X-RAY GAS IONIZATION STUDIES WITH A SINGLE-WALLED NANOTUBE (SWNT) SENSOR

A Thesis Presented

By

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ABSTRACT

Due to their high surface areas, Single-Walled Nanotubes (SWNTs) and their thin films, are wonderful media for sensing applications. To establish the radiation sensing mechanism and limits of an SWNT ion sensor, we performed gas ionization studies with an Amptek Mini-x x-ray source. We irradiated the sensor under varying conditions of source to sensor distance, gas concentration, and fill-gas type. The mechanism of the SWNT sensor, which is responsive to the gaseous ions the radiation generates, resembles that of conventional gas filled detectors with reduced power requirements. Additionally, the small size of the sensor favors the development of miniaturized and portable radiation detectors which retain high sensitivity. Sensor responsiveness to radiation, such as x-rays and gamma rays, can be maximized by enclosure in various noble gasses, which are chemically non-reactive, have small magnitudes of ionization potentials, and readily produce charged species when subjected to radiation.

When encapsulated with gaseous argon, the SWNT sensors were shown to be up to 246% more sensitive to 1.34 Sv dose of 20 keV x-rays than sensors enclosed in air. The dependence of the sensors’ electrical response to fill-gas material helps to push the minimum detection capabilities of this technology to new limits. Optimal performance may result through the use of more readily ionized gasses such as xenon or penning gas mixtures. Through Monte Carlo simulation and PIN diode measurements, the x-ray source intensity was determined and used to establish exposure rates for each test case. These sensors display changes in resistivity when exposed to charged ions, with no direct response to x-rays, gamma rays, and neutron radiation particles.
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Special appreciation to the Army for granting the opportunity to participate in this fellowship; hope to build from the learning experience here and use it for those we lead.

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Mama, Dada, Akos, Ben, Gyedua, and the house of PANE Afey Meh Wei.

To I AM all praise is due. 32:2
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1 INTRODUCTION

Many modern devices are increasingly designed with advancement of functionality as the process guiding goal. Next generation devices will need to leverage nano scale technology and properties to continue the advancement of multi-functioning tools which rely on decreasing levels of power. For radiation detection, Carbon Nanomaterials (CNM) has been shown to be suitable sense media for gas ionization measurements by Modi et. al [23]. The small scale electrical properties of this class of material are very promising for the development of highly sensitive low power devices. The potential for these materials to be incorporated in to lithographical processes makes devices built with CNMs to be possible for integration into current Ultra Large Scale Integrated (ULSI) components [17]. This paper evaluates a Single-walled carbon nanotube (SWNT) as an x-ray detector. The SWNT device works by detecting the presence of ionized gaseous ions and molecules from a dose of radiation. For cations the SWNT sensor displays an increase in resistivity and a decrease in current, for anions the electrical response is opposite [17].

1.1 Overview

In this paper we start with an explanation of the different types of radiation that can be detected, describe how a radiation transport model was used to model the x-ray source in the experiments, and show how to calculate energy spectra and deposition in different materials. We investigate the mechanisms by which x-rays generate an electrical response in a conventional silicon-based diode radiation detector and the SWNT sensor. We then present an experimental setup that can be used to calibrate the SWNT sensor relative to the diode based on simulation data. The experiment involves distance variation, air pressure variation, and fill-gas variation. After a thorough analysis of sensor performance future avenues for improved design and evaluation are presented.

Compared to conventional ion gauges, it’s predicted that the SWNT sensor will display greater efficiency in radiation detection with a lower power supply. Overall this experimentation should yield data to support further miniaturization and design
integration of the sensor. The SWNT sensor (left) is shown in Figure 1 below along with the Amptek Mini-X (right).

![Image](image_url)

Figure 1. SWNT Sensor on SiO$_2$ substrate, shown with connecting electrodes (left) and Amptek Mini-X x-ray source used for sensor irradiation (right) [1].

2 RADIATION FUNDAMENTALS AND TRANSPORT

Much of the uncertainty and fear around radiation stems from the invisible nature of these energetic particles. Devices which can detect these particles are greatly needed for monitoring and safety. Radiation particles and radiation sensors can be used in a controlled manner for imaging, focused health treatment, and even environmental monitoring [14]. Radiation transport simulations are routinely used to aid in the development of sensors, hardware, and materials designed to perform in a radiation field. MCNP6, Monte Carlo n particle, is radiation transport simulation software which applies probabilistic weighting to simulate the interactions of subatomic particles [6].

2.1 Sources

Ionizing radiation can be broadly divided between directly ionizing and indirectly ionizing radiation. Directly ionizing radiation consists of charged particles which lose energy through charged based interactions with electrons in the atomic cloud. This group of radiation includes alpha particles (helium nucleus), beta particles (electrons), and protons (hydrogen nucleus). Indirectly ionizing radiation carries no charge and must first generate ions through collisions which then result in the production of ions from
collisional excitation. Included in this class of radiation are neutrons, x-rays, and gamma rays [14].

Overall the particles of radiation interact with their surroundings by depositing energy to the matter they interact with. Each particle type has different modes of energy loss and thus varying potential to cause damage to a surround material.

<table>
<thead>
<tr>
<th>Particle</th>
<th>Ionization</th>
<th>Key Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alpha</td>
<td>Direct</td>
<td>Bethe formula (Collisional Energy loss): $-\frac{dE}{dx} = \frac{4\pi e^2}{m v^2}NB$</td>
</tr>
<tr>
<td>Proton</td>
<td>Direct</td>
<td>Radiative Energy loss: $-\frac{dE}{dx} = \frac{NEZ(Z+1)e^4}{137m_o^2c^4} \left(4\ln\frac{2E}{m_o c^2} - \frac{4}{3}\right)$</td>
</tr>
<tr>
<td>Beta</td>
<td>Direct</td>
<td>Photon Attenuation ($\mu$) summation: $\mu = \tau(\text{photoelectric}) + \sigma(\text{Compton}) + \kappa(\text{pair})$</td>
</tr>
<tr>
<td>Gamma</td>
<td>Indirect</td>
<td>Neutron Attenuation Summation: $\frac{I}{I_o} = e^{-\Sigma_{\text{neutron}} \cdot t}$</td>
</tr>
</tbody>
</table>

Table 1. Summary of Radiation Particle types and associated equations [14]

As seen in Table 1 charged particles of heavy mass lose energy primarily through collisional transfers with their surroundings. Low mass charged particles such as electrons additionally lose energy through radiative processes. Uncharged particles rely on attenuation through media to transfer their energy. For neutrons, which can be modeled with the equation in Table 1, energy loss depends on heavily on the energy dependent material cross sections for interactions $\Sigma$ and the thickness of the material $t$. In Figure 2, x-rays from the Amptek Mini-X were used to expose photographic film and the resulting beam profile can be seen, with greatest intensity at the center.

Figure 2. Exposure image of Amptek Mini-X, x-ray source beam at 50 kV and 80uA operation
2.2 Interaction Types and Effects

As seen in Table 2, mass and charge properties of the different radiation particles play a large influence in determining both the diffusion path and materials which can be used to shield the given radiation.

<table>
<thead>
<tr>
<th>Radiation Particle</th>
<th>Characteristics</th>
<th>Stopping Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alpha</td>
<td>Heavy mass, low scattering</td>
<td>Gas</td>
</tr>
<tr>
<td>Beta (Electron)</td>
<td>Highly scattered diffusion path</td>
<td>Thin Solid (Paper)</td>
</tr>
<tr>
<td>Proton</td>
<td>Highly linear diffusion path</td>
<td>Concrete</td>
</tr>
<tr>
<td>Neutron</td>
<td>Highly linear diffusion path</td>
<td>Low atomic number material (water, hydrogen, boron)</td>
</tr>
<tr>
<td>X-ray</td>
<td>Linear travel path, Massless</td>
<td>Material with high number of protons such as lead or tungsten</td>
</tr>
<tr>
<td>Gamma</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Summary of general characteristics for various radiation particles [16]

If all the particles had the same energy, alphas would by far travel the shortest distance, these particles can be routinely stopped by a small volume of gas such as air due to their high rate of charge ionization with the electron cloud. Beta particles exhibit a more random diffusion motion but are nevertheless generally shielded by thin layers of solid such as paper or clothing. As such alpha and beta particles are generally harmless external sources but can wreak havoc as internal ionizing sources [16]. Protons, like alpha particles, generally travel in straight paths; this particle is usually highly energetic and generates many secondary ions as it decelerates. Photons, like neutrons, generally travel in straight paths but neutrons scatter more readily in low atomic number material while photons release energy more readily in high atomic number materials. Biological effects from radiation include collisional energy transfer to organic matter, ionization of molecules, and destruction of cells/tissue. Material effects of radiation damage include damage to the crystal structure of material; through excitation of substituent particles to a higher unstable state. Ultimately, this could make the material unsuitable for functional service due to electrical property changes [21].
X-rays interact with matter primarily through the photoelectric effect. As shown in Figure 3, for energies less than 511 keV the photoelectric effect (PE) is the dominant process through which energy transfer occurs [16]. In PE, incident radiation interacts with the atom of a material through collisions which excite bound electrons. As electrons return to the ground state they emit photons of energy equivalent to that of incident particle minus initial electron binding energy. Interaction probability increases greatly with decreasing energy and increasing material atomic number as shown in Figure 3.

Figure 3. X-ray and gamma ray interactions with matter [14]

### 2.3 Measurement Units

As seen in Figure 4, dose is a measure of the energy deposited per unit mass, while exposure refers to the charge deposited per unit mass by incoming radiation [21]. Information for absorbed dose is in the left column while the data for exposure is in the right column. The units for dose are in mrad(Radiation Absorbed Dose)/hour, while for exposure it’s m(Roentgen)/hour. Both equations depend on the radiation energy with an integration required when the source is poly-energetic. Material effects can be seen in the values of the attenuation coefficient ($\mu$) which will depend on the material of concern and density which is a material property.
Dose can be quantified in two ways: KERMA (Kinetic Energy Released in Matter) and absorbed dose [21]. In the context of this experiment KERMA refers to the transfer of kinetic energy from low energy photons to gas molecules. As a measure of dose, KERMA tracks the energy transfer from photons through the photoelectric effect and assumes the energy absorbed by the target medium is equivalent to the energy imparted by ionizing radiation to intermediary particles. KERMA is therefore a theoretical maximum value for the imparted dose. Depending on particle energy and target geometry KERMA can be equivalent to absorbed dose. Absorbed dose as modeled in the equation in Figure 4, accounts for the energy deposited by ionized particles in the material of interest. Absorbed dose is heavily dependent on the diffusive behavior of the ionized particles in the irradiated material. For small volumes and low material densities not all of the energy can be contained in the volume due to diffusion. As material density and volume increase, it becomes increasingly more probable that ionized electron particles will fully deposit their energy within the volume of interest. If these particles completely scatter and deposit energy within the volume of interest then absorbed dose will be equivalent to KERMA [21]. Conversely, if ionized particles are energetic enough to scatter outside the target material before depositing all their energy then KERMA and Absorbed dose will differ. Equation 1 below, gives a ratio of the parameters that govern
the relationship between KERMA and Absorbed dose. For the work in this thesis, the ratio between KERMA and absorbed dose is unity for the silicon based detector but not the gas based SWNT sensor.

\[
\frac{D(z)}{K(0)} = \frac{\mu_e}{\bar{\mu} - \mu_e} (e^{-\bar{\mu}z} - e^{-\mu_e z})
\]  

Equation 1, relating absorbed dose \( D(z) \) to KERMA \( K(0) \), \( \mu_e \) is the linear attenuation coefficient for electrons in the material of interest, \( \bar{\mu} \) is the radiation source’s mean attenuation coefficient, and \( z \) is the material thickness [25]. When absorbed dose and KERMA are equal then the material in which radiation is being deposited is said to be in equilibrium. As shown in Figure 5, the thickness required for this equilibrium varies with the radiation type, energy, and the target material’s attenuation factors for the incident radiation and secondary particles it may generate [26]. For this thesis, this equilibrium occurs if incident x-rays in the volume of interest generate secondary electrons which subsequently fully de-excite within the material of interest. Generated electrons must fully deposit energy they gain from the x-rays whilst not escaping the region of interest.

![Plot of ratio between absorbed dose to KERMA vs. material thickness](image)

Figure 5. Plot of ratio between absorbed dose to KERMA vs. material thickness [26]

Exposure normalizes dose to units which account for the amount of energy, \( W \), necessary to ionize a molecule and create an ion within air. For example, in air about 33
eV of energy is required to liberate an electron and create cations and anions of air molecules. The amount of energy deposited in a mass of air can easily be converted to charge the liberated in that mass. Equation 2 below provides the relation between KERMA $K$ and exposure $X$. For comparison a noble gas such as argon has a $W$ value less than that of air. For equal volumes of argon and air, more ions would be generated in argon than air based on Equation 2.

$$X = K\left(\frac{e}{W_{\text{air}}}\right)\frac{1 - \bar{g}}{1}$$  \hspace{1cm} (2)

In Equation 2 that relates exposure $X$ to KERMA $K$, $W_{\text{air}}$ is the energy required to generated ion pairs in air, $e$ is the electron charge, and $\bar{g}$ is the radiative fraction which is a function of material and radiation energy [26].

In Figure 4, the factor $f$ is used to account for the material and radiation energy properties one needs when converting from exposure to dose. Overall differences in material reactions to radiation can be leveraged to differentiate and characterize unknown material. Each material will generate a unique amount of ions and thus absorb a specific charge character from an energy deposition of radiation [14].

2.4 Transport simulations with Monte Carlo N-Particle 6

Radiation transport simulations allow for the modeling of radiation fields and interactions. A useful application for transport simulations is the design optimization of machines that generate x-ray radiation via Bremsstrahlung. Machines that generate high energy x-rays through the deceleration of electrons must be optimized so that on average the bulk slowing down of electrons through a material medium maximizes x-ray output. Transport can be used for both the design and analysis of devices such as the Amptek Mini-X x-ray source. As seen in the left plot of Figure 6, the Mini-X produces the greatest amount of x-rays with a decelerator thickness of 1 um. This confirms the actual used design thickness of 1 μm ± 0.1 μm [1]. In transport, design trade studies of the braking medium thickness is much easier and more efficient for determining the
maximum output versus experimentally preparing costly samples and iterating towards the solution. Likewise in the right plot of Figure 6, the transverse profile was calculated to aid in radiation tests where dose uniformity is desired.

Transport simulation can also be used for optimization of radio-therapy devices. For medical treatments radiation must be focused to give a max dose at the target site while minimizing energy deposition in healthy tissue [27]. These sources must be optimized to ensure dose to healthy tissue is significantly below that for the cancerous target. In gamma-based treatments Cobalt 60 sources are aligned in a hemispherical pattern to focus dose and ensure fine beam profiles while preserving intensity. Narrow beam profiles help limit the region of healthy tissue subject to dose, while a higher intensity aids in a larger concentrated dose at the target site.

Figure 6. Amptek Mini-X Decelerator thickness optimization plot (left) and transverse beam (right).

MCNP6, Monte Carlo N Particle, is simulation software which applies probabilistic weighting to simulate the interactions of subatomic particles. The Monte Carlo method is well documented and has been used to generate solutions to stochastic processes such as the estimation of inherent noise levels [7]. MCNP6 is written in Fortran90 with inputs as text files from programs such as notepad or notepad++. The code is a combination of MCNP5 and MCNPX, version 6.1 (2013) is the version used for this experiment’s modeling [6]. Coupled with VISED (Visual Editor) geometric plots of each problem can be made before simulations are run as shown in Figure 7 on the next page.
Figure 7. MCNP6 model of Amptek Mini-X x-ray source with dosimeter [31].

At its core, MCNP6 is a Monte Carlo methods based calculator for the 3D Boltzmann equation shown in Equation 3:

\[ \boldsymbol{\Omega} \cdot \nabla \phi(r, E, \Omega) + \mu_{\text{Intrxn}}(r, E)\phi(r, E, \Omega) = \iint dE'd\Omega' \mu_{\text{Prod}}^{\text{2ndry}}(r, E' \rightarrow E, \Omega' \rightarrow \Omega)\phi(r, E', \Omega') + \text{ExtSource}(r, E, \Omega) \]  

(3)

From left to right the grouping of terms are \( \boldsymbol{\Omega} \cdot \nabla \phi(r, E, \Omega) \) which represents the starting energy and flux profile of the radiation source, \( \mu_{\text{Intrxn}}(r, E)\phi(r, E, \Omega) \) accounts for collisional interactions the generated particles will undergo with surrounding media based on attenuation coefficients, \( \iint dE'd\Omega' \mu_{\text{Prod}}^{\text{2ndry}}(r, E' \rightarrow E, \Omega' \rightarrow \Omega)\phi(r, E', \Omega') \) is the production of secondary from the collisions initial particles undergo in transport, \( \text{ExtSource}(r, E, \Omega) \) helps account for external radiation sources which may affect the transport of primary and secondary particles within the area of interest.

For simple geometries the Boltzmann equation can be reduced to yield closed form algebraic solutions. In the case of one-dimensional (1D) transport with no secondary particle production or external radiation sources, the solution to the 1D Boltzmann equation is given in Equation 4. This solution describes the exponential attenuation of incident radiation in matter.

\[ \phi_{\text{trans}} = \phi_{\text{inc}} e^{-\mu_1 x} \]  

(4)
Codes like MCNP6 work by simplifying particle transport to a process governed by probability distributions. This method of solution is the same as that describing the random walk diffusion of a dopant atom in a crystal lattice. As with crystal impurities the movement of radiation particles can be modeled by pathways of probability which are tied to the interactions the particle may undergo such as scattering, absorption, or reflection. Careful tracking can then be applied to determine energy transfer, spatial distribution, and particle conversion and loss [4]. Outside of MCNP6 Monte Carlo methods have been accurately used to estimate the energy spectra of Bremsstrahlung processes [30].

Shown below in Figure 8, on the left, is an MCNP6 calculation of the x-ray spectrum for the Amptek Mini-X x-ray source. The MCNP6 model includes the deceleration of 50 keV electrons through a 1um gold foil and 127 um of beryllium as described in the product manual [1]. The calculated spectrum compares accurately to the manufacturer provided spectrum in Figure 8 on the right. The two dominant peaks are from gold’s L-shell characteristic x-rays [35].

Figure 8. MCNP6 modeled plot of the Amptek Mini-X x-ray energy spectrum (left) and a plot from the Amptek product manual (right) [1].
2.5 X-ray Source model with Dosimetry

In the Amptek Mini-X, x-rays are generated from the deceleration of electrons through a gold foil. The resulting emitted x-rays can be modeled and plotted with MCNP6. The code provides a number of tallies by which particle flux, current, and energy deposition can be recorded and plotted [6]. The Amptek design includes a beryllium window which works to absorb any electrons that aren’t converted to x-rays in the gold and prevents them from contributing dose to the target [1]. Gold is a good material to generate x-rays because it has a high density of electrons. The high density electron cloud allows it to be thin so that there is a high probability of electron-electron excitation and thus x-ray generation, and the gold will be thin so generated x-rays will not be attenuated and can escape [35].

Figure 9. X-ray Ion Generation Theory. Illustration of x-ray generation from electron beam deceleration. Electron beam comes in from the left, decelerates through gold foil, and generates x-rays. These x-rays are filtered through the beryllium layer and can then subsequently ionize gas molecules such as air.

Figure 9 is an illustration of the Bremsstrahlung generation process in gold. Electron-electron excitation leads to x-ray emission; through collimation these particles can be
concentrated to create an x-ray beam. Sample MCNP6 code for this modeling is presented in Appendix B. The electrons are transported through a thin gold foil, and by applying interaction probabilities MCNP6 is able to generate an output of the expected x-ray flux and the corresponding spectrum [4]. When combined, this information can be used to predict the dose and flux field of the Amptek Mini-X source.

From earlier, the material factor ($f$), relating dose from a material such as silicon or tissue to exposure or air ionization, can be calculated through MCNP6 modeling. This is done by depositing x-rays with the energy spectrum of the Mini-X into equivalent volumes of air and silicon at their nominal densities and taking the ratio of the energy deposited between the two calculations.

![Figure 10](image.png)

*Figure 10. Plot of MCNP6 calculated dose in silicon (red) vs. dose in air (blue)*

Sample code for this calculation can be found in Appendix C. Plots were generated by comparing equivalent volumes of silicon and air and are shown in Figure 10. Dose in silicon is higher because silicon is denser than air; essentially there are more electrons with which incoming x-rays can scatter and deposit their energy. Additionally, once excited, these electrons can further undergo collisions with rest state electrons by which they can subsequently continue the process of energy deposition.
3 RADIATION DETECTION

Although x-rays are massless particles, detection can be done by exploiting the processes of ionization. Gaseous ionization gauges exploit the charge liberated in air as a means of quantifying x-ray radiation intensity [14]. Another method is through the ionization of materials such as semiconductors. In this approach incoming radiation generates electron-hole pairs whose current flow can then be equated to radiation intensity [21]. Different forms of radiation require slightly varied methods of detection based on characteristics discussed in section 2. With the use of instrumentation, radiation events are converted to an electrical response or optical discoloration which can be compared to a base level of background radiation.

As each x-ray travels in a medium it possesses an amount of energy with which it can interact with surrounding matter. For x-rays this interaction is predominantly photoelectric scattering with material electrons. When designing a detector the energy and charge characteristics of the radiation of interest serve as boundary conditions for detector design.

3.1 Conventional Detection Principles

<table>
<thead>
<tr>
<th>Design Parameters</th>
<th>Specific Factors</th>
</tr>
</thead>
<tbody>
<tr>
<td>Noise Minimization</td>
<td>Resolution, Fill Gas</td>
</tr>
<tr>
<td>Detection Efficiency</td>
<td>Dead time, Geometric efficiency</td>
</tr>
<tr>
<td>Carrier Migration</td>
<td>Recombination, Diffusion, Mobility</td>
</tr>
</tbody>
</table>

Table 3. Summary of design parameters for gas based radiation detection [14]

As seen in Table 3, design parameters common for radiation detection are noise reduction, efficiency, and information carrier migration. One method to minimize background noise is through the selection of fill-gas. Gasses with lower ionization energy
make it easier to differentiate narrower energy bands. Efficiency factors can be addressed through the selection of detector geometry which increases the active area open to radiation. Carrier migration can for instance be influenced by the strength of the applied electric field which would result in more force applied to generated ions.

Radiation detectors based on detection through the use of gaseous media can be grouped into Ion Chambers, Proportional counters, and Geiger-Mueller (GM) detectors. These three classes of detectors utilize a fill-gas as a means of generating an electrical response from radiation. Two methods in which radiation events can be measured from these devices are pulse mode and current mode [21]. In pulse mode each radiation particle interaction is responsible for a quantized electrical response and thus energy spectroscopy is possible. In current mode individual event histories are agglomerated over time to yield a unified electrical signal. In general, pulse mode is more reliable for precise imaging and radiation source discrimination. On the other hand, current mode is better for simple determination of the presence of radiation and the strength of the radiation field [14].

Geiger-Mueller (GM) detectors operate with a high voltage applied between an anodic wire which bisects a cathodic cylindrical housing. With incident radiation, negative gaseous species will drift to the outer housing while positive ions drift to the central wire. As seen in Figure 11, the charge separation results in a current flow driven by incident radiation. With GM detectors pulse mode operation is generally applied [14]. In essence charge liberated by incident radiation accumulates until reaching a threshold from which it is then released. GM detectors utilize an avalanche or “cascade effect” to amplify the electrical response from ionization radiation. A disadvantage of this design is the lack of connection between electrical response and radiation energy [21].

Figure 11. Schematic of common design of GM detector and Proportional counter [14].
Proportional counters, similar to GM detectors, operate from the basic schematic of Figure 11. In addition to an anode wire and the cathodic shell, these detectors also use high voltage potentials to induce charge multiplication from the ion pairs generated from radiation [26]. As they’re duly termed, Proportional counters exhibit linear charge increased based of operating voltage. Unlike GM detectors, Proportional counters maintain a direct relationship between ions generated from incident radiation and the detector’s output electrical signal. Electric field strength in the vicinity of Megavolts/meter is what is generally necessary for operation in the proportional detection region.

Ion Chambers likewise utilize high accelerating voltages to direct the flow of ionized charge from radiation events. These types of detectors are primarily operated in current mode with no significant signal amplification [14]. Ion Chamber detectors are limited in resolution by the energy required to ionize the fill-gas of the detector. As seen in Figure 12, a large voltage applied across parallel plates in the midst of a gaseous enclosure serves as the basis of the Ion Chamber. The subsequent current, driven by the action of the ionizing radiation with surrounding gas molecules, can then be measured and equated to specific radiation intensity. For radiation particles with charge, the Ion Chamber design both accumulates a direct current from these particles and also from the secondary ionization they generate as they move through the detector medium.

![Figure 12. Schematic of general Ion Chamber design [14].](image-url)
Similar to the Ion Chamber approach, the SWNT detector presented in this paper is a current mode device. The SWNT detector however leverages a minuscule potential voltage so the radiation induced electrical signal is severely limited by the mobility of ionized molecules drifting across the biased region. The benefits of this design approach are that less power consumption is required while still maintaining the “yes/no” capabilities of detectors operated in current mode. In terms of minimal detectable limit, SWNT detectors presented would seek to improve upon and at least be competitive with current Ion Chamber detection standards [17].

![Diagram of detector operating regions](image)

Figure 13. Common gas filled detector operating regions [10]

Figure 13 illustrates the increase in voltage necessary for detector operating in the Ion Chamber region, Proportional region, and GM detection regions respectively. Greater operating voltages allow for the incorporation of design components which amplify the signal of ions the detector measures. Depending on application constraints, one can then select the detector model most economical to the application. Due to the low operating voltages of the SWNT detector, it essentially detects radiation in the recombination
Thus the ions measured by the SWNT sensor are not all which are liberated by the incident radiation; but only those with the mobility to drift across the active region.

3.2 PIN Diode Detector

The PIN diode detector deviates from the aforementioned detectors in that it measures the radiation response from its interaction with a solid material. For a PIN Diode detector the material is silicon. Immediate differences between this solid state detector and that of gas filled detectors are carrier types and ionization energy required. In silicon, a semiconductor, the information carriers are electron and hole pairs; this is in contrast to the cations and anions produced in gases. Additionally the energy to create an electron hole pair is 3.6 eV while that required for the formation of cations and anions in gasses such as air, argon, helium, and nitrogen are 33.8, 26.4, 41.3, and 34.8 eV respectively [14]. The mobilities of electron and hole pairs in silicon are also magnitudes greater than that of ions drifting through a gaseous medium [12].

A marked benefit from the lower energy requirement of silicon is the ability to attain better energy resolution. Essentially bin width, which is limited by the energy required to generate the ion pairs, is 7-10 times smaller than that for gas based radiation detectors and accounts for the improved resolution [14]. Figure 14 provides a general schematic for the PIN Diode layout.

Figure 14. Hamamatsu PIN Diode schematic with an incident x-ray flux from the left [33].
The diode tested for this paper was a Hamamatsu PIN diode, model S3902. This diode has an intrinsic region of silicon sandwiched by two n and p-doped layers of silicon respectively. N-doped silicon has a greater concentration of electrons than holes, whereas p-doped silicon has the reverse concentration. Intrinsic silicon refers to un-doped crystalline silicon with the natural equilibrium balance between holes and electrons. The layer of intrinsic silicon helps to form a region of no electron and hole pairs known as the depletion region. Overall this PIN diode was encased in an epoxy cover glass as shown in Figure 14, with a ceramic backing and electrical leads protruding [33]. When reverse biased the depletion region becomes sensitive to ionizing radiation. The PIN diode can even be designed to generate electron-hole pairs in the presence of EM radiation in the visible range. The flow of liberated charge can then be equated to radiation intensity by linking energy deposited to generated current. As they are liberated, electrons and hole pairs are almost immediately swept from the depletion region to the p or n-doped region based on their polarity [9]. Figure 15 illustrates the depletion layer devoid of electrons (red) and holes (yellow). Upon the introduction of incident radiation, liberated electron-hole pairs result in a detectable current flow.

![Depletion Area](image)

**Figure 15.** Underlying illustration of a reverse biased PIN diode

When reverse biased at 70 volts, the width of the depletion region for the PIN diode becomes 0.3 mm, this region represents the volume available for electron and hole pair generation as incident radiation passes through the diode [33]. When combined with the density of silicon this volume helps with determining the mass in which incident
radiation is deposited. Radiation dose is defined as the energy deposited in a unit mass, so this measure of radiation is then readily calculated from the material parameters.

Overall the low energy required for ionization makes PIN diodes an excellent method for determining low energy radiation dose as the number of information carriers generated is a function of the W value of the material (for Si W = 3.6 eV) [21]. Generally, applied voltages greater than 50V are necessary to operate in reversed bias mode with a fully depleted intrinsic region although it can also be operated unbiased [33]. These devices provide a compact but low power requirement option for radiation detection. These devices can be used to detect higher energy radiation with the addition of filters such as Cu, Pb, or Sn [22]. Filters absorb the high energy radiation and release secondary particles which can then be detected with the PIN diode [9]. For experimental purposes the diode will serve as a control to benchmark the intensity of radiation events the SWNT sensor is exposed to. This calibration of sorts, will greatly aid in the determination of SWNT sensitivity.

3.3 SWNT Sensor

Since the identification of carbon nanotubes by Iijima in 1991 [3], CNMs have undergone intense research and investigation to harness their tremendous properties for structural and functional applications. SWNTs represent a 1-D structure with inner diameter generally around 1.4 nm [32]. Other carbon nanotubes include the Buckminster fullerene, multi-walled nanotubes, and graphene. Collectively this class of materials demonstrates ideal conduction of electricity, heat, along with tremendous structural strength. Many of the growth and processing techniques of CNMs such as SWNTs directly derive from processes common to silicon crystal growth and processing. Three methods of SWNT production are laser ablation, chemical vapor deposition, and arc discharge [32]. These varying approaches provide differing purities, diameters, and nanotube quality. Growth method is generally selected based on the application of fabricated tubes, i.e. economics govern the selection process.

Ongoing challenges with the growth of nanotubes include the production of characteristically uniform species which are also high in production concentration. Nanotubes have shown to be amenable to growth alignment and patterning [5]. This
holds promise for the use of SWNT’s in functional materials due to the possibility of arranging this small scale substance into electrically advantageous arrays. Overall manufacturing using SWNTs in solid and solution form already takes place on the large scale [17]. Techniques for SWNT identification and characterization include use of the Scanning Electron Microscope (SEM), Tunneling Electron Microscope (TEM), Atomic Force Microscope (AFM), and Raman Spectroscopy [3].

SWNTs can exist as semiconductor or metallic tubes. This distinction arises from the chirality that the nanotubes can possess. Semiconducting tubes can be doped p and n-type as with solid state silicon [12]. As with other semiconducting material SWNTs have been developed into transistors, which are fundamental devices for logic circuitry [3]. During the SWNT growth process, semiconducting tubes can be grown and selected for. Through functionalization it’s possible to separate between metallic and semiconducting tubes [32]. Processes today allow for solutions of SWNT which are over 95% purely metallic or semiconducting [17]. The increasing amount of selectivity for SWNT type allows for the application of the theorized SWNT properties due to uniformity in nanotubes that are used. Semiconducting material, as are SWNTs, can have direct band gaps as shown in Figure 16 as well as indirect band gaps.

![Figure 16. Band gap of semiconductor shown between Valence and Conduction bands (left) with band structure for direct band gap semiconductor (center) and indirect band gap semiconductor (right) [12].](#)
Limitations generally plaguing nanomaterials as sensors can often be minimized with efficient processing steps. Dr. Abdul Sha highlights several critical design factors for nano applications for gas sensors [32]. In this publication he highlights common properties to all gas sensors and then discusses avenues which could be taken by nanotubes in fulfilling these requirements. Some common traits mentioned are good signal discrimination, fast response time, low detection threshold, low power consumption, and good stability [32]. The allure of these properties served to drive the SWNT ion sensor development of this thesis.

Figure 17 illustrates the underlying working architecture and detection mechanism of the SWNT sensor. At the nanoscale quantum effects become increasingly pronounced and greatly influence the flow of signal carriers. There is an appreciable change in the electrical resistivity and thus conductivity of the nanotubes in the presence of ions. Incident cations result in increased resistivity and a coincident decrease in current while anions have the opposite effect [17]. There is the possibility that the irradiation will alter tube to tube connections and thus result in changes to the bulk band gap of the nanotubes over time [11].
During the fabrication process, the SWNT sensors are produced through steps common to lithographical devices. These steps include preparation, exposure, and etching cycles [36]. The gating is achieved with the help of a unique photo mask which ensures appropriate patterning of the SWNTs [17]. While in solution form, SWNT sensors were gated and then deposited as a thin film on top of the substrate (SiO$_2$) as labeled in Figure 18. Thin films were prepared from a commercially available SWNT solution. Thin films of SWNT allow for greater uniformity in SWNT electrical properties when compared to individual nanotubes [5].

Through the KOSTAS center at Northeastern University it was possible to spin coat wafers, complete necessary patterning, and conduct etching as needed. SWNT in solution form was pre-purchased from an outside vendor at low cost and with high concentration of semiconducting SWNTs. Raman spectroscopy was used to characterize the composition of the SWNT solution before fabrication. In this characterization method an electromagnetic laser is used to cause vibration in the sample. Each material resonates at an intrinsic frequency which can be used for identification [24]. This pre-characterization
allows for the identification of any irregularities which would result in abnormal SWNT sensor function.

As seen in Figure 19, the device architecture is built on a silicon substrate. Source-drain connectors such as titanium or gold could be for the contact region. Aluminum could also be used for the contact with the device still displaying comparable sensitivity at a much reduced cost. To ensure incident ions don’t disrupt the gating effect the SWNTs source-drain contacts are enveloped in inert photo resist material This helps ensure that incident ions don’t induce secondary interactions with the gold contacts [17]. Overall the miniature design of these SWNTs allow for them to be readily incorporated into existing silicon devices. The low operating voltages at which ionized particles can be measured makes this device similar to that of solid state detectors. The sensitivity of the sensor to low levels of charge flow builds upon the capabilities of present day detectors. To overcome challenges based on ion mobility, arrays of the SWNT devices could be integrated to provide a clearer ionization field perspective.

An area of concern for sensor performance is in regards to possible structural changes due to radiation interactions with the SWNTs. In Jamie E. Rossi et. al, mechanisms by which sensors based on CNMs may be damaged by an incident flux of ions during operation are explored [29]. This damage results from changes in geometry induced on
nanotubes by the incident radiation. Nanotube geometry is important because the energy band gap can vary with diameter changes [3]. Conversely, irradiation could be used to sculpt nanotubes into desired morphologies as demonstrated by McDonnell et. al [20]. It has been shown by Suzuki that SWNTs under a fluence of photons can undergo structural changes [34]. Although this damage is reversible, the process Suzuki mentions to promote defect healing such as inert gas high temperature annealing make such recovery impractical for continuous radiation monitoring applications. The massless nature of x-rays tremendously reduces the probability that mass balance defects take place under irradiation. The SWNT sensor will have to be design with this in mind. Essentially, a tolerance for ion induced electrical signal changes and modifications brought about from radiation collisions should be accounted for.

![Raman spectra shifts from 20 keV electron irradiation on SWNTs (left) and spectra recovery behavior as function of annealing temperature (right) from Suzuki [34].](image)

In Figure 20 the left image depicts differences in Raman spectroscopy from recovered, irradiated and unirradiated SWNTs. The frequency and thus wavelength of tubes has shifted left from irradiation. Upon annealing, SWNTs display a recovery behavior albeit incomplete. The figure on the right represents temperature annealing effects on the Raman profile of irradiated SWNTs. Lower temperature irradiated tubes are more susceptible to changes in vibration profile and thus Raman behavior [34]. This agrees with the work reported by Murakami et. al where it is reported that D to G band ratios experience dramatic changes after irradiation [24].
Overall the critical step in the sensing mechanism is ensuring that generated ions are incident on the detector surface and this could be addressed by fixing and controlling variables such as temperature, pressure, and humidity [28]. In their patent, Li et. al, describe such testing on an ion sensor; they provide analysis of ion sensor response in pressure controlled testing [17]. Their results build upon previous work with highly purified SWNT films as excellent material for vapor and gas detection [18]. Overall the SWNT sensor is based on indirect gas ionization with incident radiation and subsequent detection of these charged particles [14]. As with the PIN diode the carrier generation is a function of fill-gas w-value and density of molecules (i.e. pressure). Even at low applied voltages CNM can maintain high local electric field at their tips [23]. The high aspect ratio and sharp tips of nanotubes have allowed them to be used as x-ray emission sources [13]. Although radiation exposure can causes changes in the Raman spectra of nanotubes it is believed the x-rays in this experiment are not of sufficiently high energy to introduce structural defects to the tubes. CNM readily adsorbs gas molecules due to the relatively high surface area of these nano structures. A positive aspect of ion based detection is that SWNT using this mechanism could recover at a faster rate through ion desorption than they otherwise would with gas adsorption [37].

4 TESTING CONDITIONS

In order to establish the detection mechanisms, sensitivity profile, and stability behavior of the sensor, testing was conducted with distance, pressure and fill-gas variation for both the SWNT sensor and a PIN diode. For each test a low level bias of 0.2V was applied across the gated SWNT region with a KIETHLEY 2400 source meter. Once applied, the sensor was allowed to stabilize for a period of at least 20 minutes before data collection. Upon bias application, localized heating occurs between the electrical contacts, bonding material, and gold; this can result in wild current fluctuations within the first few minutes of sensor operation. For the PIN diode an applied bias of 70V was used; additionally no pretest stabilization period was necessary. For each type of test current versus voltage measurements were collected for ten minutes, with four minutes of data before 2 minutes of irradiation, and then another 4 minutes of data collection after the 2 minutes of irradiation. As seen in Appendix A, the current was recorded every
0.5 second interval within LABVIEW. Table 4, is a summary of the various test cases, variables of concern, and sensor performance predictions for each type of test.

<table>
<thead>
<tr>
<th>Test Case</th>
<th>Variables</th>
<th>Hypothesis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vary Distance</td>
<td>2,5,7,10 (cm)</td>
<td>↑ Sensitivity with ↓ distance</td>
</tr>
<tr>
<td>Vary Pressure</td>
<td>760,380,190,95 (torr)</td>
<td>↓ Sensitivity with ↓ pressure</td>
</tr>
<tr>
<td>Vary Fill-gas</td>
<td>Air, Ar, N2,He</td>
<td>↑ Sensitivity w / ↓ fill-gas W value</td>
</tr>
</tbody>
</table>

Table 4. Testing Table Summary with Test cases, variables, and hypotheses

4.1 Distance test

The primary goal of the distance test was to determine whether or not the SWNT sensor sensitivity followed the intensity fall-off of the x-ray source. Testing with the PIN diode was conducted at matching distances of the SWNT sensor. PIN diode data served to help corroborate x-ray intensity calculations with the MCNP6 software. Based on the circular nozzle of the Mini-X, it was predicted that x-ray intensity would fall as modeled by equation 5, where $z$ is the axial distance, $A$ is the dose at the face of the Mini-X, and $R_o$ is the radius of the disc opening of the source.

$$Dose(z) = A \times \ln[1 + \left(\frac{R_o}{Z}\right)^2]$$  \hspace{1cm} (5)

Supporting calculations for the Mini-X intensity fall-off profile are found in Appendix D. This fall-off can be attributed to the spatial dispersion of the ionizing particles as they distribute from the source to the target. With increasing distance, fewer x-rays would intercept with the PIN diode surface and generate electron-hole pairs. For the SWNT sensor a reduction in intercepting x-rays would likewise produce a decreased electrical response but this would be due to a decrease in the spatial density of ions generated.
Testing was performed within a brass enclosure to keep radiation inside and ensure ALARA irradiation standards were maintained.

![PIN diode](image1.png)

**Figure 21.** Typical PIN diode setup for distance testing.

As seen in Figure 21, the PIN diode was aligned horizontally and vertically with the Mini-X before testing. After alignment a 70V reverse bias was then applied, this was in order to ensure the depletion region was at the manufacturer provided depth. Data was then collected with x-ray source exposure each 0.5 seconds at distances of 2, 5, 7, and 10 cm’s. Results were then compared with the SWNT sensor and MCNP6 modeled data.

![SWNT sensor](image2.png)

**Figure 22.** Typical SWNT sensor setup for distance testing.

For each distance the SWNT sensor was first stabilized with a bias applied to electrodes as shown in Figure 21. After device stabilization data was then collected with x-ray source exposure and current recorded every 0.5 seconds at distances of 2, 5, 7, and 10 cm’s. Results were then compared with the PIN diode and MCNP6 modeled data. Similarly to the PIN diode the SWNT was also aligned before the 0.2 V biased was then applied. The device displays ideal electrical behavior in that its current is proportional to any applied voltage. 0.2 V was selected to minimize the bias driven localized heating, while providing ample resolution in current to allow for identification of incident ions.
4.2 Vacuum study

Pressure variation tests were useful in confirming that the SWNT sensor was sensitive to ionized gas molecules unlike the PIN diode which is directly sensitive to incident ionizing radiation. Testing with the PIN diode was conducted at pressures of 760 Torr and 45 Torr to establish that PIN diode electrical response was invariant to pressure. In order to achieve testing pressures of 95, 190, 380, and 760 Torr, a glass vacuum chamber with electrode outlets was used. The chamber had a volume of 961 cm$^3$, and could achieve an ultimate vacuum of 43 mTorr. Pumping was performed with an EDWARDS RV-12 pump with a pumping rate of 12 m$^3$h$^{-1}$. Pressure was monitored with a 1mTorr – 1500 Torr wide range diaphragm manometer and maintained ± 5 Torr during testing.

![Figure 23. Typical Vacuum pumping setup.](image)

As the left side of Figure 23 shows the general setup had the vacuum pump on the right connected to the glass chamber on the left. The SWNT sensor and PIN diode were electrically connected to the chamber through the enclosure insert. This design allows for electrical data to be collected under vacuum conditions. The glass chamber was connected to the vacuum using an NW 50 to NW 25 adapter. The right side of Figure 23
shows a close up of the x-ray source oriented at the sensor within the glass chamber. The biases applied to the PIN diode and SWNT sensor for distance variation studies were also applied for vacuum variation studies. As pressure decreased there were less and less gaseous molecules which could become ionized by the x-ray source. This trend drove the prediction that SWNT sensor sensitivity would decrease as a function of pressure while the PIN diode would remain constant across pressure changes. Results were compared for the PIN diode, MCNP6 modeling prediction, and SWNT sensor response measurements. Pressure variation testing was also performed with argon gas.

Figure 24. Glass chamber stopper close up with SWNT sensor attached.

For vacuum and fill-gas tests the sensor was attached to the glass chamber using the stopper shown in Figure 24 shown above. This stopper allowed for the measurement of current whilst maintaining the sensor under vacuum. Overall these studies provided comparison data for that collected at atmospheric conditions.

4.3 Fill-gas tests

Figure 25. Gas variation test setup for argon.

Gas variation tests helped to demonstrate the potential of the SWNT as an imaging device. Gasses tested were air, argon, nitrogen, and helium and all with different W values as shown in Table 5. It was predicted that argon, with the lowest W value would
result in the most generated ions and thus the greatest electrical response from the sensor. All gasses were tested and compared at atmospheric pressure of 760 Torr. For this batch of experiments no data was collected with the PIN diode. The glass chamber was first evacuated to maximum vacuum of 300 mTorr in the configuration shown in Figure 25. Each respective gas was slowly bled into the chamber to attain a pressure of 760 Torr.

<table>
<thead>
<tr>
<th>Gasses Tested</th>
<th>W Value (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>33.8</td>
</tr>
<tr>
<td>Argon</td>
<td>26.4</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>34.8</td>
</tr>
<tr>
<td>Helium</td>
<td>41.3</td>
</tr>
</tbody>
</table>

Table 5. Tested gasses and their W values [14].

Once at atmospheric pressure the sensor was stabilized with the 0.2V bias before irradiation with the Mini-X. Figure 26, below, shows the various gasses configured for testing. Except for air, all gasses tested were industrial grade purity from Airgas.

Figure 26. Gas variation testing setup for nitrogen.
With this selection of gasses it was desirable to determine if differences such as monoatomicity or diatomicity of the gas size played a role in SWNT sensor behavior. Additionally these gasses possess different atom sizes, ion mobilities, and recombination patterns. The results of this testing provide a good base from which subsequent design and improvement of the sensor can proceed. SWNT sensor performance across all gasses was compared to determine how much of a sensitivity increase could be gotten from fill-gas selection. Coupled with the distance variation tests and pressure variation tests, any changes in SWNT sensor sensitivity across fill-gas could readily be isolated to groupings of potential variables which may warrant future attention for design.

5 RESULTS & ANALYSIS

![Graph of sensor current over time]

Figure 27. Sample 10 minute test data (top) and subsequent sensor recovery (bottom).
Figure 27 provides a profile of sensor behavior upon exposure to two minutes of the Amptek Mini-X the SWNT sensor (top) and subsequent recovery behavior of the sensor. After initial stabilization the sensor is relatively stable with current fluctuations of 5-10 nA during the 4 minutes before and after irradiation. Over the two minutes of exposure the SWNT sensor was exposed to doses of 11.3 Sv at 2 cm, 0.43 Sv at 10 cm, and 1.34 Sv in the glass vacuum housing. Comparatively, dose for a routine CT scan of the abdomen would be about 0.01-0.02 Sv [27]. The SWNT sensor must be able to resolve an image in 0.21 seconds at 2 cm, 5.6 seconds at 10 cm, and 1.8 seconds in the glass vacuum housing at those respective dose levels to be used in current imaging processes. Additional exposure values for the Amptek Mini-X are provided in Tables 6 and 7 of Appendix D.

Figure 28 presents sample pretesting stabilization data for the SWNT after the application of a bias. Before testing each sensor was stabilized in its testing environment for a period of 30 minutes to isolate any trends which would govern irradiation behavior of the sensor. The natural rate of current decrease in the sensor is 0.125 nA/s. This rate can be attributed to localized heating between electrical connections and for each test is
recorded for four minutes before irradiation. After stabilization, data for distance variation, pressure variation, and fill-gas variation was collected and analyzed. This section presents the results of theoretical calculations along with experimental measurements on the rate of ion generation from the Amptek Mini-X. All results come from the x-ray source operating at 50 kV and 80 uA for a time period of two minutes. For each mode of testing a comparison is made between theoretical and measured results to tease out variables which may be influencing the deviations from predicted behavior.

5.1 Ion detection vs. distance

As shown in Figure 29, the SWNT should theoretically display the greatest sensitivity at 2 cm and then at 5, 7, and 10 cm, sensitivity should fall by approximately 6.3x, 12.9x, and 26.4x respectively. These values were found by calculating radiation intensity at 2, 5, 7, and 10 cm for the x-ray source. These values were then overlaid with electrical response values from the PIN diode. From MCNP6 the dose values at the mentioned distances are 10.12, 1.61, 0.78, and 0.38 Roentgen/s as provided in Appendix D. These exposure values were found by converting from the Monte Carlo calculated dose in silicon (MeV/g) values. The PIN diode current based approach related the current generated to a J/kg value of energy deposit in the intrinsic region of the diode. Due to the
un-calibrated state of the PIN diode data from it was primarily used to corroborate the radiation intensity fall off from MCNP6 calculations. There was good agreement between MCNP6 predictions and PIN diode measured intensity falloff.

Based on the experimentally measured 5cm data; theoretical current falloff for 2, 7, and 10 cm were then generated as functions of the x-ray source intensity at those locations. This approach ignores any limiting behavior in the sensor such as possible ion saturation i.e. at high ion generation rates the sensor fails to proportionally decrease in current. Another variable this model ignores is the effects carrier migration characteristics such as diffusion, recombination, and mobility may have on the lifetimes of generated ions as they migrate to the detector.

![Figure 30](image.png)

Figure 30. Experimental SWNT device sensitivity to radiation vs. distance.

Figure 30 presents data for the 2, 5, 7, and 10 cm test cases. Although there is good agreement between the 5, 7, and 10 cm test cases between the theoretical prediction and measured data, there is a large departure for 2 cm. For the closest distance measured of 2 cm, there is a factor of 3x difference between measure and predicted sensitivity. Possible factors for this discrepancy could be combinations of sensor saturation and carrier migration patterns. The case for sensor saturation is supported by testing at 3 and 4. At these distances the sensor again displayed underwhelming sensitivity to the number of ions that were generated. From this result it seems that at close distances or high
ionization rates the sensor’s ability to register ions fails to account for all incident particles.

Additional sources which could explain the difference between the theoretical prediction and measure data at high rates is the role of carrier migration factors. In order to be detected by the SWNT sensor, generated ions must flow across active the SWNT region. Although overall the device has a low applied bias, the needle like structure of the nanotubes make for high levels of electric field across the gates. This field provides a driving force to attract generated ions but is relatively confined to a small area of 0.5mm$^2$. Another factor affecting generated ion movement is general diffusion. As modeled by Fick’s law in equation 6, species have a natural tendency to flow from regions of high concentration to low concentration. In the equation for Fick’s second law the rate of diffusion $\frac{\partial c_i}{\partial t}$ is defined by the product of $D_i$, the diffusion coefficient of the medium of interest, with $\nabla^2 c_i$, which is the gradient between the regions diffusion is tracked [16]. As a result of this principle, generated ions will have a tendency to spatially disperse with only a small geometric percentage actually intercepting with the sensor.

$$\frac{\partial c_i}{\partial t} = D_i \nabla^2 c_i$$

(6)

As the ions drift in space there will also be a tendency for opposite charge species to come together and neutralize one another. This process known as recombination could be a potential source of loss of ions created and could result in a lower than the theoretical rate.

$$\frac{1}{n} = \frac{1}{n_o} + \alpha t$$

(7)

In the recombination equation 7, as a function of time $t$, the density of ions is represented by $n$, with $n_o$ representing the number of initially generated ions. The key variable in this expression is $\alpha$, the recombination coefficient which is a function of the medium in which experiments are conducted. For the gasses tested this coefficient is
relatively constant across pressure for each gas type [14]. At high ionization rates this may become the dominant mechanism by which generated ions are lost before detection.

5.2 Ion detection vs. pressure

For pressure variation studies, theoretical predictions were again obtained by normalizing other pressures to the experimentally values for one fixed pressure (i.e. atmospheric). It was expected that there would be a linearly proportional relationship between decreasing pressure and SWNT sensitivity as shown in Figure 31. Results were extrapolated for pressures of 92, 190, 380, and 757 Torr. The 92 Torr represented the lower limit in air at which an appreciable change in SWNT current behavior could be measured as a function of irradiation with the x-ray source.

![Figure 31. Theoretical SWNT sensitivity to pressure changes.](image)

Figure 32 is a plot of the experimental pressure dependence and the effects on SWNT sensitivity for air (top) argon (bottom). The argon data more closely aligns with the theoretical prediction. For air fluctuations in the SWNT signal contributes to the lack of close agreement between the theoretical model and experimental results. Due to the high sensitivity of the sensor in argon noise fluctuations have less of an impact for sensor current response at the tested pressures. Overall some discrepancies between the
measured and experimental results can be attributed in systematic error arising from difference between gauge pressure and the actual gas pressure in at the sensor.

Figure 32. Measured SWNT sensitivity to pressure conditions air (top) and argon (bottom).
5.3 Ion detection vs. fill-gas

To calculate the expected sensitivity due to fill-gas variation; material density, ionization energy threshold, and mobility were analyzed. Ionization energy displays an inversely proportional relationship with sensor behavior while material density and intrinsic species mobility have a linearly proportional relationship with SWNT sensor sensitivity. For instance in argon the percent current change was 2.24 times greater than that required for air. Argon has ion generation energy 1.3x less than air, and from literature it appears positives ions in argon have a 1.4x greater mobility in argon than positive ions do in air. Additionally argon is 1.38x denser than air at room temperature. When these 3 factors are combined you would then predict a response of about 2.5x greater sensitivity for the sensor in argon than air. This is only about 10% greater than the measured value.

For a gas such as helium, which has a higher energy required for ionization than air, less responsiveness is predicted for the sensor. Helium requires about 1.2x more energy to ionize than air, and has a density of about 7x less than that of air, these two factors alone indicate that the SWNT sensitivity will be about only 11.5% of that in air. Differences between argon, helium, and air can be attributed to variations in atom and molecule size.

Figure 33. Relative covalent radii of helium, nitrogen, and argon [19].
Figure 34. Theoretical prediction of current change for the SWNT sensor in nitrogen, air, argon, and helium.

For a gas such as nitrogen which has an equivalent energy required for ionization to air similar responsiveness is predicted for the sensor. Nitrogen requires about 1 eV more energy to ionize than air, and has a density of about 2% less than that of air. Combining these two factors leads to a prediction that SWNT sensitivity in nitrogen will be 5% less than that observed in air.

For a mixture gas such as argon with 0.5% acetylene a lower energy is required for ionization than in argon alone so even greater responsiveness is predicted for the sensor in this medium. This penning gas mixture requires about 1.6x less energy to ionize than air, and has a density equivalent to that of argon due to the low levels of acetylene doping. Such a mixture is called a Penning gas mixture, as the prevalence of the Penning effect results in lower levels of energy required for ionization. The Penning effect occurs when the dominant gas species is incompletely ionize to a metastable state and subsequently collides with and ionizes a low concentration quench gas [2]. Combined these factors result in a predicted SWNT sensitivity of 3.25x higher than that found in air. The quench gas, in this case acetylene, recycles energy that would’ve otherwise been trapped in the
metastable state. Good Penning gasses generally require the dominant gas species to have a metastable energy close to that of gas that is used to quench it [2]. Figure 35 below, is a plot of the experimental fill-gas variance and its effects on SWNT sensitivity. Compared to predicted values all gasses slightly underperformed but the performance difference between them matches nicely with predicted trends.

![Figure 35. Experimental values of current change for the sensor in nitrogen, air, argon, and helium.](image)

6 Conclusion

This series of experiments helped to lay the groundwork for the sensitivity limits of the SWNT sensor. Additional variables can be tested to establish boundary conditions for sensor performance. This could include experimentation with external applied electrical field, studies with temperature and humidity effects, experimentation with miniaturization (gaseous encapsulation), better stabilization of sensor before testing, experimentation with different voltages, and verification of possible x-ray damage to SWNTs using Raman Spectroscopy. Additional fill-gas variation could be explored for neutron detection applications. BF$_3$ or He-3 gas could be used as ion producing neutron capturing gasses. Additionally solid material incorporating B-10 could be used to generate ions from the neutron capture process [14].
This thesis analyzed x-ray gaseous ionization with the Amptek Mini-X source. Through distance variation, pressure variation, and fill-gas variation, the sensitivity limits of the SWNT sensor were established. The operation of the SWNT sensor in view of conventional radiation detectors was presented and compared and contrasted. Overall this device possesses great potential for future development. Although theoretical and experimental results didn’t show perfect agreement major trends of the hypothesis were nevertheless confirmed. Variations between these two sets of data could be attributed to systematic error.

The SWNT detector has great potential to be designed to track low rate single particle events. The detector operates at a lower voltage than that needed to deplete a PIN diode and thus would consume less power. The PIN diode has an advantage in ionization medium density of silicon versus gaseous media such as argon which gave the best tested performance for the SWNT detector. Both devices are highly sensitive to environmental temperature fluctuations [14] [28]. The PIN diode provides more information carriers (electrons/holes) per incident radiation [21]. Overall, the SWNT sensor can be more readily optimized to differentiate between different types of radiation through vacuum operation and fill-gas variation. The active area of the SWNT detector is 0.05mm² vs. that of the tested PIN diode active area of 1cm². A last advantage of the SWNT detector is that the ion charge dependence could allow for the direct discrimination between alpha and beta radiation [17].

6.2 Future considerations

Figure 36. Diagram of external imaging array of SWNT sensors.

For future applications the sensor could be uniquely engineered to meet imaging needs. Due to its high CMOS compatibility it could be readily integrated into existing
technologies [17]. Figure 36 shows an arrayed approach whereby encapsulated sensors are patterned to mimic pixilation common to today’s CMOS imaging. The small scale of the sensor makes it possible to be integrated into confined-space imaging such as that of internal medical practice. Figure 37 presents the properties of the PIN diode, a solid state detector, which the SWNT sensor would work to duplicate and surpass as a radiation detector. A design parameter which could result in SWNT sensors with greater resistance to radiation damage would be through the use of lower diameter nanotubes. It has been shown by Rossi et. al, that narrower tubes generally have less radiation induced defects [29]. This hardiness would limit the changes to electrical property the SWNT sensor would experience due to the changing geometry of the constituent tubes.

![PIN diode properties as a function of applied reverse voltage](image)

Carbon nanotubes have been shown to be promising as bio sensors [15]. For internal imaging applications, the sensor could be sent into the patient within a protective housing and then bombarded with an ionizing source. The sensors could then be used to image based on the migration of anions and cations across the device’s sensitive region.
REFERENCES


APPENDIX A: LABVIEW diagram
APPENDIX B: Sample MCNP6 input code for energy spectrum calculation

c Mini-X x-ray Spectrum Calculation

c generate x-rays with electrons converter

c   Electron Energy:  50 kV

c   Converter:

c     1.0 micrometer gold

c     127 micrometer Beryllium window

c Cell definitions (set imp for photons, electrons)

c-----------------------------------------------

11 1 -.193 -11  $ Au converter
21 2 -.1.85 -21  $ Be window
31 3 -.8.07 -31 41  $ Brass
41 0 -.41 21 11 56  $ Hollower
50 5 -.2.33 -50  $ Si dosimeter
56 0 -56  $ Spectrum Tallier
60 3 -.8.07 -60 70  $ Brass
70 4 -.0.001205 -70 31 80 50  $ Air surrounding
80 3 -.8.07 -80 35 31  $ Electronics housing
85 0 -85 31  $ Pre Chamber vacuum
98 0 -99 60  $ gap
99 0 99  $ void outer

c-----------------------------------------------

c Surface definitions (dimensions in cm)

c-----------------------------------------------

11 rcc 0 0 0.5151 0 0 .0001 .88  $ Au (1.0um)
21 rcc 0 0 0.5153 0 0 .0127 .88  $ Be (127um)
31 TRC 0 0 0 0 1.13 1.5 0.43  $ Brass
41 TRC 0 0 0 0 1.13 1.4 0.32  $ tunnel
56 RCC 0 0 1.10 0 0 .03 .32  $ Spectrum Tallier
50 sph 0 0 2.64 0.1  $ Si dosimeter
60 rpp -.39 39.0 39.0 -39.0 39.0 39.0  $ Brass Box
70 rpp -.38 38.0 38.0 -38.0 38.0 -38.0 38.0  $ Air in Brass Box
80 rpp -.2.9 2.9 -.1.27 1.27 -.14.3 .5  $ Electronics Box
85 rpp -.2.5 2.5 -.5 .5 -13.8 .3  $ Pre Chamber Vacuum
99 sph 0 0 0 60  $ limit boundary

c-----------------------------------------------

c Material Definitions

c-----------------------------------------------

m1 79197 1  $ gold
m2 4009 1  $ beryllium
m3 26000 0.001002 29000 0.674918 30000 0.320956 50000 0.001451 82000 0.001673
c m3 is brass
m4 6000 0.000150 7014 0.784431 8016 0.210748 18000 0.004671 $Air
m5 14000 1 $Silicon

---1---------2---------3---------4---------5---------6---------7---------8

c Mono-Energetic Electron source
c Mono-Directional (+z-dir)
c
sdef par=3 erg=0.050 pos 0 0 .5 axs 0 0 1 vec 0 0 1 dir=1 ext=0 rad=d1
SI1 0 0.008
SP1 -21 1
mode p e
imp:p,e 1 10r 0
c --------------------------------------------------------
c Number of particles to start
c --------------------------------------------------------
nps 1E10
c --------------------------------------------------------
c Tally definition
c --------------------------------------------------------
c --1---------2---------3---------4---------5---------6---------7---------8
+F506 50 $ Energy deposition tally
F564:p 56 $ Photon Energy Spectrum tally
e564 0.001 98i .05
F574:e 56 $ Electron Energy spectrum tally
e574 0.001 98i .05
E8 0 1E-5 1E-3 198i 0.2
F8:e 50 $ Pulse height tally
PRDMP 2J -1
print
APPENDIX C: Sample MCNP6 input code for dose factor calculations

MCNP6: Geometry

c
-----------------
c Cell definitions
-----------------
  2 2 -2.33 -2  $ 8 um Si
  98 4 -0.001205 -99 2  $ gaps
  99 0 99  $ void outside

-----------------
c Surface definitions
-----------------
  2 sph 0 0 0 0.05  $ 1 mm Si
  99 sph 0 0 0 10  $ limit boundary

-----------------
c Material Definitions
-----------------
  m2 14000 1  $ Si
  m4 6000 0.000150 7014 0.784431 8016 0.210748 18000 0.004671 $Air

-----------------
c Source Definition
-----------------
c Mini X

------------------------------------------
sdef par=2 erg=d3 x=d1 y=d2 z=-2 vec 0 0 1 dir=1
si1 -0.0003 0.0003
sp1 0 1
si2 -0.0003 0.0003
sp2 0 1
c

c Mini-X 50 kV Spectrum

c
--- Energies
si3 A 1.00E-03 2.00E-03 3.00E-03 4.00E-03 5.00E-03
  6.00E-03 7.00E-03 8.00E-03 9.00E-03 1.00E-02
  1.10E-02 1.20E-02 1.30E-02 1.40E-02 1.50E-02
  1.60E-02 1.70E-02 1.80E-02 1.90E-02 2.00E-02
  2.10E-02 2.20E-02 2.30E-02 2.40E-02 2.50E-02
  2.60E-02 2.70E-02 2.80E-02 2.90E-02 3.00E-02
  3.10E-02 3.20E-02 3.30E-02 3.40E-02 3.50E-02
  3.60E-02 3.70E-02 3.80E-02 3.90E-02 4.00E-02
  4.10E-02 4.20E-02 4.30E-02 4.40E-02 4.50E-02
  4.60E-02 4.70E-02 4.80E-02 4.90E-02 5.00E-02
c -- Prob. density (differential particle number Fluence)
sp3  0.00E+00  1.63E-06  1.74E-05  4.76E-05  8.35E-05
    1.05E-04  1.11E-04  1.10E-04  1.06E-04  1.01E-04
    1.25E-04  8.56E-05  6.88E-05  6.38E-05  5.65E-05
    5.37E-05  5.13E-05  4.81E-05  4.55E-05  4.39E-05
    4.10E-05  3.89E-05  3.81E-05  3.51E-05  3.36E-05
    3.12E-05  2.99E-05  2.74E-05  2.65E-05  2.42E-05
    2.28E-05  2.22E-05  1.97E-05  1.94E-05  1.96E-05
    1.73E-05  1.59E-05  1.47E-05  1.43E-05  1.29E-05
    1.15E-05  1.09E-05  9.55E-06  8.64E-06  7.24E-06
    6.35E-06  4.98E-06  4.00E-06  2.68E-06  1.18E-06

c  --------------------------------------------------
c set mode

c  ----------------------------------------------
mode p e

imp:p,e 1 1 1 0

c  ----------------------------------------------
c Number of particles to start

c  ----------------------------------------------
nps 1e7

c  -----------------------------------------------------------------------------
c
e8 0 1e-5 1e-3 198i 0.2
f8:e 2
c
+F606 2 $ Energy Deposition tally

print
APPENDIX D: Amptek Mini-X axial dose profile calculations

\[
Dose(z) = A \times \ln[1 + (\frac{R_o}{Z})^2]
\]

<table>
<thead>
<tr>
<th>Distance</th>
<th>MCNP6 (Rad (Si) /s)</th>
<th>Theoretical (Rad (Si) /s)</th>
<th>PIN Diode (Rad (Si) /s)</th>
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<tbody>
<tr>
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<td>66.50</td>
<td>66.50</td>
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</tr>
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</table>

Table 6. Dose Values from MCNP6, theoretical disc source, and PIN Diode measurements
<table>
<thead>
<tr>
<th>Distance (cm)</th>
<th>Roentgen/s</th>
<th>Roentgen Total</th>
<th>Sieverts Total</th>
</tr>
</thead>
<tbody>
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<tr>
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<td>4.86</td>
<td>582.77</td>
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<td>307.60</td>
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<td>193.00</td>
<td>1.80</td>
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<td>6</td>
<td>1.10</td>
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<td>30.96</td>
<td>0.29</td>
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<tr>
<td>Vacuum Housing</td>
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</tr>
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</table>

Table 7. Amptek Mini-X exposure and accumulated dose values as function of distance and testing situation