure 50. Fitted peak with 2 Gaussians for Detector 01 at room temperature (24C), pre heating

Detector 01, pre heating, T=24 C, P=94 mTorr

$\mu_1 = 2589.1 \text{ channel}$
$\sigma_1 = 13.4 \text{ channels}$
FWHM$_1 = 31.6 \text{ channels}$

$\mu_2 = 2566.6 \text{ channel}$
$\sigma_2 = 21.7 \text{ channels}$
FWHM$_2 = 51.1 \text{ channels}$

Figure 51. Fitted peak with 2 Gaussians for Detector 01 at T=52C

Detector 01, T=52 C, P=92 mTorr

$\mu_1 = 2594.0 \text{ channel}$
$\sigma_1 = 14.1 \text{ channels}$
FWHM$_1 = 33.2 \text{ channels}$

$\mu_2 = 2572.0 \text{ channel}$
$\sigma_2 = 23.0 \text{ channels}$
FWHM$_2 = 54.2 \text{ channels}$
All of the alpha peaks were fit to a single or double Gaussian using the built in Gaussian fitting function in MATLAB. A custom script was written to execute the plots and fits but the script was based on MATLAB operations. In Figure 48 and Figure 49, the single Gaussian fitted data from room temperature and 50 degrees Celsius is shown. These spectra show a significant tailing on the low channel or low energy side of the spectra. The significant tailing leads to using a double Gaussian as described in Equation (3) and seen in Figure 50 and Figure 51. In the spectra for 195-205 degrees Celsius (Figure 52) and 395-405 degrees Celsius (Figure 53), this tailing is not as easily observed. Thus, fitting with a single Gaussian seems to be a better choice. Also, as stated previously, for these two high temperature spectra there is a drop in the total counts in the peak channel as well as a drop in the total number of counts in the full peak. The spectra in Figure 52 and Figure 53 were only counted for 1500 seconds.

Figure 52. Fitted peak for Detector 01 at T=195-205C
Figure 53. Fitted peak for Detector 01 at T=395-405°C

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<tr>
<th>Temp (°C)</th>
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<th>Normalization (chan)</th>
<th>µ (chan)</th>
<th>σ (chan)</th>
<th>FWHM (keV)</th>
<th>Energy Res (%)</th>
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Table 2. Single Gaussian fitting parameters and associated data for Detector 01

Table 2 lists all of the fitting parameters for all of the alpha peaks collected during this run with Detector 01 and fit with a single Gaussian. Some of the fitted peaks can be found in this section while the rest can be found in Appendix D.
4.4.2 Detector 08

Detector 08 was used in three different runs, two of which are reported on in this section. The first run, Run 1, was completed on August 20, 2011 and the second run, Run 2, was completed on August 23, 2011. After the second run, the detector fell apart so the electrical contacts to the detector were reformed. Unfortunately, even though electrical connections were reformed in a new location on the Schottky contact, the detector was still too noisy at room temperature so data from a third run could not be taken. This result from an attempted third run may point to something changing within the silicon carbide diode rather than just within the electrical connections.

Run 1 was taken at room (23C), 50 and 100 degrees Celsius. Run 2 was taken at the same temperatures but the data for 100 degrees Celsius was too noisy to be interpreted. A table outlining the fitting parameters for peaks fit with single Gaussians can be see in Table 3 for Run 1 and Table 4 for Run 2.
Table 3. Single Gaussian fitting parameters and associated data for Detector 08, Run 1 (Run 082011)

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<thead>
<tr>
<th>Temp (°C)</th>
<th>Tot Counts in Peak</th>
<th>Count Time (s)</th>
<th>Normalization (chan)</th>
<th>μ (chan)</th>
<th>σ (chan)</th>
<th>FWHM (keV)</th>
<th>Energy Res. (%)</th>
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<td>15.85</td>
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<td>2738.87</td>
<td>16.01</td>
<td>75.86</td>
<td>1.38</td>
</tr>
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</table>

Figure 54 shows the combined low and higher temperature spectra for Detector 08’s first run. The FWHM and resulting energy resolution of the peaks were very good. As temperature increased from room temperature, the peak channel moved slightly to a higher channel. The energy resolution still remained relatively stable, slightly under 1.4%. The total number of counts also remained pretty constant at 15553 counts +/- 1.31%.
The second run for Detector 08 was not as successful. The electrical contacts made to the detector were not changed in between runs. The detector could be operated at 23 and 50 degrees Celsius before becoming too noisy. A change in the alpha peak can easily be observed at 50 degrees Celsius and the peak is gone by 100 degrees Celsius in Figure 55. The total number of counts in the peak decreased by 24.49%. The same window scale was used for Figure 54 (Run 1) as Figure 55 (Run 2) so a broadening of the room temperature measurement can also be observed. This is confirmed by the normal distribution fitting parameters given for Run 2 in Table 4. Furthermore, one can see that the noise is orders of magnitude greater for the 50 and 100 degree Celsius runs than for the room temperature run, given in Figure 56. It is also of a different form than the noise in the spectra associated with Detector 01 (see Figure 89 of Appendix D).

![Figure 55. Zoomed in spectrum for Detector 08, Run 2, heating between 23 and 100C.](image-url)
Figure 56. Full alpha spectrum between 23 and 100°C from Detector 08, Run 2

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<tr>
<th>Temp (°C)</th>
<th>Tot Counts in Peak</th>
<th>Count Time (s)</th>
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Table 4. Single Gaussian fitting parameters and associated data for Detector 08, Run 2 (Run 082311)

Figure 57, Figure 59 and Figure 60 show the single Gaussian fitted distributions to the alpha peaks collected from Detector 08. Figure 57 is from Run 1 while Figure 59 and Figure 60 are from Run 2. The room temperature spectrum from the first run (Figure 57) shows significantly more tailing on the low channel side than the room temperature spectrum from the second run (Figure 59) though the tailing is still visible. Figure 58 and Figure 61 show the room temperature spectra from Run 1 and Run 2, respectively, fit with double Gaussians. Similar behavior to the room temperature spectrum from Run 1
with Detector 08 can be seen in the 51 and 100 degree Celsius measurements in Appendix D. Figure 60, on the other hand, from Run 2 at 51 degrees Celsius no longer has the same low energy tailing. The peak has broadened enough that the tail now appears as part of the peak.

Figure 57. Fitted peak for Detector 08, Run 1 at room temperature (23C), pre heating
Figure 58. Fitted peak with 2 Gaussians for Detector 08, Run 1 at room temperature (23C), pre heating

Figure 59. Fitted peak for Detector 08, Run 2 at room temperature (23C)
Figure 60. Fitted peak for Detector 08, Run 2 at 51°C

Figure 61. Fitted peak with 2 Gaussians for Detector 08, Run 2 at room temperature (23°C)
Detector 21 was used in two different runs for this experiment. The first run, Run 1, was conducted on August 21, 2011. Data was taken in one continuous run up to 300 degrees Celsius in 50 degree Celsius intervals. The spectrum taken at 350 degrees Celsius was too noisy to interpret. The second run, Run 2, was taken on August 24, 2011 and was done a bit differently. The detector fell apart after Run 1 and had to be reconnected. The second electrical connection to the Schottky contact was made in a different location from the first electrical connection. Then, a series of different temperature measurements were taken to try to determine if it was the nickel cement connections that were failing or if it was the actual silicon carbide detector.

Run 2 had a measurement taken at room temperature and then the heater was slowly heated to 300 degrees Celsius. The detector was heated at no more than 10 degrees Celsius per minute. After the 300 degrees Celsius count, the detector was cooled down at 10 degrees Celsius per minute or slower to room temperature again where another room temperature spectrum was taken. Finally, the detector was heated to 300 degrees Celsius again and a shorter count was taken to ensure the repeatability of the previous 300 degrees Celsius measurement. Then, the detector was heated to 400 degrees Celsius for its final measurement. After the 400 degree Celsius measurement, a room temperature spectrum was attempted but the detector had become too noisy.
Figure 62. Detector 21, Run 1 spectra between room (24C) and 200C

Figure 63. Detector 21, Run 1 spectra between 200C and 335C including room temperature
The fitting parameters and other associated information for Run 1 can be seen in Table 5 and two spectra showing the temperature results can be seen in Figure 62 and Figure 63. Figure 62 shows the lower temperature runs from room temperature to 200 degrees Celsius. Figure 63 shows the spectra from 200 degrees Celsius to 335-341 degrees Celsius. The figure also includes the spectrum from room temperature as a reference. The last run at 335-341 degrees Celsius was only counted for 250 seconds because it was becoming too noisy to interpret. The total number of counts for the lower temperature data taken between room and 150 degrees Celsius averaged 17473 counts +/- 2.26%. As for the higher temperature runs, the total counts averaged 8918 counts +/- 1.14%.

In Figure 62, the previously seen trend in the other detectors is again apparent. As temperature increases from 24 to 150 degrees Celsius, the peak channel moves to a higher channel. At 200 degrees Celsius, the peak channel begins to decrease and the FWHM increases as evidenced by Figure 63. Table 5 also shows that the energy resolution is less than two percent for temperatures between 24 and 100 degrees Celsius. After that point, the energy resolution continues to increase with increasing temperature. Furthermore, after the detector was heated to temperatures above 300 degrees Celsius, its final room temperature measurement taken after cooling did not recover to its previous peak position and resolution.
Table 5. Single Gaussian fitting parameters and associated data for Detector 21, Run 1 (Run 082111)

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<th>Temp (°C)</th>
<th>Tot Counts in Peak</th>
<th>Count Time (s)</th>
<th>Normalization (chan)</th>
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<th>σ (chan)</th>
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<td>8860</td>
<td>1500</td>
<td>52.52</td>
<td>1773.75</td>
<td>65.73</td>
<td>314.00</td>
<td>5.72</td>
</tr>
<tr>
<td>295-305</td>
<td>9036</td>
<td>1500</td>
<td>45.36</td>
<td>1674.44</td>
<td>78.68</td>
<td>375.86</td>
<td>6.85</td>
</tr>
</tbody>
</table>

Figure 64 and Figure 65 show the second round of spectra taken with Detector 21 after it was given new electrical connections. Table 6 outlines the fitting parameters and other associated information for Run 2. The total number of counts in the peak was similar to the other data runs. For all of 3000 second counting time, the total number of counts averaged 17473 counts +/- 2.26%. For the shorter counts (1500s), the total counts in the peak averaged 8918 counts +/- 1.14%. Figure 64 shows the repeatability of the measurements taken by Detector 21 for at least 2 heating and cooling cycles. The 400 degrees Celsius measurement, highlighted by Figure 65, is the outlier in the group. Something must have happened between the 300 and 400 degrees Celsius measurements. Figure 65 also shows a strange behavior after 1500 seconds in the 400 degrees Celsius count. After 1500 seconds, a second, large noise peak grows with a peak about 400 channels lower than the desired alpha peak.
Figure 64. Detector 21, Run 2 spectra measurements taken at room (24C), 300C and 400C

Figure 65. Detector 21, Run 2 spectra taken at 400C showing change in noise after 1500s
In Table 6 it interesting to note that despite the change in the location of the peak of the 400 degrees Celsius measurement, the energy resolution remains relatively constant when compared to that of the 300 degrees Celsius measurement. The energy resolution is still much smaller for the room temperature measurements but for elevated temperatures, the resolution is somewhere around eight or nine percent.

Figure 66, Figure 67 and Figure 68 show three spectra fitted with a single Gaussian from Detector 21, Run 1. Figure 66 is the first room temperature measurement taken before heating while Figure 68 is the room temperature measurement taken after the detector was heated to high temperature. Figure 67 shows the fitted data for the high temperature 295-305 degree Celsius measurement. Figure 69 shows the room temperature spectrum, pre heating, fitted with two Gaussians instead of one which takes the low energy tailing into account better than the single Gaussian.
Figure 66. Fitted peak for Detector 21, Run 1 at room temperature (24C)

Figure 67. Fitted peak for Detector 21, Run 1 at 295-305C
Figure 68. Fitted peak for Detector 21, Run 1 at room temperature (23°C), post-heating

![Graph showing data and Gaussian fit for Detector 21, post-heating, T=23 C, P=91 mTorr]

- $\mu = 1615.7$ channel
- $\sigma = 93.2$ channels
- FWHM = 219.5 channels

Figure 69. Fitted peak with 2 Gaussians for Detector 21, Run 1 at room temperature (24°C), pre-heating

![Graph showing data and two Gaussian fits for Detector 21, pre-heating, T=24 C, P=91 mTorr]

- $\mu_1 = 2705.7$ channel
- $\sigma_1 = 13.8$ channels
- FWHM$_1 = 32.5$ channels
- $\mu_2 = 2686.2$ channel
- $\sigma_2 = 22.6$ channels
- FWHM$_2 = 53.2$ channels
The pre (Figure 66 and Figure 69) and post heating (Figure 68) room temperature measurements show a markedly different response in spectral form and fitting parameters as given in Table 5. The post heating measurement is visibly skewed to higher energies and is not very Gaussian in shape. The pre heating measurement on the other hand fits a normal distribution very well and its low energy tailing is taken care of by the use of two Gaussians. Furthermore, the post-heating spectrum has a much larger standard deviation and its mean is about 1100 channels lower than that of the pre heating measurement. It is interesting to observe the high energy tailing in Figure 68 because such behavior is not visible in the high temperature spectra.

Figure 70. Fitted peak for Detector 21, Run 2 at room temperature (24C), Measurement 1
Figure 71. Fitted peak for Detector 21, Run 2 at 300C, Measurement 2

Figure 72. Fitted peak for Detector 21, Run 2 at room temperature (24C) after heating to 300C, Measurement 4
Figure 70, Figure 71 and Figure 72 depict fitted spectra from Run 2 of Detector 21. Figure 70 and Figure 71 are room temperature spectra taken before and after heating to 300 degrees Celsius, respectively. Figure 72 shows the fitted spectrum from the high temperature data taken at 300 degrees Celsius. All three spectra look relatively similar aside from slight changes in the viewing window but the 300 degrees Celsius spectrum is noticeably broader than the room temperature ones. Table 6, which contains the fitting parameters and other associated data, confirms these observations. The room temperature measurements both have an energy resolution around two or three percent while the high temperature data has an energy resolution of almost nine percent. Furthermore, at room temperature, the low energy tailing is still visible in Figure 70 and Figure 71. Unfortunately, the data could not be fit with double Gaussians, which may have provided further insight into the actual shape of the peaks. The MATLAB code used to fit the data broke down on these spectra despite repeated attempts and modifications.

4.4.4 Detector 05

As previously discussed, Detector 05 has an 8 mm Schottky contact and was only subjected to two runs at relatively low temperature before becoming too noisy for more data collection. The spectra taken at 75 and 90 degrees Celsius are visible in Figure 73. While both peaks have approximately the same mean, the 90 degrees Celsius measurement is slightly broader and shorter than the spectrum taken at 75 degrees
Celsius. Based on Table 7, the total number of counts in the peak averaged 198425 +/- 1.67%.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>Tot Counts in Peak</th>
<th>Count Time (s)</th>
<th>Normalization (chan)</th>
<th>μ (chan)</th>
<th>σ (chan)</th>
<th>FWHM (chan)</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>200770</td>
<td>3000</td>
<td>2556.52</td>
<td>2710.41</td>
<td>30.71</td>
<td>72.32</td>
</tr>
<tr>
<td>90</td>
<td>196079</td>
<td>3000</td>
<td>1884.02</td>
<td>2710.52</td>
<td>41.25</td>
<td>97.13</td>
</tr>
</tbody>
</table>

Table 7. Single Gaussian fitting parameters and associated data for Detector 05

Figure 73. Detector 05 runs at 75C and 90C
Figure 74. Fitted peak for Detector 05 at 75°C.

Figure 75. Fitted peak for Detector 05 at 90°C.
Figure 74 and Figure 75 show the fitted data from the 75 and 90 degrees Celsius runs, respectively. A variation in viewing window appears to be the only easily visible difference between the peaks. Though low energy tailing is still slightly visible, there is much less in these distributions than in the detectors with smaller contacts. The spectrum can still be fit with two Gaussians though it appears to make less of a difference in the goodness of the fit. The double Gaussian fit for 90 degrees Celsius can be seen in Figure 76.
Chapter 5 Analysis

This section outlines the analysis of the data collected and reported on in Chapter 4. Just like the previous chapter, the results will be organized into four sections: detector fabrication, current-voltage measurements, theoretical alpha energy spectra and experimental alpha particle detection.

5.1 Detector Fabrication Analysis

The detector fabrication process was successful but could be greatly improved in the future in order to make detectors that can withstand high temperatures for extended periods of time. The cleaning and deposition of contacts in the cleanroom appear to be effective processes for building diodes. The gold and titanium layers which were deposited on top of the nickel layer appear to eliminate the oxidation of the nickel Schottky contact at elevated temperatures. In order for this to happen, the titanium must act as a diffusion barrier between the gold and nickel. Furthermore, the sample holder used to deposit the contacts was an effective solution. It was very easy to use and its results were repeatable.

The alumina substrate was also an effective solution for the first iteration of packaging the detectors. Depositing a metal pad in the center and smaller surrounding
pads was a good idea because it allowed for easy electrical connections, Figure 77, and for the detector to easily be centered under the alpha source and on the top of the heater. The only downfall of the packaging was that it needed to be clamped down to the heater as seen in Figure 78 to ensure good thermal conductivity in vacuum.

Figure 77. Diode 01 packaged into Detector 01 and ready for testing
Unfortunately, the braid used to shield the connections to the diode was not enough to eliminate the noise produced by the heater. The grounded copper sheet used to shield the detector was relatively effective in reducing the noise to a couple of hundred channels in the lower energy region of the spectra. Unfortunately, this did not work for all of the high temperature spectra collected, which resulted in noise interference with the data. Some spectra could not be collected simply due to high levels of noise which washed out the alpha spectrum or alpha peaks being located too close to the noise. Furthermore, the grounded copper shield used to isolate the detector from the heater had to be changed after each run as the alumina paint flaked off, see Figure 79.
The biggest issue in the detector fabrication was the electrical contacts made to the Schottky and ohmic contacts. As previously stated, nickel paste from Ted Pella, Inc. was used. After long-term exposure to heat (greater than four hours), the detector connections became brittle. Oftentimes, the detectors fell apart into their constituent pieces as seen in Figure 80. The nickel paste seems to have stuck to the pure nickel ohmic contact and left very little residue on the gold pad. The nickel paste was also very successful at bonding the Nickel 220 wire together as seen in the left hand side of the figure. As for the Schottky contact, there seems to be nickel cement left on both the wire and on the Schottky contact in the 11 o’clock position. It could be that long term exposure to high temperature causes the nickel cement to become brittle due to oxidation and or radiation damage from alpha particles though the former seems more likely. It
could also be that there is not good adhesion between nickel and gold or that heating a sample too quickly causes the connection to weaken due to differences in thermal expansion.

Figure 80. Detector 21, which has fallen apart into its constituent pieces after heating

5.2 Current-Voltage (IV) Measurements Analysis

The current voltage measurements taken for all of the 3 mm detectors (Detector 01, 08 and 21) seem to show some similar trends. As diodes, all three detectors have leakage currents on the order of \(10^{-10}\) Amps at -50 Volts. As packaged detectors, the three have leakage currents in the range of \(-5\times10^{-9}\) and \(-2\times10^{-8}\) Amps at -200 Volts. Furthermore, the packaging does not seem to severely change the leakage current as most of the detectors also have a leakage current in the range of \(-1\times10^{-10}\) to \(-5\times10^{-9}\) Amps at -50 Volts.
Assuming the detectors do not fall apart, heating does seem to change the IV behavior of these 3 mm detectors. After heating, the leakage current at -200 Volts increases about three to four orders of magnitude. However, after repackaging the detector after one heating cycle, the IV curve behavior returns to its pre-heated value as shown by Detector 21 in Figure 35. These observations along with the fact that the detectors sometimes fall apart (Figure 80) seem to lead to the belief that something must be happening in the electrical connections to the Schottky and ohmic contacts that were made with nickel cement rather than a change in the behavior of the silicon carbide, Schottky contact and/or ohmic contact.

The similarity of the diode IV and detector IV curves is important because it points to the repeatability of the experiment. Multiple diodes can be fabricated at one time or in multiple runs and achieve nearly identical results. Furthermore, the similarity between pre and post packaged results indicates that the packaging does not greatly interfere with the electrical properties of the detector and can be repeated across multiple detectors.

5.3 Theoretical Alpha Energy Spectra Analysis

SRIM2008 is an important tool in simulating actual experimental results. One of the most important confirmations given by these SRIM2008 analyses is that Am-241 particles will fully be stopped within the active volume of silicon carbide. Plus or minus three standard deviations incorporates 99.7% of all results for a normal distribution.
Three standard deviations above the mean has a length of 19.24 microns which means the depletion layer, if fully depleted, is just long enough to allow all americium-241 particles to fully deposit their energy.

The transmission of ions through the metal Schottky contact provides data that show a very small change in alpha particle energy. The average energy is 5.44 MeV, which is about 0.73% lower than their incident energy of 5.48 MeV. A 99.7% confidence interval gives the bounds of (5.423 MeV, 5.456 MeV), which are 1.04% below and 0.44% above, respectively, the mean energy of the alpha particle. While this spread in energy can be calculated, it is only a small contribution to the broadening of the alpha peak. The FWHM of this calculation is only 13 keV or 0.013 MeV.

5.4 Experimental Alpha Particle Detection Analysis

The experimental alpha particle detection set-up did not yield as many results as originally thought but there are some conclusive patterns. One of the major discoveries was that the designed and built detectors had problems operating at elevated temperatures for prolonged periods of time. This claim is supported by the significant changes in alpha spectra produced for elevated temperature operation. It is further supported by the inability of most of the detectors to reproduce a good, room temperature spectrum after heating to high temperature. The anomaly in high temperature operation is Run 2 of Detector 21. This run at least shows that the detector can be operated at high temperature for a short period of time before breaking down.
The phenomena associated with the detector breakdown are still unknown. The two main possible breakdown points are related to the actual silicon carbide detector and deposited contacts or the electrical connections to the detector. The more likely of the two options is the electrical contacts formed by nickel cement. If the silicon carbide were deteriorating, it would most likely be due to radiation damage from the alpha particles and/or the high temperature operation. But, silicon carbide is known to be radiation hard and able to operate at high temperatures. Thus, the electrical connections are left in question.

After operation at high temperatures, many of the detectors fell apart into their constituent pieces as shown in Figure 80. This points to the deterioration of the nickel cement. While the nickel cement is able to be heated to 538 degrees Celsius and operate in vacuum, it could possibly still become brittle over time with long-term exposure to both conditions due to oxidation or other associated mechanisms. While much less likely, alpha particle radiation could also cause the cement to become brittle.

A positive turn for the testing was that the energy resolution always remained below 1.57% for temperatures 100 degrees Celsius and lower. These results were repeatable across all runs of all three detectors except for the second run of Detector 08 where the detector became too noisy for operation and fell apart shortly thereafter. Above 100 degrees Celsius, the energy resolution varied between just over 1.5% and as high as 14.83% but usually remained around 6 or 8%. For temperatures below 150 degrees Celsius, the energy resolution was always below 3.46%. The energy resolution also tended to increase with increasing temperature.
One of the goals of the original project proposal was to build a detector that could operate at high temperature and maintain the ability to differentiate between alpha particles with energies as close as 100 – 300 keV. In order to do this, the FWHM must remain small despite increasing temperature. The FWHM is given in channels in all of the corresponding tables for each detector run, but can easily be converted to energy units by converting the peak channel to energy and then using the same calibration factor for the FWHM. The FWHM generally remained below 100 keV for temperatures below 150 degrees Celsius. For some counts, such as Run 2 with Detector 08, the low temperature runs have a FWHM that is 103 keV and greater. As for the high temperature runs, the FWHMs are greater than the desired range of 100 – 300 keV. The values start around 190 keV and go up to values such as 655 keV. Even Detector 21, Run 2 which showed measurement repeatability had a FWHM in the range of 300 – 350 keV at temperatures of 300 degrees Celsius and above. Such large FWHMs are detrimental when the difference in alpha particle energy between americium-241 and plutonium-239 is less than 40 keV.

In order to achieve the above results, the alpha particle spectra had to be analyzed and fit. Choosing the appropriate fit can be a tricky business if one is unaware of what the data should look like. As discussed earlier, americium-241 emits two alpha particles with energies less than 100 keV apart. Because these detectors did not have good enough resolution to naturally show two different peaks and distinguish between the two particles, the lower, less frequent alpha particle contributes to a broadening and low energy tail of the primary alpha peak. A double Gaussian was fit to some of the distributions and provided a better fit than the single Gaussian function. All of the alpha
peaks could not be fit with two Gaussians due to some unphysical results, which were output from the MATLAB function. Furthermore, the MATLAB code often found fits with means about 15 to 30 channels apart for each Gaussian. This is good as the second alpha peak should fall somewhere in this range depending on the location of the primary alpha peak. Fitting the broad spectra with two Gaussians or one did not seem to make much of a difference in the goodness of fit. Around 200 degrees Celsius, the spectra became very broad such that they took on almost a pure Gaussian shape because the low energy tailing was no longer visible.
6.1 Conclusions

This project was successfully able to meet its primary goal of developing a silicon carbide Schottky diode detector and operating it at elevated temperatures with a FWHM between 100-300keV. The fabrication process was straightforward and able to be completed in a one day run. The designed, fabricated and tested detectors were able to operate at room temperature and up to 100 degrees Celsius with few changes in their operation for these temperatures. The energy resolution remained below 1.57% and the FWHM was below 100 keV. The goal was to keep the FWHM below 300 keV for low and high temperature operation based on the original research proposal, which funded this research. At temperatures below 150 degrees Celsius, the energy resolution remained below 3.46% and the FWHM was below 190keV. Unfortunately one problem encountered is related to the peak shift as temperature increases. An ideal detector would show no shift in peak centroid as temperature increases or decreases. While unable to operate up to 500 degrees Celsius as hoped, the detectors in this project still showed potential and laid the foundation for future modifications and high temperature studies.
6.2 Future Work

One of the largest challenges facing this project is to develop a method for making electrical connections to the Schottky contact. Another method beyond using nickel paste should be investigated. This could be using silver cement, which is also available from Ted Pella, Inc. Silver cement may not be the solution though, as silver is also known to readily oxidize at high temperatures. It may be that wire bonding is the solution. Wire bonding was previously attempted in this project with a wedge wire bonder but was unsuccessful. A new formulation of Schottky contact materials and detector packaging may be the solution to allow wire bonding to work. Or, wire-bonding methods such as ball bonding may prove to be successful when wedge wire bonding was not. EWI is experienced in this type of joining procedure and may be able to provide guidance.

In addition to a new method for joining, a new style of packaging should be developed. Many detectors are packaged in TO (Transistor outline) Packages. Companies such as Schott sell these products, which are available in many different forms. The only complication may arise is devising a way to heat the detector in such a package. A benefit to using a TO package is that they are a standard piece of packaging design and would make mass production of the developed Schottky diode detector much easier.

As for the actual diodes being used in this experiment, it would be helpful to experiment with different dopings and epitaxial layer thicknesses of the silicon carbide, the Schottky contact sizes and alpha source set-up. Modifying one or a few of these
parameters may improve the signal collection capabilities and resulting spectra of the
detector. For example, multiple small diameter contacts could be used instead of one
large one. This may help to increase the resolution while still collecting as many alpha
particles as possible. Another improvement could be made to the detector resolution by
collimating the alpha source. Perfectly normally incident alpha particles should show a
smaller FWHM and energy resolution than an uncollimated source.

Once many of these joining and fabrication issues have been solved, the detector
will need to be tested in a high radiation, high temperature and chemically aggressive
environment. The pyroprocessing environment can be very corrosive and detrimental to
many materials and joining technologies that would otherwise be suitable under normal
operating conditions. In order to irradiate the detector with alpha particles, a thin LiF foil
could be place over top of the detector and then the whole setup be placed in a neutron
rich environment. Another option is to use a LiF gel or liquid and submerse the detector
in it. Using a gel or liquid could be complicated since the whole setup will need to be
heated and irradiated with neutrons at the same time.

There is a lot of development that can still be done on this detector. The
foundation has been built and the future opportunities are endless. The biggest struggle
will be finding materials that can withstand high temperature, high radiation and
chemically aggressive environments while still performing their necessary duties.


A. Ivanov, E. Kalinina, G. Kholuyanov, N. Strokan, G. Onushkin, A. Konstantinov, A. Hallen, and A. Kuznetsov. High energy resolution detectors based on 4H-SiC.


F. Ruddy, J. Seidel, and P. Sellin. High-resolution alpha spectrometry with a thin-window

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W. Windl and T. E. Blue. SiC Schottky diode detectors for measurement of actinide
concentrations from alpha activities in molten salt electrolyte. 2009.


Appendix A Wafer Washing Procedure
This appendix contains a detailed description of the wafer washing procedure used to clean the SiC dies before the evaporation and annealing process was carried out. The RCA clean is very similar to the SC-1 and SC-2 wafer washing procedures. Numerous facilities and universities have slight variations on the RCA clean, but all of the cleaning procedures contain at least:

1. Removal of organic compounds and contamination (Organic clean)
2. Removal of oxide layers (Oxide strip)
3. Removal of ionic compounds and contamination (Ionic clean)

The process used in this research contains all of these steps as well as an extra oxide strip. Stephen Stone also utilized this cleaning procedure in his Master’s research conducted at The Ohio State University (Stone 2008). It is important to follow all safety procedures associated with these chemicals as well as those taught by the Nanotech West staff. Furthermore, it is important to make sure to use HDPE tweezers as metal ones can cause contamination of the samples. A quick outline of the wafer washing procedure can be seen in Table 8.

The first step in the cleaning process was to conduct an organic clean of the samples in a hydrogen peroxide (H$_2$O$_2$), ammonium hydroxide (NH$_4$OH) and distilled water (DI) solution with a composition of 1:1:5. This solution was heated to 80 degrees Celcius using a lab hot plate and checked with a thermometer. The samples were placed in the bottom of the 150 mL beaker and allowed to soak for 15 minutes. After the soak, they were briefly rinsed for approximately 2 minutes in a beaker of DI water, which was changed in between each rinsing. The samples were then washed in an oxide solution of hydrofluoric acid (HF) and DI water with a composition of 1:50 for 15-20 seconds. In
order to accomplish this, the samples were placed in a 50 mL HDPE beaker, which had holes drilled in the bottom to allow for drainage. This allowed for quick and easy removal of the samples from the dangerous solution. After the oxide strip, the samples were quickly rinsed for about 10-15 seconds in DI water, to prevent reformation of oxides, and individually placed in the ionic compound removal solution. This solution was made up of hydrochloric acid (HCl), hydrogen peroxide (H₂O₂) and DI water with a ratio of 1:1:5. The samples were soaked at 80 degrees Celsius for 15 minutes (as with the organic clean) and afterwards once again rinsed for 2 minutes. Finally, the samples were again placed in the HDPE beaker and dipped in an HF and DI water solution (1:50) for 15-20 seconds and rinsed in DI water for another 10 seconds. The samples were then dried with a nitrogen (N₂) gun as needed.

<table>
<thead>
<tr>
<th>Clean</th>
<th>Solution</th>
<th>Temp</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Clean</td>
<td>H₂O₂: NH₄OH: DI (1:1:5)</td>
<td>80 C</td>
<td>15 min</td>
</tr>
<tr>
<td>DI Rinse</td>
<td></td>
<td>Room</td>
<td>2 min</td>
</tr>
<tr>
<td>Oxide Clean</td>
<td>HF: DI (1:50)</td>
<td>Room</td>
<td>15-20 sec</td>
</tr>
<tr>
<td>DI Rinse</td>
<td></td>
<td>Room</td>
<td>10-15 Sec</td>
</tr>
<tr>
<td>Ionic Clean</td>
<td>HCl:H₂O₂:DI (1:1:5)</td>
<td>80 C</td>
<td>15 min</td>
</tr>
<tr>
<td>DI Rinse</td>
<td></td>
<td>Room</td>
<td>2 min</td>
</tr>
<tr>
<td>Oxide Clean</td>
<td>HF: DI (1:50)</td>
<td>Room</td>
<td>15-20 sec</td>
</tr>
<tr>
<td>Dry with N₂ gun</td>
<td></td>
<td>Room</td>
<td>As needed</td>
</tr>
</tbody>
</table>

Table 8. RCA cleaning procedure
Appendix B Detector Evaporation Procedure
This appendix contains detailed information on the evaporation procedures used to fabricate the silicon carbide Schottky diode detectors. All of the evaporations took place at the Nanotech West cleanroom located on the west campus of The Ohio State University. A CHA Solutions electron beam evaporator was used to deposit the metal on the silicon carbide, Figure 12, and a rapid thermal annealer (RTA), Figure 13, was used to perfectly form the Schottky and Ohmic contacts.

It is very important to remember to properly wash the samples according to Appendix A. Improper washing will leave a residue and/or oxide layer on the detector, which will interfere with contact deposition and alpha particle collection. The first step in the process is to deposit the ohmic contact followed by annealing, then to deposit the Schottky contact and anneal once again.

To evaporate the ohmic contact, follow all directions for operating the evaporator as outlined by the Nanotech West staff in the equipment procedure. The ohmic contact should be 100nm thick of pure nickel. Properly secure the substrate side of the SiC in a sample holder with a wire or metal clip as seen in Figure 81. As little silicon carbide as possible should be covered up by the wire or clip. For the first 80 angstroms, the nickel should be evaporated at 0.5 angstroms/second. After that, the nickel should be evaporated at 3 angstroms/second up to 900 angstroms. The rate should be ramped to 3 angstroms/second in 40 seconds. Finally for the last 100 angstroms, the deposition rate should be ramped down to 0 angstroms/second in 1 minute and 7 seconds.
After the deposition, the SiC should be placed in the RTA on a pyrometer disk with the metalized side of the SiC facing up. The pyrometer disk must be used because the temperature is too high for the thermocouple to function properly. The process ZELAS950 was used to anneal the ohmic contact. This process ramps the heat up to 700 degrees Celsius, then 900 degrees Celsius and finally 950 degrees Celsius, where it remains at steady state for 30 seconds and then promptly turns off. Afterward, the RTA cools down for 900 seconds.

Next, the SiC samples must be placed back in the evaporator for deposition of the Schottky contacts. The contact holder as seen in Figure 10 and the masks seen in, Figure 11, must be used. The masks need to be put in the shallower depressions cut in the
sample holder and the clean side of the SiC placed on top. It is important to center the
piece of SiC as much as possible to reduce the probability of forming a contact that goes
off the edge of the SiC. It is difficult because the ohmic contact is facing up and the
clean side of the SiC is facing downward, touching the mask. Once the silicon carbide is
perfectly placed, it must be secured with wire and/or metal clips; refer again to Figure 81.
Take care not to scratch the ohmic contact when securing the device.

A similar procedure to the ohmic contact deposition should be followed for the
Schottky contact deposition. The only change is that in addition to the 100nm of nickel,
10nm (100 angstroms) of titanium and 10nm (100 angstroms) of gold should also be
deposited. The titanium works as a diffusion barrier between the nickel and gold and the
gold prevents the oxidation of the nickel. This small addition of metal should not
interfere with the collection of alpha particles. Again, for the first 80 angstroms, the
nickel should be evaporated at 0.5 angstroms/second. The nickel should then be
evaporated at 3 angstroms/second up to 900 angstroms, with the rate reaching its desired
deposition rate in 40 seconds. Finally for the last 100 angstroms, the deposition rate
should be ramped down to 0 angstroms/second in 1 minute and 7 seconds. The second
layer of metal is titanium and should be evaporated at a rate of 0.5 angstrom/second for
the first 10 angstroms and then 3 angstroms/second until 90 angstroms are deposited.
The deposition rate should ramp up to 3 angstroms/second in 8 seconds and ramp down
to 0 angstroms/second in 20 seconds to finish off the second layer of metal. This same
procedure should be followed for the 100 angstroms of gold, which is deposited as the
final layer.
After evaporation of the Schottky contact, the detectors should be annealed at 650 degrees Celsius for 30 seconds. The thermocouple wafer can be utilized this time, as the temperature is low enough for it to function properly. The procedure ZELAS650 was utilized to do this annealing. The temperature is ramped up to 250 and then 650 degrees Celsius. Upon reaching 650 degrees Celsius, the temperature is held constant at 650 degrees Celsius for 30 seconds. After that time, the RTA is shut off and cooled down for approximately 600 seconds or until the temperature falls below 150 degrees Celsius. Two point probe IV measurements can then be done on the fabricated detectors at Nanotech West to check their behavior.
Appendix C Silicon Carbide Dicing
This appendix contains information related to the dicing of silicon carbide for this project. As previously stated, SiC is one of the hardest materials, a 9 on the Mohs Scale of Hardness, with only diamond being harder. This makes cutting or dicing silicon carbide particularly challenging. Most companies use diamond blades to cut silicon carbide into its desired shapes.

Two dicing companies considered for this project were Kadco Ceramics of Easton, PA and American Dicing, Inc. of Liverpool, NY. Both companies are experienced with dicing silicon and silicon carbide. American Dicing, Inc. was used by Stephen E. Stone to dice his hall bars for research completed in 2008. Due to their significantly cheaper cost relative to American Dicing, Inc., Kadco Ceramics had previously been chosen to dice a few of Mr. Stone’s left over SiC samples for this project. Successful with these earlier samples, Kadco Ceramics was again selected to process the large, 2” wafer purchased for this project.

The dicing process went relatively smoothly aside from a few questions and sample location adjustments. One of the major difficulties in the dicing process was related to the complicated nature of the dicing map as seen in Figure 82. The dicing diagram became more complicated when it had to be flipped on the vertical axis to represent the epitaxial face, which was believed to be facing upward and instantly visible upon removing the top to the wafer box. A modified dicing diagram can be seen in Figure 83, which also includes the final sample numbers given by Kadco. Kadco Ceramics had originally assumed that the wafer would be lithographically patterned before it arrived in their lab. Unfortunately, it was not and instead required a very detailed dimensional drawing. After dicing, Kadco Ceramics sent back a copy of the
dicing diagram labeled with each sample number, Figure 83, and the detector samples numbered and arranged on a blue sticky paper as in Figure 84. These sample numbers allow each newly diced detector to be matched up to its original position.

Figure 82. Wafer dicing scheme for SiC wafer, serial number CM0786-04
Figure 83. Returned wafer dicing map with sample numbers

Figure 84. Diced detectors as returned from Kadco and shown next to two alumina substrates (left side). The numbers correspond to the locations in Figure 83
Kadco Ceramics stated that they returned each and every piece in the same orientation in which it was originally cut. Therefore, on all of the newly diced pieces the epitaxial face should be facing upward and the substrate or bulk should be stuck to the blue sticky paper. This seemed acceptable and detector fabrication began. Unfortunately, fabrication met with mixed success.

Despite multiple runs of cleaning, evaporating and annealing, the detector fabrication process was not consistently successful. Each time less than one-third of the samples were “successful” where “successful” was defined as a smooth contact that did not peel on the edges and had a leakage current on the order of $1 \times 10^{-9}$ amps or smaller at -50V as given by IV measurements. An example of a good and bad detector made during the same run can be seen in Figure 85. Sample 20 was a bad detector because the edges of its contact were peeling and it had a very high leakage current. On the other hand, Sample 19 was very smooth and reflective and had a small leakage current. Multiple detectors fabricated during other runs exhibited similar behavior. For example, Figure 86 shows a picture of a particularly bad detector fabricated during a different run. Sample 16 was discolored in the center and the edges had particularly bad adherence to the SiC.
Figure 85. Two detectors fabricated at the same time. (a) is Sample 19 and a “good” detector while Sample 20 (b) is a “bad” detector.

Figure 86. Photo of Sample 16, a particularly bad detector.
After numerous unsuccessful runs and much painstaking research, it was determined that not only was the original wafer’s epitaxy and substrate misidentified, the diced pieces had come back in an unknown orientation. Originally, the epitaxial layer was identified as being instantly visible upon opening the wafer box. After re-examining some documentation from Cree, this side was truly the substrate and the epitaxy was actually facing downward (not instantly visible). To further complicate the problem, some pieces of the diced SiC had the true substrate facing upward (the correct orientation) while other pieces had the true epitaxy facing upward (the incorrect orientation). Using a microscope, this discovery was confirmed through observations of detectors made during the same run as Samples 19 and 20. While looking for the reasoning behind some detectors being “good” and some not despite being fabricated the exact same way and at the same time, Dr. John Carlin at Nanotech West noticed that “scratches” or “striations” were visible on one side of each of the SiC samples. Sometimes these striations occurred on the ohmic side but more often they occurred on the Schottky side. Dr. Carlin suggested that if he were to guess, he would surmise that these striations were on the substrate face. Eventually, these striations were identified as a remnant of mechanical polishing on the substrate, carbon face as viewed in Figure 87.

The striated side was further confirmed to be the substrate side as these striations occurred on the same side as the laser printed serial number; see Figure 88. Figure 88 displays the serial numbers from Samples 7 and 17. Sample 17 also has scratches on top of the laser printed serial number, which were created to differentiate one side from another during fabrication. The scratches, created with a diamond tipped scribe, were always placed on the side of the silicon carbide that was immediate visible when peeling
back the colorless transparent layer that sealed the samples onto the blue sticky paper. The side that was scratched was not stuck to the blue sticky paper.

As expected, the other side of the wafer, the epitaxial face, was smooth since it was chemically polished. Cree, the manufacturer of the purchased silicon carbide wafer, confirmed these observations. This occurrence clarifies why some detectors were successful while others were not. If the Schottky contact was not deposited on the epitaxy as it should have been, there was, as expected, a high leakage current. Furthermore, the deposition of the Schottky contact on the striated side could explain why some contacts began to peel or turn up at the edges as in Figure 85 (b) and Figure 86.

![Figure 87](image.png)

Figure 87. Photos of observed striations on SiC taken through a microscope. (a) is of an unmetallized piece of SiC while (b) shows the striations still visible after metallization
Figure 88. Photos of laser printed serial number of substrate side of SiC diced wafer. (a) is from Sample 7 while (b) is from Sample 17.
Appendix D Alpha Spectra
This appendix contains some of the alpha spectra collected and analyzed (including data tables) that were not included in the body of this thesis.

D.1 Detector 01

Figure 89. Spectrum of Detector 01 for temperatures 24-200°C
Figure 90. Fitted peak for Detector 01 at T=100C

Figure 91. Fitted peak for Detector 01 at T=150C
Figure 92. Fitted peak for Detector 01 at T=245-255C

Figure 93. Fitted peak for Detector 01 at T=295-305C
Figure 94. Fitted peak for Detector 01 at T=345-355C

Figure 95. Fitted peak for Detector 01 at T=445-455C
Figure 96. Fitted peak for Detector 01 at T=495-505°C

Figure 97. Fitted peak for Detector 01 at T=23°C, post heating
Figure 98. Fitted peak with 2 Gaussians for Detector 01 at T=100C

Figure 99. Fitted peak with 2 Gaussians for Detector 01 at T=150C
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Table 9. Double and single Gaussian fitting parameters for data from Detector 01
D.2 Detector 08

Figure 100. Combined spectrum for Detector 08, Run 1

Figure 101. Fitted peak with Detector 08, Run 1 at T=50C
Figure 102. Fitted peak with Detector 08, Run 1 at T=100C

Detector 08, T=100 C, P=90 mTorr

\[ \mu = 2738.9 \text{ channel} \]
\[ \sigma = 16.0 \text{ channels} \]
\[ \text{FWHM} = 37.7 \text{ channels} \]

Figure 103. Fitted peak with Detector 08, Run 3 (082511) at T=24C

Detector 08, T=24 C, P=95 mTorr

\[ \mu = 2557.7 \text{ channel} \]
\[ \sigma = 132.7 \text{ channels} \]
\[ \text{FWHM} = 312.4 \text{ channels} \]
Figure 104. Fitted peak with 2 Gaussians for Detector 08, Run 1 at T=50C

![Graph showing fitted peak with 2 Gaussians for Detector 08, Run 1 at T=50C.]

- \( \mu_1 = 2732.6 \) channel
- \( \sigma_1 = 13.0 \) channels
- FWHM\(_1\) = 30.6 channels
- \( \mu_2 = 2711.3 \) channel
- \( \sigma_2 = 22.2 \) channels
- FWHM\(_2\) = 52.2 channels

Figure 105. Fitted peak with 2 Gaussians for Detector 08, Run 1 at T=100C

![Graph showing fitted peak with 2 Gaussians for Detector 08, Run 1 at T=100C.]

- \( \mu_1 = 2742.4 \) channel
- \( \sigma_1 = 12.4 \) channels
- FWHM\(_1\) = 29.2 channels
- \( \mu_2 = 2723.4 \) channel
- \( \sigma_2 = 21.8 \) channels
- FWHM\(_2\) = 51.4 channels
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Table 10. Double and single Gaussian fitting parameters for data from Detector 08, Run 1
Figure 106. Detector 21, Run 1 full spectra measurements taken between room (24C) and 200C
Figure 107. Fitted peak with Detector 21, Run 1 at T=48°C

Figure 108. Fitted peak with Detector 21, Run 1 at T=100°C
Figure 109. Fitted peak with Detector 21, Run 1 at T=150°C

Figure 110. Fitted peak with Detector 21, Run 1 at T=198°C
Figure 111. Fitted peak with Detector 21, Run 1 at T=195-205C

Figure 112. Fitted peak with Detector 21, Run 1 at T=245-255C
Figure 113. Fitted peak with Detector 21, Run 1 at T=255°C

Figure 114. Fitted peak with Detector 21, Run 1 at T=335-341°C
Figure 115. Fitted peak with 2 Gaussians for Detector 21, Run 1 at T=48°C

Figure 116. Fitted peak with 2 Gaussians for Detector 21, Run 1 at T=100°C
Figure 117. Fitted peak with 2 Gaussians for Detector 21, Run 1 at T=150C
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Table 11. Double and single Gaussian fitting parameters for data from Detector 21, Run 1

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Table 12. Double and single Gaussian fitting parameters for data from Detector 21, Run 2
Figure 118. Detector 21, Run 2 spectra for all measurements taken between 24 and 400°C

Figure 119. Fitted peak for Detector 21, Run 2 at T=300°C, Measurement 5
Figure 120. Fitted peak for Detector 21, Run 2 at T=400°C, Measurement 6 at 1500s

Figure 121. Fitted peak for Detector 21, Run 2 at T=400°C, Measurement 6
Figure 122. Fitted peak for Detector 21, Run 2 at T=24C, Measurement 1

Figure 123. Fitted peak for Detector 21, Run 2 at T=26C, Measurement 4
Figure 124. Fitted peak for Detector 21, Run 2 at T=300°C, Measurement 5, 1500s

D.4 Detector 05
Figure 125. Spectra for Detector 05 at 75 and 90C

Figure 126. Zoomed in spectra for Detector 05 at 75 and 90C
Figure 127. Fitted peak with 2 Gaussians for Detector 05 at T=75°C

- $\mu_1 = 2713.7$ channel
- $\sigma_1 = 23.4$ channels
- FWHM$_1 = 55.2$ channels
- $\mu_2 = 2702.2$ channel
- $\sigma_2 = 43.0$ channels
- FWHM$_2 = 101.3$ channels
Appendix E MATLAB Code
This appendix contains some of the MATLAB scripts used to analyze the data files associated with this project.

E.1 dofit.m, Single Gaussian Fitting

function[total]=dofit(detectorstring, filename, L, U)
% This function fits a gaussian to the data and saves the plot. It also
% records the sum total of the counts in +/- 3*sigma around mu in the file
% 'counts.txt'. The arguments to the function are as follows:
%  
%  - detectorstring: a string that ends up in the title of the graph after
%    'Detector'. Nominally it should be the detector number, but if you want
%    another annotation, you can set it here, e.g. '21, pre heating' will end
%    up as 'Detector 21, pre heating, T=...
%  - filename: the filename of the data file
%  - L: the lower bound of the data used for the fit
%  - U: the upper bound of the data use for the fit

% Parse the filename

% split filename
fn = regexp(filename, '.', 'split');
% remove the parens from the basename. ignore the extension (TKA)
basename = regexprep(char(fn(1)), '[()]', '-');
% convert it to a string - it was a cell array. who the fuck wants that? This
% will be used for the graph pdf file.
basename = char(basename);
% split the first part of the file name on parens and scrape for information
rundat = regexp(char(fn(1)), '[()]', 'split');
% Temp is the 1st part. convert to a string again - I'll no longer comment
% this action
temperature = char(rundat(1));
% replace the '_' in the file name with a '-'
temperature = regexp(temperature, '[_]', '-');
% Do some stupid shit to find the average temperature when there was a range of
% temps
tmp = regexp(temperature, '[_-]', 'split');
[crap, num] = size(tmp);
if num == 1
    avgtemp = str2num(char(tmp(1)));
else
    avgtemp = mean([str2num(char(tmp(1))), str2num(char(tmp(2)))]);
end
temperature = char(temperature);
% pressure is the 3rd part of the file name. ignore the 2nd part.
pressure = char(rundat(3));
% we need to strip out the _post crap in the filenames
pressure = regexp(pressure, '_', 'split');
payload = char(pressure(1));

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Get the data %
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

% Load the data
Y=load(filename)';
% Index of bin
X=1:4096;
% Set the first two values to zero
Y(1:2)=0;

% Apply our upper and lower bounds to the data. This is to constrain the fit.
A=X(L:U);
B=Y(L:U);

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Do the fit and some math %
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
lowerbounds = [10 0 5 10 0 5];
[tmp, middle] = max(B);
middle = middle + A(1);
initguess = [100 middle 40 100 middle-30 40];
% Calculate mean and standard deviation using gaussian fit
fitdat=fit(A', B', 'gauss2', 'Lower', lowerbounds, 'StartPoint', initguess);
% extract the fitted parameters from the fit object 'fitdat' for later use
p_out = coeffvalues(fitdat);
% order the pouts the right way. mu1 needs to be bigger.
if p_out(5) > p_out(2)
    N2 = p_out(1);
    mu2 = p_out(2);
    sigma2 = p_out(3) / sqrt(2);
N1 = p_out(4);
mu1 = p_out(5);
sigma1 = p_out(6) / sqrt(2);
else
N1 = p_out(1);
u1 = p_out(2);
sigma1 = p_out(3) / sqrt(2);
N2 = p_out(4);
u2 = p_out(5);
sigma2 = p_out(6) / sqrt(2);
end

% calculate the FWHM of the fitted gaussian
FWHM1 = 2*sqrt(2*log(2))*sigma1;
FWHM2 = 2*sqrt(2*log(2))*sigma2;

% do the count summing in +/- 3*sigma of the raw data, not in the fit
x0 = int16(mu1);
sigma0 = int16(sigma1);
x1 = x0 - 3 * sigma1;
x2 = x0 + 3 * sigma1;
total = sum(Y(x1:x2));

% Write the output files %
% Write the output files %

% open a file to append our count data to. The 'a' is to append data to a
% preexisting file.
myfile = fopen('counts.txt', 'a');
% print some output and close the file
fprintf(myfile, '%s	%i
', filename, total);
close(myfile);

% open a file to append the fit data to
myfile = fopen('mydat.txt', 'a');
% print some output and close the file
fprintf(myfile, '%s	%f	%i	%e	%e	%e	%e	%e
', filename, avgtemp, total, N1, mu1, sigma1, FWHM1, N2, mu2, sigma2, FWHM2);
close(myfile);

% this is not really used, but we could use it if we didn't feel like using the
% fit object directly to plot.
% Generate a pdf using the parameters determined by the gaussian fit
\[ g_1 = N_1 \cdot \exp(-((A-mu_1)/\sigma_1)^2); \]
\[ g_2 = N_2 \cdot \exp(-((A-mu_2)/\sigma_2)^2); \]

figure;
stairs(A-0.5, B, 'LineWidth', 2);
% bar(A-0.5, B, 1);
hold;

% we can do one of two things here. Either plot the gaussian using the
% parameters from the fit (plot(Z)), or use matlab's nifty fit objects (fitdat)
% and just plot that. I'm going to opt for the latter. However, I did pull
% the fit parameters out into specific variables, N, mu, and sigma. Plotting
% fitdat directly plots the fitted function over the whole X extents of the
% graph. If you prefer only to plot the fit over the bounds defined by (L,U),
% you would probably be better off uncommenting the next line and commenting
% out the line after (plot Z and not fitdat).
% h=plot(A-0.5,Z,'r');
h = plot(fitdat);
set(h,'LineWidth',2);
plot(A, g1, 'k--');
plot(A, g2, 'k--');

% set up the string for the title
titlestr = sprintf('Detector %s, T=%s C, P=%s mTorr', detectorstring, temperature,
pressure);
title(titlestr);
ylabel('Counts');
xlabel('Channel Number');
hleg2=legend('Data','Fit');
set(hleg2,'Location','NorthWest');

% get the axis limits for text placement
ylims = ylim;
xlims = xlim;
% 5% to the right of the left side of the graph
tx0 = (xlims(2) - xlims(1)) * .05 + xlims(1);
% 5% of y_max on the axis
tyd = (ylims(2) - ylims(1)) * .05;
% 15% down from the top of the axis - should clear the legend
ty0 = ylims(2) - tyd * 4;

% place our annotations
\text{txt}(tx0, ty0, [\text{\mu_1 = ', num2str(mu1, '%.1f'), '} channel']);
\text{txt}(tx0, ty0 - tdy, [\text{\sigma_1 = ', num2str(sigma1, '%.1f'), '} channels']);
\text{txt}(tx0, ty0 - 2*td, [\text{FWHM_1 = ', num2str(FWHM1, '%.1f'), '} channels']);
E.2 dofit2.m, Double Gaussian fit to data

function[total]=dofit(detectorstring, filename, L, U)
% This function fits a gaussian to the data and saves the plot. It also
% records the sum total of the counts in +/- 3*sigma around mu in the file
% 'counts.txt'. The arguments to the function are as follows:
% %
% % - detectorstring: a string that ends up in the title of the graph after

% old annotations. I left these here in case what I did wasn't satisfactory.
% I DIDN'T try to recreate the MeV notation. We can do that on select graphs
% if you want, or if it would be easier just to do it in the text of your
% thesis, do it there. But if you want it in the graphs, it should be
% relatively easy to do.
% text(mu-.285*mu,N*1.45,['\mu = ',num2str(mu, '%.1f\,' 'channel']);
% text(mu-.075*mu,N*.90,['Mu = ',num2str(mu*(5.485/mu), '%10.3e\,' 'MeV']);
% text(mu-.285*mu,N*1.35,['\sigma = ',num2str(sigma, '%.1f\,' 'channels']);
% text(mu-.075*mu,N*.80,['Sigma = ',num2str(sigma*(5.485/mu), '%10.3e\,' 'MeV']);
% text(mu-.285*mu,N*1.25,['FWHM = ',num2str(FWHM, '%.1f\,' 'channels']);
% text(mu-.075*mu,N*.70,['FWHM = ',num2str(FWHM*(5.485/mu), '%10.3e\,' 'MeV']);

% check to see if the 'fits/' directory exists. If it doesn't, make it.
if ~exist('fits', 'dir')
    mkdir('fits');
end

% set up our output filename for the graph to be in the fits directory, using
% hte nonextension part of the data filename and '-fit.pdf'. If you don't want
% a pdf, change pdf to what you do want.
outfn = strcat('fits/', basename, '-fit.pdf');
% save the file
saveas(gcf, outfn);
% close the figure. Look at the pdf instead. If you don't want to do that,
% just comment the close() out.
close();
'Detector'. Nominally it should be the detector number, but if you want another annotation, you can set it here, e.g. '21, pre heating' will end up as 'Detector 21, pre heating, T=...'

- filename: the filename of the data file
- L: the lower bound of the data used for the fit
- U: the upper bound of the data used for the fit

Parse the filename

% split filename
fn = regexp(filename, '\.', 'split');
% remove the parens from the basename. ignore the extension (TKA)
basename = regexprep(char(fn(1)), '[(])', ' ');
% convert it to a string - it was a cell array. who the fuck wants that? This will be used for the graph pdf file.
basename = char(basename);
% split the first part of the file name on parens and scrape for information
rundat = regexp(char(fn(1)), '[(])', 'split');
% Temp is the 1st part. convert to a string again - I'll no longer comment this action
temperature = char(rundat(1));
% replace the '_' in the file name with a '-'
temperature = regexprep(char(temperature), '_', '-');
% Do some stupid shit to find the average temperature when there was a range of temps
tmp = regexp(temperature, '-', 'split');
[crap, num] = size(tmp);
if num == 1
    avgtemp = str2num(char(tmp(1)));
else
    avgtemp = mean([str2num(char(tmp(1))), str2num(char(tmp(2)))]);
end
temperature = char(temperature);
% pressure is the 3rd part of the file name. ignore the 2nd part.
pressure = char(rundat(3));
% we need to strip out the _post crap in the filenames
pressure = regexp(pressure, '_', 'split');
pressure = char(pressure(1));
% Load the data
Y=load(filename)';
% Index of bin
X=1:4096;
% Set the first two values to zero
Y(1:2)=0;

% Apply our upper and lower bounds to the data. This is to constrain the fit.
A=X(L:U);
B=Y(L:U);

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%  
% Do the fit and some math  
%%%%%%%%%%%%%%%%%%%%%%%%%%%%
lowerbounds = [10 0 5 10 0 5];
[tmp, middle] = max(B);
middle = middle + A(1);
initguess = [100 middle 40 100 middle-30 40];
% Calculate mean and standard deviation using gaussian fit
fitdat=fit(A', B', 'gauss2', 'Lower', lowerbounds, 'StartPoint', initguess);
% extract the fitted parameters from the fit object 'fitdat' for later use
p_out = coeffvalues(fitdat);
% order the pouts the right way. mu1 needs to be bigger.
if p_out(5) > p_out(2)
  N2 = p_out(1);
mu2 = p_out(2);
sigma2 = p_out(3) / sqrt(2);
N1 = p_out(4);
mu1 = p_out(5);
sigma1 = p_out(6) / sqrt(2);
else
  N1 = p_out(1);
mu1 = p_out(2);
sigma1 = p_out(3) / sqrt(2);
N2 = p_out(4);
mu2 = p_out(5);
sigma2 = p_out(6) / sqrt(2);
end

% calculate the FWHM of the fitted gaussian
FWHM1 = 2*sqrt(2*log(2))*sigma1;
FWHM2 = 2*sqrt(2*log(2))*sigma2;
% do the count summing in +/- 3*sigma of the raw data, not in the fit
x0 = int16(mu1);
sigma0 = int16(sigma1);
x1 = x0 - 3 * sigma1;
x2 = x0 + 3 * sigma1;
total = sum(Y(x1:x2));

% Write the output files %
% open a file to append our count data to. The 'a' is to append data to a
% preexisting file.
myfile = fopen('counts.txt', 'a');
% print some output and close the file
fprintf(myfile, '%s	%i
', filename, total);
fclose(myfile);

% open a file to append the fit data to
myfile = fopen('mydat.txt', 'a');
% print some output and close the file
fprintf(myfile, '%s	%f	%i	%e	%e	%e	%e	%e	%e	%e
', filename, avgtemp, total, N1, mu1, sigma1, FWHM1, N2, mu2, sigma2, FWHM2);
fclose(myfile);

% this is not really used, but we could use it if we didn't feel like using the
% fit object directly to plot.
% Generate a pdf using the parameters determined by the gaussian fit
g1=N1.*exp(-((A-mu1)./sigma1).^2);
g2=N2.*exp(-((A-mu2)./sigma2).^2);
figure;
stairs(A-0.5, B, 'LineWidth', 2);
% bar(A-0.5, B, 1);
hold;

% we can do one of two things here. Either plot the gaussian using the
% parameters from the fit (plot(Z)), or use matlab's nifty fit objects (fitdat)
% and just plot that. I'm going to opt for the latter. However, I did pull
% the fit parameters out into specific variables, N, mu, and sigma. Plotting
% fitdat directly plots the fitted function over the whole X extents of the
% graph. If you prefer only to plot the fit over the bounds defined by (L,U),
% you would probably be better off uncommenting the next line and commenting
% out the line after (plot Z and not fitdat).
% h = plot(A-0.5, Z, 'r');
h = plot(fitdat);
set(h, 'LineWidth', 2);
plot(A, g1, 'k--');
plot(A, g2, 'k--');
% set up the string for the title
titlestr = sprintf('Detector %s, T=%s C, P=%s mTorr', detectorstring, temperature, pressure);
title(titlestr);
ylabel('Counts');
xlabel('Channel Number');
hleg2 = legend('Data', 'Fit');
set(hleg2, 'Location', 'NorthWest');

% get the axis limits for text placement
ylims = ylim;
xlims = xlim;
% 5% to the right of the left side of the graph
tx0 = (xlims(2) - xlims(1)) * .05 + xlims(1);
% 5% of y_max on the axis
ty0 = ylims(2) - ylims(1) * .05;
% 15% down from the top of the axis - should clear the legend
% ty0 = ylims(2) - tyd * 4;

% place our annotations
text(tx0, ty0, ['\mu_1 = ', num2str(mu1, '%.1f'), ' channel']);
text(tx0, ty0 - tyd, ['\sigma_1 = ', num2str(sigma1, '%.1f'), ' channels']);
text(tx0, ty0 - 2*tyd, ['FWHM_1 = ', num2str(FWHM1, '%.1f'), ' channels']);
text(tx0, ty0 - 3*tyd, ['\mu_2 = ', num2str(mu2, '%.1f'), ' channel']);
text(tx0, ty0 - 4*tyd, ['\sigma_2 = ', num2str(sigma2, '%.1f'), ' channels']);
text(tx0, ty0 - 5*tyd, ['FWHM_2 = ', num2str(FWHM2, '%.1f'), ' channels']);

% old annotations. I left these here in case what I did wasn't satisfactory.
% I DIDN'T try to recreate the MeV notation. We can do that on select graphs
% if you want, or if it would be easier just to do it in the text of your
% thesis, do it there. But if you want it in the graphs, it should be
% relatively easy to do.
% text(mu-.285*mu,N*1.45, ['\mu = ', num2str(mu, '%.1f')], ' channel']);
% text(mu-.075*mu,N*.90, ['Mu = ', num2str(mu*(5.485/mu), '%10.3e')], ' MeV']);
% text(mu-.285*mu,N*1.35, ['\sigma = ', num2str(sigma, '%.1f')], ' channels']);
% text(mu-.075*mu,N*.80, ['Sigma = ', num2str(sigma*(5.485/mu), '%10.3e')], ' MeV']);
% text(mu-.285*mu,N*1.25, ['FWHM = ', num2str(FWHM, '%.1f')], ' channels']);
% text(mu-.285*mu,N*.70, ['FWHM = ', num2str(FWHM*(5.485/mu), '%10.3e')], ' MeV']);
% check to see if the 'fits/' directory exists. If it doesn't, make it.
if ~exist('fits', 'dir')
    mkdir('fits');
end

% set up our output filename for the graph to be in the fits directory, using
% the nonextension part of the data filename and '-fit.pdf'. If you don't want
% a pdf, change pdf to what you do want.
outfn = strcat('fits/', basename, '-fit.pdf');
% save the file
saveas(gcf, outfn);
% close the figure. Look at the pdf instead. If you don't want to do that,
% just comment the close() out.
close();

E.3 EnergyFWHM.m, FWHM calculation of energy spread from SRIM

function [ output ] = energyFWHM( file_name,fn )
data = dlmread(file_name,'	');%make the txt read in correctly, tab delineated
output = data(:,3);%assign variable name
hold on
histfit(data(:,3),50)
[a,b]=normfit(data(:,3))

FWHM=2*sqrt(2*log(2))*b

s = strcat('Counts per Alpha Energy - ', fn)
title(s)
xlabel('Alpha Energy (eV)')
ylabel('Counts')
axis([5.34e6 5.48e6 0 3000])
h = findobj(gca,'Type','patch')
set(h,'FaceColor','b')

text(5.37e6,2300,['     Parameters'])
text(5.37e6,2150,['\mu = ',num2str(a, '%10.3e\n'),' eV'])
text(5.37e6,2000,['\sigma = ',num2str(b, '%10.3e\n'),' eV'])
E.4 rangeFWHM.m, FWHM calculation of range of alpha particles from SRIM

function [ output ] = rangeFWHM( file_name,fn )
data = dlmread(file_name);%make the txt read in correctly, tab delineated
output = data(:,2);%assign variable name

hold on
histfit(data(:,2),50)
[a,b]=normfit(data(:,2))

FWHM=2*sqrt(2*log(2))*b

s = strcat('Ion Range Primary Alpha -- ', fn)
title(s)
xlabel('Distance (Angstrom)')
ylabel('Counts')
h = findobj(gca,'Type','patch')
set(h,'FaceColor','b')

text(1.4e5,2500,['
\mu = ',num2str(a, '%10.3e\n'),' angstroms'])
text(1.4e5,2350,['\sigma = ',num2str(b, '%10.3e\n'),' angstroms'])
text(1.4e5,2050,['FWHM = ',num2str(FWHM, '%10.3e\n'),' angstroms'])

hold off

saveas(gcf,strcat(fn, '-histo'), 'pdf') %save graph of histogram with fit