THIN FILM MATERIALS AND DEVICES FOR
RESISTIVE TEMPERATURE SENSING APPLICATIONS

A Dissertation in
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by
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ABSTRACT

Thin films of vanadium oxide (VO$_x$) and hydrogenated amorphous silicon (a-Si:H) are the two dominant material systems used in resistive infrared radiation detectors (microbolometers) for sensing long wave infrared (LWIR) wavelengths in the 8–14 μm range. Typical thin films of VO$_x$ (x < 2) currently used in the bolometer industry have a magnitude of temperature coefficient of resistance (TCR) between 2%/K – 3%/K. In contrast, thin films of hydrogenated germanium (SiGe:H) have |TCR| between 3%/K to 4%/K. Devices made from either of these materials have resulted in similar device performance with NETD ≈ 25 mK. The performance of the microbolometers is limited by the electronic noise, especially 1/f noise. Therefore, regardless of the choice of bolometer sensing material and read out circuitry, manufacturers are constantly striving to reduce 1/f noise while simultaneously increasing TCR to give better signal to noise ratios in their bolometers and ultimately, better image quality with more thermal information to the end user.

In this work, thin films of VO$_x$ and hydrogenated germanium (Ge:H), having TCR values > 4 %/K are investigated as potential candidates for higher sensitivity next generation of microbolometers. Thin films of VO$_x$ were deposited by Biased Target Ion Beam Deposition (BTIBD) (~85 nm thick). Electrical characterization of lateral resistor structures showed resistivity ranging from 10$^4$ Ω–cm to 2.1 × 10$^4$ Ω–cm, TCR varying from −4%/K to −5%/K, normalized Hooge parameter ($\alpha_H/n$) of 5 × 10$^{-21}$ to 5 × 10$^{-18}$ cm$^3$. Thin films of Ge:H were deposited by plasma enhanced chemical vapor deposition (PECVD) by incorporating an increasing amount of crystal fraction in the growing thin films. Thin films of Ge:H having a mixed phase, amorphous + nanocrystalline, having a |TCR| > 6 %/K were deposited with resistivity < 2,300 Ω–cm and a normalized Hooge’s parameter ‘$\alpha_H/n$’ < 2 × 10$^{-20}$ cm$^3$.

Higher TCR materials are desired, however, such materials have higher resistivity and therefore unacceptable large electrical resistance in a lateral resistor configuration. This work looks at an alternate bolometer device design which incorporates higher TCR materials in a vertically integrated configuration.
Thin films of high TCR hydrogenated germanium (Ge:H, $|\text{TCR}| > 6\%/K$) and vanadium oxide (VO$_x$, TCR > 5%/K) were integrated in lateral and through film configuration. The electrical performance of the vertically integrated devices is compared with lateral resistance structures. It was confirmed experimentally that the device impedance was significantly lowered while maintaining the signal to noise ratio of the lateral resistor configuration. The vertically integrated devices allow higher device currents without any increase in self heating. These structures may help reduce integration time and may result in higher frame rate.

Finally, one dimensional arrays were fabricated using both lateral and vertically integrated configurations and their performance was evaluated. It was found that the performance of the lateral devices was limited by noise floor of the measurement setup used. However, due to the lower impedance of the vertically integrated resistors, a higher signal and therefore higher signal to noise ratio could be obtained. These vertically integrated devices exhibited low RMS noise values of 12 mK.
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Chapter 1. Introduction and Goals

1.1. Introduction

Temperature sensing is the measure of the heat of a body. There are two types of thermal sensors, contact and non-contact sensors. The contact sensors touch the object being measured, while the noncontact sensors measure the temperature by measuring the infrared radiation emitted by that object. The second chapter provides background on temperature sensing and compares the different types of temperature sensors available in order to put in perspective the work done in this thesis.

Noncontact thermal imaging systems, also known as infrared imaging arrays, have seen a marked increase in the last decade and find applications in both military and civil applications. These systems consist of arrays of micromachined sensors, known as microbolometers, based on an electrical response from resistors, capacitors, etc. Resistive thin films of VOₓ and Si₁₋ₓGeₓ:H are the imaging materials that are the most dominant types of infrared imaging systems. The second chapter also provides an overview of the state-of-the-art resistive microbolometer technology based on VOₓ and Si₁₋ₓGeₓ:H thin films, and discusses important figures of merit and the read out circuits used in such devices. These discussions will help understand why detectors based on materials with vastly different electrical properties lead to similar detector sensitivity.

Chapter 3 provides an overview of experimental aspects of this thesis including the processing and characterization techniques used in this work. Relevant literature on the thin film deposition techniques is reviewed in this section. Details on thin film lithographic processing, electrical characterization, X-ray diffraction (XRD), atomic force microscopy (AFM), spectroscopic ellipsometry (SE), Rutherford backscatter spectroscopy (RBS) are also discussed in context with this chapter.
1.2. Statement of Problem

The performance of thin films of VOₓ and Si₁₋ₓGeₓ:H has remained relatively unchanged since the first literature appeared detailing their use. For years, much of the effort to improve the detector performance was focused towards improvement of the performance in the underlying read out circuits. However, as the need for higher density pixel arrays increases, the performance of these infrared systems is being limited by the signal to noise ratio of existing thin films. This motivated the study of high TCR materials having reasonably low electrical noise, which has been explored in Chapter 4 of this work. Thin films of VOₓ and Ge:H have been explored with a focus on depositing and characterizing thin films having |TCR| values > 3%/K.

Although numerous materials have a high TCR, the accompanying large resistance in a lateral pixel configuration makes integration with existing readout circuits difficult. Chapter 5 investigates an alternate pixel geometry so as to reduce the device resistance and facilitate integration of high TCR materials.

Finally, the high TCR thin films of VOₓ were exploited in temperature sensors arrays fabricated on glass substrates. Chapter 6 describes the fabrications of these thin film sensor arrays using both the lateral and the vertically integrated configuration. Two dimensional arrays were also fabricated and were multiplexed using integrated ZnO transistors fabricated on the underlying circuits. The resulting devices were characterized for their performance.
Chapter 2. Introduction to Temperature Sensing

Temperature is a measure of heat of an object. The concept of temperature invokes the physiological experience of touching an object and describing them as cold, cool, warm, hot, etc. Warmer objects transfer heat to colder objects till both their temperatures have reached equilibrium.

Temperature is an important physical parameter which influences all physiological, and thermal processes. Proper functioning of these processes requires accurate measurement of the corresponding temperature. There is a vast and an ever-growing number of methods for temperature sensing, however, the measurement techniques best suited for the application must be chosen in order to obtain readings which are as precise as required by that application.

Temperature sensors (or thermometers) can be classified based on heat transfer mechanisms as either contact or non-contact sensors.

2.1. Contact Temperature Sensors

The most accurate type of contact sensors are the electrical response contact sensors. In these types of sensors, the temperature response is converted into an electrical signal. Some commonly used electric contact sensors are discussed below:

2.1.1. Thermocouples

The thermocouple is based on the phenomenon known as the Seebeck effect. Named after the T. Seebeck who first observed this effect, he noted that there is a current flow in a closed loop of two dissimilar metals if their junctions are kept at two different temperatures. The corresponding voltage associated with this effect is known as the thermal electromotive force. A device which uses the Seebeck effect for the measurement of temperature is known as a thermocouple.

A thermocouple consists of two wires of dissimilar conductors (metals, alloys or semiconductors) connected at one end. The connected end is placed at the point of measurement, while the unconnected
end is the reference junction and can be used to readout the electromotive force developed using a voltmeter. Depending on the material used, these thermocouples can have an operation range of up to 3000 °C [1]. The most sensitive thermocouples are based on two alloys made from Nickel-chromium or Nickel-copper [1]. Thermocouples made from these alloys can achieve a sensitivity of 6.32 mV/100°C and have an operating range of 0°C to 1100°C [1]. The accuracy of such thermocouples is ≈ ±1 °C [1].

2.1.2. Resistance Thermometer Detectors (RTD)

Resistance thermometers consist of thin wires of a metal or an alloy whose resistance changes linearly as a function of temperature. Platinum, Nickel, Copper and alloys of Rhodium-Iron are some commonly used materials in resistance thermometers [1] [2]. Nickel has the largest temperature response that of 0.62 %/K while Platinum has the highest range of operation between −260 K to 1100 K [1]. Due to the low resistivity of these materials, they are usually thin, long wires wound into a small form factor. The typical accuracy of measurement of RTDs in this form factor is ≈ ±0.2 °C. Typical limitations include a relatively large form factor, cost associated with platinum wires and a high response time associated with the large thermal mass of the wires. Thin films have also been fabricated using these materials and thus have the advantage of miniaturization and the ability to fabricate temperature sensing arrays.

2.1.3. Semiconductor Thermometers

Semiconductor thermometers are made from semiconductor materials and can be divided into two main groups: bulk effect and junction effect sensors. In these types of temperature sensors, the conductivity of the semiconductor material changes due to a change in temperature of that material.

2.1.3.1. Thermistors

In these types of materials the resistance of the material changes as a function of temperature. This change in resistance can be readout electrically to give the change in temperature of the body under investigation. The resistance change of a thermistor is non-linear and follows a thermally activated behavior described by the Arrhenius equations such that:
\[ R_T = R_0 e^{-\frac{E_a}{k_B T^2}} \]  

Equation 1

Where \( R_T \) is the temperature of the thermistor at a temperature \( T \). \( R_0 \) is the temperature at room temperature, \( k_B \) is the Boltzmann’s constant, and \( E_a \) is the activation energy which is a material property of the thermistor.

The temperature response or the temperature coefficient of resistance (TCR) of the thermistor can then be estimated as:

\[ TCR \ (in \ \%/K) = \frac{-E_a}{k_B T^2} \times 100 \]  

Equation 2

Thermistors are typically made using ceramic manufacturing techniques from oxides of Mn, Ni, Co and Cu; they are pressed at high pressures and sintered at temperatures up to 1000 °C [1] [3]. Typical TCR values of thermistor lie between –2 %/K to –6 %/K [1]. This temperature response makes these thermistors some of the most sensitive types of temperature detectors with temperature errors as low as ± 0.1 °C [1] [4]. Limitations of thermistors include a lower and a smaller temperature range of operation (typically between -100 °C and 200 °C) and the non-linearity of the electrical response [5].

### 2.1.3.2. Diode and Transistor Based Thermometers

The output characteristics of a junction are dependent on its temperature of operation such that:

\[ I_d (T) = I_s \left( \frac{V_p}{e^{\frac{V_T}{nV_T}} - 1} \right) \]  

Equation 3

Where \( I_d \) is the forward current of the diode, \( I_s \) is the reverse saturation current, \( V_p \) is the forward bias voltage, \( n \) is the ideality factor and \( V_T \) is the thermal voltage associated with temperature \( T \) (26 mV at 300 K). The change in \( V_T \) associated with the change in temperature can be used to calculate the temperature of the object with respect to a reference. Commercial temperature sensors based on junction effect are available and have a sensitivity of –2 mV/°C for silicon based diodes and –1.25 mV/°C for germanium
based diodes [1]. In addition, sensitivity as high as 40 mV/°C has been demonstrated using ZnO based transistors [6].

2.2. Non-contact Thermometers or Pyrometers

Pyrometers or infrared detectors are thermometers which measure the temperature of the object based on the emitted thermal radiation of that object. Pyrometers are commonly used in applications such as infrared imaging [7] [8] [9], temperature sensors for biological applications [10] [6], aerodynamics and space exploration [11], monitoring industrial processes, etc.

According to the Stefan-Boltzmann’s law, the total radiant intensity $W_0$ of a black body depends on its temperature $T$ [1]:

$$W_0 = \sigma_0 T^4$$  \hspace{1cm} \text{Equation 4}

$\sigma_0$ is the Stefan-Boltzmann’s constant and has a value of $5.67 \times 10^{-8}$ W/m$^2$K$^4$

The spectral radiant flux distribution is given by [1]:

$$W_{0\lambda} = \frac{c_1 \lambda^{-5}}{\sigma c_2 / \lambda^4 - 1}$$  \hspace{1cm} \text{Equation 5}

where $W_{0\lambda}$ is the spectral radiant flux distribution of a black body in W/m$^2$μm, $T$ is the temperature of the black body in K, $c_1 = 3.7415 \times 10^{-16}$ W.m$^2$ and $c_2 = 14,388$ μm.K.

There are numerous detectors that can be used for detection of thermal radiation. Some common detector types are discussed in detail in the following subsections.

2.2.1. Thermopiles

A thermopile is a series connection of numerous thermocouples whose hot junction is in contact with a black body which absorbs the incident radiation. Since a single thermocouple produces a small
electromotive force, a series of such thermocouples can produce thermometers with high sensitivity. Such devices have demonstrated sensitivity of several hundred μV/K. Detailed descriptions of state-of-the-art thermopiles can be found elsewhere in refs. [3] [12].

2.2.2. Pyroelectric Detectors

Pyroelectric detectors are based on the principle that the dipole moments in a pyroelectric material change their orientation as a function of temperature. Materials such as lead zirconium titanate (PZT), triglycine sulphate (TGS) and barium strontium titanate (BST) have a large pyroelectric coefficient response $> 2 \times 10^{-8} \text{C} \cdot \text{cm}^{-2} \cdot \text{K}^{-1}$ [1] [3]. Thin film temperature sensing arrays have been fabricated using pyroelectric materials, the details of which can be found in ref. [3] [13] [14].

2.2.3. Photoconductive Detectors

Photoconductive detectors are made from materials whose electrical conductivity changes due to the generation of carriers in the material as a function of incident radiation. Depending on the choice of material, the detector can have a different spectral response. For example, CdS has a spectral response in the visible spectral region while PbS is in the 0.5 μm–4 μm wavelength range.

2.2.4. Photovoltaic Cells

A photovoltaic device generates a voltage depending on the incident infrared radiation. The photovoltaic material absorbs radiation, exciting valence band electrons into its conduction band. Similar to other photoelectric detectors, the spectral response depends on the choice of material. InAs has a spectral response between 1 μm–3.8 μm while InSb has a maximum sensitivity to radiation of wavelength 8 μm.

2.2.5. Thermistor or Metal Bolometers

Bolometers based on thermistors or metal bolometer arrays are thermometers based on the temperature coefficient of resistance of the thermistors or metals. However, in these non-contacting detectors, the heating of the resistor takes place by absorption of the infrared radiation by the material or by an absorber layer for non-absorbing thermistor materials [3]. These types of detectors are by far the
most popular for imaging in the 8 \( \mu \text{m} \)–14 \( \mu \text{m} \) range [15]. Bolometers, especially microbolometers are the focus of this work and are discussed in greater detail in the subsequent sections.

Figure 2-1. Calculated spectral radiant emissive power of a black body (in \( \text{W/m}^2 \cdot \mu\text{m} \)) at different temperatures. The maximum emissive power for objects \( \approx 300 \text{ K} \) lies between 8 and 14 \( \mu \text{m} \).

Figure 2-1 shows the spectral emissive power of a black body in accordance with Equation 5 at different temperatures of the object. The choice of infrared detector depends on the desired temperature range of measurement. One of the biggest applications for temperature sensing is room temperature imaging of objects at temperatures between \(-40\ ^\circ\text{C}\) and \(120\ ^\circ\text{C}\). Today, the most common type of detector for room temperature infrared imaging is based on thermistor materials whose resistance changes as it heats up due to incident infrared. This imaging technique is described in the subsequent subsections in further detail.
2.3. Resistive Infrared Imaging or Microbolometer

A bolometer is a temperature-sensitive electrical resistor whose resistance changes due to an increase in temperature caused by incident infrared radiation. The magnitude of the resistance change is proportional to the temperature of the black body emitting the incident radiation. Two dimensional arrays of miniaturized resistors (microbolometer) can be used for mapping the thermal field of the scene under investigation.

![Graph of electromagnetic spectrum and atmospheric opacity](image)

*Figure 2-2. The electromagnetic spectrum and the opacity of the atmosphere at different wavelengths, from ref. [16].*

Since a microbolometer is a non-contact temperature sensor, the infrared radiation must travel through the atmosphere before it reaches the detector element. H₂O and CO₂ molecules present in the atmosphere absorb certain wavelengths of radiation, as can be seen in Figure 2-2 [16]. Radiation in the 3–5 μm range (Mid-Wavelength IR or MWIR) and 8–14 μm range (Long Wavelength IR or LWIR) is transmitted through the atmosphere and can be used for detecting infrared radiation. Due to the higher intensity of radiation in the 8–14 μm band at 300K (Figure 2-1), LWIR detectors are preferred. However, the choice of detector depends on the specific application at hand.
The actual sensor used for infrared imaging is a monolithic structure prepared by silicon micromachining, originally developed by Honeywell [8]. A simplified schematic of such a pixel structure is shown in Figure 2-3. The detecting area is a thin film of the temperature sensitive material employed (metal or semiconductor) having a temperature coefficient of resistance (TCR) of $\alpha$, atop a thin free standing membrane, usually Si$_3$N$_4$ [17] [3]. The membrane is supported by legs which have a low thermal conductance to maximize thermal isolation from the environment. The legs also have a thin film of metal which serve as electrical contacts to the resistive thin film. In addition, the underlying substrate has a reflective thin film which causes the unabsorbed radiation to reflect back to the resistive layer [17] [3]. It was found that the reflection is at a maximum when the spacing between the reflective layer and the suspended resistor is one quarter of the wavelength being sensed [3] [17].

### 2.3.1. Figures of Merit

The performance of a microbolometer depends on two important properties which are discussed in further detail in the following subsections.

#### 2.3.1.1. Temperature Coefficient of Resistance (TCR)
The performance of a microbolometer depends on several factors, one of which is the temperature coefficient of resistance or TCR. The temperature coefficient of resistance is defined as the percentage change in resistivity of the material per degree Kelvin of increase in its temperature. TCR can be negative or positive. While metals have a positive TCR, semiconductors have a TCR which is typically negative. In general, TCR can be defined using Equation 6:

\[
TCR (\alpha) = \frac{1}{\rho} \frac{d\rho}{dT}
\]  

Equation 6

where \( \alpha \) is the TCR of the material, \( \rho \) is the resistivity and \( d\rho \) is the change in resistivity of the material associated with a change in its temperature \( dT \).

The resistivity of a material is given by Equation 7:

\[
\rho = \frac{1}{ne\mu}
\]  

Equation 7

where \( n \) is the free carrier concentration, \( \mu \) is the mobility of carriers and \( e \) is the charge of an electron.

There are two factors which may contribute to a change in the resistivity of the material with temperature as observed in Equation 7, namely, the free carrier concentration (\( n \)) and the mobility (\( \mu \)). Metals show little change in the free carrier concentration as a function of temperature. However, the mobility of the charge carriers decreases with increasing temperature, resulting in a positive TCR, which is constant as a function of temperature. On the other hand, in the case of intrinsic semiconductors, both the free carrier concentration and mobility of the material changes with temperature, this is usually results in a TCR which is negative. The resistivity of semiconductors changes as a result of thermally activated charge carriers across the band gap such that:

\[
\rho = \rho_0 \exp \left( \frac{\Delta E_a}{kT^2} \right)
\]  

Equation 8

where, \( \Delta E_a \) is the activation energy, \( k \) is the Boltzmann’s constant. Thus it follows that for a semiconductor, the TCR is given by:
Since all the work discussed in this thesis is based on uncooled infrared detection, $T$ is taken to be room temperature (300 K) when reporting the TCR of a material.

Given this relationship, it is easy to calculate the theoretical TCR which can be achieved by using some common semiconducting materials such as silicon and germanium. For single c-silicon, the activation energy is roughly half of the band gap ($E_g/2$) and is equal to 0.55 eV, which results in a $|TCR|$ of 7.2%/K. However, in the case of single c-germanium, whose activation energy is 0.335 eV, a $|TCR|$ of 4.3%/K can be achieved. Although, higher TCR materials are desired, increasing activation energy (and therefore increasing bandgap) results in a decrease in the free carrier concentration; this results in high material resistivity (from Equation 7). Undoped crystalline silicon has a resistivity of $3.2 \times 10^5 \Omega$-cm, whereas crystalline germanium has a resistivity of 50 Ω-cm. Unfortunately high resistivity materials tend to have higher noise [3] [18] [19] [20] [21] [22] [23] [24] [25] [26] [27], which is another figure of merit for a detector, as we will see in the subsequent sections. In addition, single crystal materials such as c-Ge are hard to integrate with standard MEMS fabrication protocol.

2.3.1.2. Electrical Noise

Device performance of microbolometers does not solely depend on TCR of the thin film. For example, although thin films of SiGe:H have $|TCR| > 5\%/K$, they inherently have higher resistivity and higher electrical noise. Electrical noise is an important limiting factor in the sensitivity of a semiconductor-based temperature sensor. An important figure of merit that relates thin film properties directly to the device performance is the Noise Equivalent Temperature Difference or NETD. In terms of the sensing material properties, the NETD can be derived [17]:

$$NETD \propto \frac{I_{\text{noise, total}}}{I_{\text{bias}} \times TCR}$$  \hspace{2cm} \text{Equation 10}
where $I_N$ is the noise current due to the thin film, $I_{bias}$ is the bias current due to the applied voltage bias

$$I_{bias} = \frac{V_{bias}}{R_b}$$

and TCR is the temperature coefficient of resistance of the sensing material.

The ratio $\frac{I_{bias}}{I_N}$ is also known as the signal to noise ratio and is given by the ratio:

$$SNR = \frac{I_{bias}}{\sqrt{I_{noise, total}^2}}$$  \hspace{1cm} \text{Equation 11}

where $I_{noise, total}$ is the total noise current due to the detector material and is comprised of mainly two components, the Johnson noise ($I_{noise, J}$) and the flicker noise or $1/f$ noise ($I_{noise, 1/f}$).

**Johnson noise**

Johnson noise is the electrical noise generated as a result of thermal agitation of carriers and is related to the resistance and the temperature of that resistor. Depending on the bias conditions, Johnson noise can manifest itself as either current noise or voltage noise. The current noise is given by [17]:

$$I_{noise, J}^2 = \frac{4k_bT(f_2 - f_1)}{R_d}$$  \hspace{1cm} \text{Equation 12}

where, $k_b$ is the Boltzmann’s constant, $T$ is the temperature of the bolometer, $R_d$ is the resistance of the device, while the bandwidth of measurement extends from $f_1$ to $f_2$. The voltage Johnson noise is given by [17]:

$$V_{noise, J}^2 = 4k_bTR_d(f_2 - f_1)$$  \hspace{1cm} \text{Equation 13}

From the above two equations it can be seen that the Johnson noise is independent of the frequency but depends on the temperature and the resistance of the device as well as the bandwidth of measurement.

Equation 12 describes the current Johnson noise contribution to the detector signal. Due to the inverse relationship between the current Johnson noise and the detector resistance, the Johnson noise decreases with increasing device resistance. This inverse relationship allows for small Johnson noise contributions in constant bias bolometers even though high resistance thin films are used [18]. It is for this reason that
most bolometer designs use a voltage bias with current as the output signal. Further discussions on electrical noise will focus on current noise.

**Flicker noise or 1/f noise**

1/f noise is the fluctuation in conductance with a power spectral density proportional to $f^{-\gamma}$ where $\gamma = 1 \pm 0.1$ [28]. The current 1/f noise is given by empirical relationships proposed by F. N. Hooge [28]:

$$I_{noise, 1/f}^2 = I_{bias}^2 \frac{\alpha_H}{nVf}$$

Equation 14

Where $I_{bias}$ is the bias current as a result of the applied voltage, $V$ is the volume of the sample; $f$ is the frequency range of measurement; $\alpha_H$ is Hooge’s parameter; and $n$ is the carrier concentration. For a bandwidth of measurement between $f_1$ and $f_2$, the corresponding 1/f noise becomes [3]:

$$I_{noise, 1/f}^2 = I_{bias}^2 \frac{\alpha_H}{nV} \int_{f_1}^{f_2} \frac{1}{f} df = I_{bias}^2 \frac{\alpha_H}{nV} \ln \left( \frac{f_2}{f_1} \right)$$

Equation 15

The two components, Johnson noise and 1/f noise, combine to produce a total mean square microbolometer current noise given by:

$$I_{noise, total}^2 = I_{noise, f}^2 + I_{noise, 1/f}^2$$

Equation 16

Figure 2-4 shows an equivalent circuit diagram for a resistor which consists of an ideal resistance of value $R_B$ and a current noise source in parallel with the ideal resistor.
2.3.2. Choice of Resistive Material

Figure 2-5 shows a plot of resistivity versus TCR of selected temperature sensitive materials which have been explored for use in infrared imaging [3] [18] [19] [20] [21] [22] [23] [24] [25] [26] [27]. These materials are metals, their alloys, semiconducting materials, and thermistor materials such as spinels of manganese, cobalt and nickel oxides.
2.3.2.1. Metals

In the low resistivity range of Figure 2-5, metals and alloys such as titanium (0.43%/K), platinum (0.18%/K) and Ni-Fe alloys (0.23 %/K) dominate [17]. Although metals and their alloys have low TCR values, their resistivity and therefore the inherent noise is extremely low, making them potential candidates for infrared imaging. In addition, processes exist for controllable, repeatable deposition of metal thin films. In fact, some of the first microbolometers were made using sputter deposited metals on Si₃N₄ membranes [3]. Because the resistivity of metals is low, very long serpentine resistors were fabricated to form the bolometer resistors [17]. Numerous metals and their alloys were explored as potential candidates; however in the early 1990s, semiconductor materials having five to ten times larger TCR replaced metals as the choice for bolometer materials.
2.3.2.2. Vanadium Oxide (VO\textsubscript{x}) Thin Films

Most of the vanadium oxide microbolometer work was classified research and was kept secret until 1991, when Wood et al. at Honeywell Inc. filed a patent on the development of thin films of mixed phase vanadium oxides (VO\textsubscript{x}) which had a TCR value of up to five to ten times higher than those of metals [8]. However, because of the United States International Traffic in Arms Regulations (ITAR) restrictions [33], the first public disclosure of the work, including details of materials and bolometer design was not published until 1994 by Wood et al. [8]. The performance of VO\textsubscript{x} thin films achieved using the patented method was sufficient and surpassed other candidates for infrared imaging. The resistivity of these films was moderately higher than those of metals, at around 0.01 – 1Ω·cm [8]. Thin films could be repeatedly deposited by ion beam sputtering at room temperatures on to commonly used substrates such as Si\textsubscript{3}N\textsubscript{4} coated Si wafers [8] [30]. For these reasons, VO\textsubscript{x} thin films have dominated the microbolometer industry in the 1990s to early 2000s [30]. All VO\textsubscript{x} thin films were made either at Honeywell or were made using technology licensed from Honeywell [31] [32]. All literature, including the Honeywell patent, gave little to no description of the microstructure of the thin films obtained [8].

By the early 2000s, the underlying read out circuit technology had matured and the detector sensitivity had reached limits imposed by the electrical noise of VO\textsubscript{x} thin films. To facilitate the development of VO\textsubscript{x} thin films, the Pennsylvania State University was awarded a grant sponsored by the Army Research Office. One of the goals of the research initiative was to develop an understanding of the relationship between the electrical and microstructural properties of the VO\textsubscript{x} thin films. In addition, alternate deposition methods were also explored to improve the throughput limitation imposed by ion beam deposition.

VO\textsubscript{x} thin films can be deposited by various sputtering methods including ion-beam sputtering [3] [33] [34], RF sputtering [35], direct current (DC) [36] and pulsed DC sputtering [37] [38] [39]. However, films deposited by ion-beam deposition exhibited better film properties, i.e. higher TCR and lower 1/f noise for comparable resistivity values with other deposition techniques [23]. Most references to
industrially manufactured vanadium oxide thin-films for use in infrared imaging, to date, call for reactive ion-beam sputtering as the primary method of deposition [3] [8] [33] [34] .

Some of the first published work on developing an understanding between the structure–property relationships for VO_x thin films was done by Kerry Wells at the Pennsylvania State University by pulsed DC magnetron deposition of thin films of VO_x using a V_2O_3 target. Thin films deposited using this method resulted in films which were voided and mostly amorphous with some nano crystalline grain growth for thicker films [40]. It was also shown that different processing parameters such as, partial pressure of oxygen during sputtering and total sputtering pressure, produced films with different microstructure and electrical properties. In general, the resistivity and TCR of films was found to increase with an increase in the oxygen partial pressure, as well as with an increase in the total sputtering pressure. TCR values between −1 %/K and −3%/K were achieved; however the resistivity was orders of magnitude higher than those of films deposited by ion beam deposition [40].

Dr. Chandrasekaran Venkatasubramaniam investigated the use of metallic vanadium target for depositing microbolometer grade vanadium oxide thin films. Dr. Venkatasubramaniam characterized the inherent process hysteresis associated with the reactive pulsed Direct Current (DC) magnetron sputtering of the metallic vanadium target. Thus, thin films with resistivity and TCR values comparable to those of films deposited by ion beam deposition were grown [20] [41]. It was found that the room temperature resistivity of films increased with increase in total flow rate as well as with the increase in the oxygen partial pressure during sputtering [20] [42].

Films with good electrical characteristics (high TCR with moderately low resistivity) were deposited at low total flow rates and low oxygen partial pressures [20] [42]. These films showed the presence of nano-crystallites in an amorphous matrix in films, with high levels of porosity, which was confirmed by both transmission electron microscopy (TEM) and grazing incidence x-ray diffraction methods (GIXRD) [20].
Post deposition annealing experiments were also performed in oxygen and nitrogen environments in an attempt to improve the trade-off between TCR and resistivity. Films annealed in oxygen were driven towards higher oxides of vanadium, which have unacceptably high resistivity at room temperature. Annealing the thin films in nitrogen ambient yielded better results; for example, for films annealed at 300° C in nitrogen, the resistivity dropped by an order of magnitude, without significant deterioration of the film TCR. Structural analysis by XRD, Rutherford backscattering spectroscopy (RBS), Raman spectroscopy, scanning electron microscopy (SEM), TEM, and atomic force microscopy (AFM) did not reveal any significant changes between the annealed and the un-annealed films [20]. It was speculated that the improvement in thin film characteristics was due to reordering between the amorphous and nanocrystallite phases or due to grain growth. Nick Fieldhouse investigated the effects of substrate heating during reactive deposition of vanadium oxide thin films using a metallic vanadium target. The thin films obtained were mixed phase with VO and V$_2$O$_3$ crystallites in an amorphous matrix with moderate improvement in the film’s electrical properties [42].

B. D. Gauntt extensively studied the microstructure-property relationships by altering processing parameters during pulsed DC magnetron deposition of VO$_x$ thin films. It was found that all thin films deposited either by ion beam deposition or by pulsed DC sputtering resulted in one of three microstructures: an amorphous often voided columnar structure, a polycrystalline structure with nanometer-sized grains, or a nano-composite structure composed of columnar or conical nanocrystallites of vanadium oxide in an amorphous matrix. Gauntt concluded that thin films of VO$_x$ exhibiting the best electrical properties were found to contain conical/columnar grains in an amorphous matrix and were deposited by ion beam deposition [43].

H. A. Basantani attributed the superior electrical properties of thin films deposited by ion beam deposition to the energetics associated with the process. To replicate the results with pulsed DC sputtering, substrate bias was applied during deposition. The electrical properties of the thin films deposited were superior (higher TCR with lower electrical noise) to those obtained with ion beam
deposition [37]. The films obtained were mixed phase with V$_2$O$_3$ nanocrystallites in an amorphous matrix. The x value in VO$_x$ was between 1.5–2 [37]. In general, the microstructure of the thin films obtained was similar to the ones obtained with ion beam deposition; however these films had a higher density (≥ 4.2 gm/cm$^3$ versus 3.8 gm/cm$^3$ for ion beam deposited thin films.

The best films of that work had a TCR of −2.6%/K with a resistivity of 0.6 Ω–cm [37]. Attempts to increase the TCR further resulted in an abrupt increase in the resistivity which was not of interest for that work [44]. Venkatasubramanium and Gauntt explored the parameter space during deposition by pulsed DC magnetron sputtering and obtained high TCR VO$_x$ thin films having very large resistivities [20] [21]. Figure 2-6 compares the electrical properties of VO$_x$ thin films deposited by pulsed DC magnetron sputtering and ion beam deposition from numerous references [3] [33] [42] [37]. It is interesting to note that although the obtainable range of TCR is between −0.2%/K and −5%/K, there are two ranges of resistivity between $10^3$ to 1 Ω–cm and $10^3$ to $10^5$ Ω–cm. At the time of writing this dissertation, thin films having intermediate resistivity between 1 Ω–cm and $10^3$ Ω–cm have not been successfully deposited.
There are fundamental differences between VO<sub>x</sub> thin films belonging to the two distinct resistivity ranges. While thin films having resistivity between $10^{-3}$ $\Omega$–cm and 1 $\Omega$–cm are mixed phase, films with higher resistivity and high TCR are x-ray amorphous [21] [37]. The high resistivity films typically have x-values in VO<sub>x</sub> between 2 – 2.5, compared with 0.8 – 2 for films with resistivity < 1 $\Omega$–cm [45]. The resistivity and the inherent 1/f noise of thin films with $|\text{TCR}| > 4\%/K$ was found to be too high for use in existing read out circuit technology at the time [3].

Both deposition techniques, ion beam deposition and magnetron sputtering, are limited in deposition rates to $\sim 1\,\text{Å/s}$ [44] [46]. Deposition rates in ion beam deposition is limited by ion current density of the ion source, whereas in magnetron sputtering the deposition rate is limited by the process...
window associated with reactive deposition and therefore the throw distance associated with the reactive deposition [42].

In order to increase throughput, industry is looking for alternate deposition techniques that would result in higher deposition rates. One such candidate is the biased target ion beam deposition which is capable of achieving deposition rates > 10Å/s. VOₓ thin films deposited by this deposition technique were investigated in this work [47].

2.3.2.3. Hydrogenated Silicon Germanium Alloys (Si₁ₓGeₓ:H)

A considerable research effort has been made in the development of thin films of amorphous hydrogenated silicon (a-Si:H) and hydrogenated silicon germanium alloys (Si₁ₓGeₓ:H) for use in large area devices such as solar cells, liquid crystal displays and most recently as sensing material for infrared detection [48] [49] [50]. In particular, alloys of Si and Ge have been heavily investigated because the optical band gap of the deposited material can be tailored between 1.1 eV and 1.7 eV depending on the composition of the alloy. However, the success in developing SiGe alloys for microbolometer applications having superior electrical properties has been rather limited.

Although VOₓ thin films have been demonstrated to be useful microbolometer TCR materials, their maximum TCR is limited to about −5%/K. Mark Unewisse and Jean Tissot in 1996 demonstrated amorphous hydrogenated silicon–germanium (SiGe:H) thin films having TCR values larger than those of VOₓ, around −2.5%/K to −8%/K [50] [50]. Although TCR values as high as −8%/K were attainable, Liddiard et al. concluded that the electrical properties were accompanied by unacceptably high 1/f noise [17].

An analogous material system lying on the other extreme of the spectrum, consisting of amorphous hydrogenated germanium (a-Ge:H), has also been investigated [51] [52] [53] [54] [55]. However, these thin films could not compete with the performance of Si:H/SiGe:H systems for photovoltaic or thin film transistor applications. While Si:H and SiGe:H have dark resistivity between
$10^{11} - 10^{13}$ Ω–cm with light resistivity as low as $10^4$ Ω–cm, Ge:H thin films show smaller dark resistivity of $10^5$ Ω–cm with smaller thermal activation [49] [52] [53] [54] [55]. However, the lower resistivities of Ge:H thin films make them good candidates for further investigation as infrared imaging materials.

Electrical properties of hydrogenated germanium have been investigates for over three decades. Numerous research groups have demonstrated thin films of amorphous Ge:H having |TCR| as large as 5%/K and having resistivity of the order of $10^5$ Ω–cm [51] [52] [53] [54] [55] 4. Campinas et al. studied the effect of deposition conditions on the electronic quality of a-Ge:H and a-Ge thin films deposited by RF sputtering [53]. Plasma enhanced chemical vapor deposition (PECVD) technique which had proved to be very effective in producing a-Si:H thin films, was also used to deposit Ge:H. The highest |TCR| obtained was 4.6 %/K with a resistivity of $10^5$ Ω–cm [55].

Ambrosio et al. demonstrated thin films of a-Ge:H and a-SiGe:H, deposited using GeF₄ diluted in H₂ having |TCR| as high as 5%/K with moderately low resistivity of 20,000 Ω–cm [56]. These publications contained no information on the underlying microstructure or the inherent 1/f noise properties of the films obtained. Further improvements on the electrical properties came in 2012, when Moreno et al. published information on polymorphous thin films of Ge:H [57].

In the work by Moreno et al., growth of nanocrystallites was encouraged by varying the total pressure of deposition from 600 mTorr to 2500 mTorr [57]. Cross sectional TEM of the thin films revealed that the films contained nanocrystals of size 2–4 nm in an amorphous matrix [57]. As a function of pressure, Moreno claimed that the grain size grew and its value reached a maximum at around 2,000 Torr before decreasing for 2.5 Torr [57].
Figure 2-7. Graph showing the effect of total deposition pressure on TCR and resistivity of deposited pm-Ge:H thin films from ref [57].

Figure 2-7 shows the resistivity and TCR data of deposited thin films as a function of deposition pressure from reference [57]. Thin films deposited in this work showed |TCR| between 3.8 %/K and 5 %/K with resistivity between 1,100 Ω–cm and 20,000 Ω–cm [57]. This resistivity – TCR tradeoff was amongst the best reported in the literature for thin films of hydrogenated germanium [57]. No 1/f noise properties were reported for these thin films.

Around the same time Saint-John et al. also investigated if the electrical properties of the Ge:H thin films can be improved by introducing small amounts of nanocrystalline Ge:H material during the natural growth evolution [51] [19]. It was hypothesized that addition of nanocrystalline germanium, which has a resistivity of 50 Ω–cm, would help reduce the resistivity and the electronic noise while keeping the high TCR associated with the amorphous Ge:H material [57].

The dilution ratio (given by \( R = [H_2]/([GeH_4] + [H_2]) \)) used during PECVD is an important parameter in the nucleation of crystallites [19] [51]. Similar to PECVD deposition of Si:H and SiGe:H
thin films, thin films of Ge:H also show a decrease in the thickness of amorphous bulk layer prior to the nucleation of crystallites [51] [58]. Figure 2-8 shows the skeletal growth evolution diagram which was developed by Saint-John for Ge:H, similar to the microstructural evolution associated with PECVD deposited Si:H, to confirm the location of the transition regions between amorphous, amorphous and nanocrystalline, and nanocrystalline microstructures [19].

![Growth evolution diagram for Ge:H thin films grown on native oxide on c-Si showing the thickness values at which crystallization becomes evident (re-plotted from ref [19]).](image)

By depositing thin films at different dilution ratios of the growth evolution diagram, Saint-John’s work aimed to assess the electrical property of the thin films as a function of microstructure. Figure 2-9 summarizes the electrical properties of thin films obtained in that work [19].
Figure 2-9. Summary of electrical properties (resistivity, TCR and normalized Hooge’s parameter) for thin films of Ge:H thin films deposited at different dilution ratios [19].

TCR values between −2 %/K and −5 %/K were obtained with resistivity between $10^3$ Ω–cm and $10^4$ Ω–cm and $\alpha_{H}/n$ values between $10^{-20}$ and $10^{-17}$ cm$^3$ [19]. However, no obvious trend in the electrical properties could be inferred as a function of $R$. Part of the reason could be that there are several factors involved. For example, the plot compares electrical properties of films having different thicknesses and therefore different microstructures. Thus it is hard to get a true sense for a relationship between the microstructure and electrical properties.

Figure 2-10 shows the electrical properties of thin films as a function of crystal fraction obtained by growing thin films of increasing thicknesses at a dilution ratio of $R = 225$. Saint-John noticed a decrease in the resistivity for small crystal fractions following which both the resistivity and TCR increased while $\alpha_{H}/n$ values remained around $10^{-18}$ cm$^3$ [19]. The largest TCR value of −3.5 %/K was obtained for the thin film with a crystal fraction of 55 % for a film thickness of 200 nm [19]. Thin films
with larger crystal fractions were not deposited in this work, and so it is unclear if larger TCR values could be obtained for films with higher crystal fractions.

Saint John noted that to obtain thin films with a larger crystal fraction for a dilution of $R = 225$, thin films must be thicker than 200 nm [19]. However, for bolometric applications, these thicker films have an unacceptably high thermal mass, thereby limiting the bolometer’s performance.

To induce grain growth at small thicknesses, Saint-John observed the microstructural evolution in thin films deposited at a much higher dilution of $R = 400$. Figure 2-11 shows the microstructure of a 200 nm thick thin film deposited on to native oxide on c-Si [19]. Saint-John observed that thin films deposited at this dilution were dense and the microstructure was nanocrystalline with planar twinning defects on the order of $\sim 1$nm [19]. Despite the unique microstructure, the films were not characterized for their electrical properties because they were not deposited on insulating substrates.
Saint John also observed the surface roughness evolution of thin films deposited at various dilutions and related it to the microstructural evolution of a growing thin film. He observed that for thin films deposited at higher dilutions, the surface roughness increased rapidly and at lower thicknesses, indicating a faster transition from an amorphous to an amorphous + nanocrystalline material [19]. He also observed that the roughness peaked at smaller thicknesses and then decreased. The peak in roughness was attributed the value of film thickness at which the grains coalesced and the film became predominantly nanocrystalline [19].

Saint John also observed that further acceleration of the microstructural evolution is made possible by lowering the substrate temperature used during deposition [19]. Figure 2-12 shows the surface roughness evolution of Ge:H thin films deposited at substrate temperatures of 140 °C, 200 °C and 260 °C [19]. The thin film deposited at 140 °C showed a prompt coalescence at a thickness of 85 nm [19].
Figure 2-12. Surface roughness evolution for Ge:H films deposited at different dilution ratios, \( R \). Higher dilution ratios show an increase in surface roughness at smaller thicknesses, indicating film transformation from amorphous to a mixed phase thin film ref. [19].

Figure 2-13. Surface roughness evolution for films deposited at increasing substrate temperatures on to SiN\(_x\) substrates on c-Si. Lower substrate temperatures result in a quicker decrease in roughness, indicating a more prompt coalescence [19].
2.3.3. Comparison of Material Performance

The total noise due to the sensing thin film depends on the bias current, but can be calculated based on values known from the industry [18] [19]. Due to their lower resistance, thin films of VO$_x$ are pulsed biased with a current of $\sim 10 \, \mu$A. SiGe:H thin films, which have a much higher resistance in comparison, are biased using a constant current of $\sim 40 \, n$A. The noise contribution of the two material systems can then be calculated from the above equations and have been summarized in Table I below.

Table 1 lists relevant thin film parameters and the calculated noise current in VO$_x$ and SiGe:H thin films.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value (VO$_x$ Thin Film)</th>
<th>Value (SiGe:H Thin Film)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistance ($T_{room}$)</td>
<td>100 K$\Omega$</td>
<td>$&gt; 100 , M\Omega$</td>
</tr>
<tr>
<td>Bias Current ($I_{bias}$)</td>
<td>10 $\mu$A</td>
<td>40 nA</td>
</tr>
<tr>
<td>Bandwidth (B)</td>
<td>15 KHz – 0.0001 Hz</td>
<td>7.8 KHz – 0.0001 Hz</td>
</tr>
<tr>
<td>TCR ($\alpha$)</td>
<td>$-2.4 %/K$</td>
<td>$-3.9 %/K$</td>
</tr>
<tr>
<td>Normalized Hooge’ Parameter ($\alpha_H/n$)</td>
<td>$10^{-21} \text{ to } 10^{-23} \text{ cm}^3$</td>
<td>$10^{-18} \text{ to } 10^{-20} \text{ cm}^3$</td>
</tr>
<tr>
<td>Thickness</td>
<td>50 nm</td>
<td>50 nm</td>
</tr>
<tr>
<td>Cell Area</td>
<td>17 $\mu$m $\times$ 17 $\mu$m</td>
<td>17 $\mu$m $\times$ 17 $\mu$m</td>
</tr>
<tr>
<td>Johnson Noise</td>
<td>50 pA</td>
<td>1 pA</td>
</tr>
<tr>
<td>1/f Noise</td>
<td>0.072 nA to 7.2 nA</td>
<td>21 nA</td>
</tr>
<tr>
<td>Total Noise</td>
<td>75 pA to 7.2 nA</td>
<td>21 nA</td>
</tr>
</tbody>
</table>

It can be seen that SiGe:H thin films can have lower Johnson noise values, yet much higher 1/f noise in comparison with VO$_x$ thin films. Recent advances in the underlying read out integrated circuits (ROIC) have made a-SiGe based thin films competitive for use in microbolometer devices, as shown in the following section.
2.3.4. Readout Circuits used in Infrared Imaging Focal Plane Arrays

As it can be seen from Table 1 above and Figure 2-3, there are many deposition techniques and numerous materials that can yield thin films that can be used as sensing materials in infrared imaging. Thin films of VOₓ and SiGe:H have all been used for infrared imaging, yielding similar device performances [9] [18]. This begs the question, how can materials having orders of magnitude difference in resistivity and five to ten times difference in TCR result in similar sensitivity of the fabricated microbolometer array? The answer lies in the resistivity of the material and the underlying readout circuitry associated with that resistivity.

2.3.4.1. Low Resistivity Materials and Pulse Biasing

In the case of materials with resistivity < 1 Ω·cm, the device resistance is typically < 10⁵ Ω. When a bias is applied to the thin film resistor on a substrate membrane of SiNₓ, the film undergoes Joule heating which leads to a rise in temperature at a rate of [3]:

\[
\Delta T = \frac{V_{bias}I_{bias}}{c}
\]

where \( c \) is the thermal capacity of the detector. For a bias of 1 Volt this results in the rate of rise of temperature of 50,000 °C/second. To avoid a large rise in temperature, the bias time is limited to about 100 μsec, which limits the temperature rise to about 5 °C [3]. Following the pulse duration, the detector is left unbiased for 29.8 msec such that the thin film cools and reaches the substrate temperature and is ready for the next bias pulse [3]. The total pixel readout time is 30 msec which results in a frame rate of \( \approx 30 \) Hz [9] [3].

Figure 2-14 shows the schematic of the Readout Integrated Circuit (ROIC) developed at FLIR Inc. to multiplex a VOₓ based bolometer array [59]. The entire array is read in batches of 20 × 128 devices at a time with 8 channels to read out the entire array of 160 × 128 [59]. Each channel contains an on-chip bias compensation to provide a uniform output over a wide range of operating temperatures. This allows the operation of the bolometer without the need for thermoelectric cooler stabilization [59].
The pulsed bias readout circuit employed by FLIR inc. is as follows [59]: The bolometers are sequentially biased and multiplexed to amplifiers in groups of eight. The detector is interfaced with the amplifier by a common gate p-channel MOSFET. The MOSFET biases the detector to a programmable voltage using on-chip DACs to vary the MOSFET gate voltage. A programmable load current is generated by thermally shunted bolometers connected to the source of an n-channel MOSFET. The drain current of the n-channel MOSFET is used to offset the detector current such that only temperature difference induced currents are integrated. The temperature induced currents is integrated over a sample period by an integrator and subsequently sampled and held for output multiplexing. The integrator can be reset using a shunt which allows amplification in addition to limiting the bandwidth of the output signal.
The pulse biasing scheme described above has been used for low resistivity materials such as VOx and metallic thin films such as permalloy (Ni-Fe) and titanium.

2.3.4.2. High Resistivity Materials and Constant Biasing

For materials with high resistivity (> 1,000 Ω·cm), the pulse bias method is not necessary and in fact limits the sensitivity of the bolometer array. As discussed in the previous sub-section, these high resistivity materials have a high TCR but also high 1/f noise. For thin films belonging to the SiGe:H material system, the ‘α_H/n’ value is around 10⁻²⁰ cm³; as seen in Table 1 this leads to a 1/f noise value of 20 nA, a value roughly half that of the current signal. At these noise levels, it would be impossible to manufacture bolometer arrays with sensitivities anywhere close to those achieved by low resistivity material.

In the early 2000s, Level-3 Communications designed a unique ROIC which allowed for a dramatic reduction in noise by averaging the continuously biased signal [60]. The ROIC technology contains an in-pixel bias subtraction as well as an in-pixel switched capacitor filter/integrating amplifier to maximize signal integration and signal gain and enables simultaneous signal integration on every pixel [60]. Hanson et al. claim that the novel ROIC design enables the possibility of increasing the array size by adding rows, does not impact the array performance and theoretically allows for multi-megapixel sized FPAs with high performance [60]. A simplified version of the unit cell switched capacitor filter integrating amplifier design is shown in Figure 2-15 [60].
The operation of the readout circuit has been described in ref [60]: in Figure 2-15, the detector pixel is represented by a resistor ($R_{\text{det}}$). Detector signal current is injected through the input field effect transistor (FET M1) by biasing its gate $V_{\text{gg}}$. The detector current is sampled continuously on the integrating capacitor $C_2$ at a rate of ~7.8KHz. The continuously sampled current signal is filtered by the switched capacitor filter network through a transfer capacitor $C_3$ and averaged on capacitor $C_4$. It is at this capacitor, $C_4$, where the signal is averaged and the 1/f noise reduced. For example, for a desired frame rate of 60 Hz, the sample can be averaged over 130 times resulting in 1/f noise reduction by a factor > 11. The processed signal on capacitor $C_4$ is sampled and read out at the desired frame rate. Hanson points out that the FPA sensitivity is not dependent on frame rate but rather on the KHz rate at which signal is integrated on $C_2$. Although a high integration frequency would help further reduce 1/f noise, the Johnson noise value would then increase. The sweeping frequency is chosen in a way that 1/f noise and the Johnson noise contribution is minimized. The filter network is chosen such that the noise bandwidth is significantly lower than the Nyquist frequency. The noise bandwidth is substantially lower than the filter Nyquist frequency.

Figure 2-15. Schematic of the L-3 EOS switched capacitor filter/integrating amplifier integrated in the unit-cell of each pixel as in ref [60].
Hanson claims that for the above mentioned reasons, L-3 ROIC’s performance is independent of frame rate making frame rates of 60Hz, 120Hz, and beyond achievable without performance loss [60].
Chapter 3. Experimental Methods

3.1. Deposition of VO\textsubscript{x} Thin Films: The LANS Biased Target Deposition System

Thin films of VO\textsubscript{x} were deposited by a novel sputtering technique known as Biased Target Ion Beam Deposition (BTIBD) or simply Biased Target Deposition (BTD) (manufactured by 4Wave Inc. based in Sterling, VA).

![Biased Target Ion Beam Deposition System](image)

Figure 3-1. Concept sketch of Biased Target Ion Beam Deposition System [61].

A concept sketch of this technique can be seen in Figure 3-1 [61]. The operation principle of the technique has been discussed elsewhere and is summarized [47]: In this deposition technique, a low energy, broad beam ion source (Energy of ions < 25 eV) is used to illuminate the target. No attempt is made to focus the ion beam on to the target material. The ion energy of the beam is low compared (<40 eV) and so the sputter yield of materials in the chamber from this beam is minimal. Once a stable ion beam has been established, a negative bias is applied to the target. This negative bias establishes a sheath in front of the target which accelerates the positively charged ions from the ion beam to cause sputtering.
The deposition system used in this work is detailed in Figure 3-2. This system is sold as the LANS BTIBD tool. In this geometry, the low energy ion-beam producing ion gun is facing the substrate and illuminating three targets. Targets are biased using a single programmable pulsed DC power supply source. The pulsed DC power source is connected to the three targets in parallel configuration to allow for simultaneous sputtering of up to three targets. Therefore the control of adatom arrival rate and the adatom energies is accomplished threefold by:

1. Sputter yield is controlled by the ion current established by the ion source.
2. Sputtering rate is additionally controlled at each target by controlling the pulse frequency and the duty cycle.
3. The adatom energy is decided by the operating pressure.

![Figure 3-2. Schematic of the Laboratory for Alloying and Nano layer Sputtering by the 4Wave Inc. used in this work [62].](image-url)
3.1.1. Ion Source used in BTD: Broad Beam, Gridless Ion Sources

A broad-beam ion source is defined as a beam of ions whose diameter (length of transverse direction) is much larger than the physical dimensions of its optics or the Debye length of the accelerating voltage [63]. These sources are much more interesting as ion sources for thin film processing applications than other types such as Von Ardenne type ion source (mainly used for high energy thin beam of ions carrying high energy and multiple charges), Electron Cyclotron Resonance Type (ECR) ion sources, electron beam ion sources, laser ion sources and vacuum arc ion sources. Broadly speaking, there are two types of ion sources commercially used in thin film processing, gridded and gridless type [63]. The fundamental difference between the two types of sources is that while gridded ion sources produce ions with a narrow energy distribution, gridless ion sources produce ion beams that are unfocussed and have a broad energy distribution [63] [64]. This section is aimed at discussing the fundamental theory of operation of the ion source in the context of this work.

The Hall-current ion sources operate on the principle of significantly increasing the plasma’s electrical resistance and the electron lifetime in the plasma using a magnetic field [63] [64] [65]. The increased electron lifetime allows for an increase in the interaction time of the electrons with the neutral atoms/working gas which is being expelled from within the ion source.

In the ion source discharge channel, the electrons are magnetized. If \( \omega_e \) is the electron cyclotron frequency and \( \tau \) is the average time between collisions with other particles and the chamber walls, then \( \omega_e \tau \gg 1 \) [61] [63] [64]. These magnetized electrons move from their source of origin (cathode/electron source) to the anode (ion source discharge channel) in a helical path prescribed by the electrical and magnetic fields. Under optimum conditions, the ions are usually not magnetized, i.e. \( \omega_i \tau \ll 1 \) (where \( \omega_i \) is the ion cyclotron frequency in the magnetic field and \( \tau \) is the average time between the ion collisions with other particles and the chamber walls) [61] [63] [64]. The ions move between their source of origin (anode discharge channel) and the cathode along the electrical field. During this path, the plasma picks up
the necessary number of electrons for neutralization, resulting in a quasi-neutral beam of ions and electrons known as the ion beam [61] [63] [64].

Unlike in gridded ion sources where the ion beam experiences space charge limitations due to limitations imposed by Child’s law, the Hall-current sources do not have any limitation for the ion beam current that can extracted [63].

Schematics of the geometry and the electrical circuit of the most popular type of ion source are shown Figure 3-3a and Figure 3-3b. A detailed description of the operation of the ion source can be found in reference [61] but is briefly discussed here: Figure 3-3b shows the electron emission from the cathode is controlled by the use of an AC power supply connected to the electron source. The anode potential is determined by the anode current, the magnetic field strength and the gas flow. The power supply to an electromagnet impresses a variable magnetic field on the electrons being emitted from the cathode. For a known regime of operation, a permanent magnet can replace the electromagnet, thereby eliminating the use of an extra power supply. A mass flow controller is used to control the amount of gas being introduced into the system, and thereby control the conductivity of the plasma. This gas flow dictates the discharge voltage of a particular ion beam discharge current. The cathode used in normal operation is either a filament type or a hollow cathode type.
3.1.1.1. Ion Generation Mechanisms

The operating principle and various processes of the anode are discussed as follows and are pictured in Figure 3-4. The neutral atoms/gas molecules are introduced using a mass flow controller (1). Electrons (2) generated at the cathode, crossed between electric field and magnetic field lines follow the path given by (3) in a helical manner towards the discharge chamber of the anode. These electrons strike the neutral atoms/molecules (4) and ionize them (5). A combination of electrons (2) and ions (5) forms the plasma which is directed downstream by the radial and axial components of the electric field as well as a gas distributor/reflectector.

Due to the conical axisymmetric shape of the discharge chamber and the positive charge enforced on the anode, the ions that are created near the discharge chamber are accelerated away from the anode in the axial direction. The velocity acquired by these ions and the positive potential on the other side of the axis forces the ions to crisscross the axis multiple times and undergo several collisions [66]. The positive space-charge and the current due to the ions are neutralized by electrons generated from the electron gun.
These processes result in a divergent, neutral beam comprised of ions (6) and electrons (7) of ions being generated and directed downstream from the discharge chamber.

Figure 3-4. Various processes in an end-Hall ion source [66].

It should be noted that discharge current of the anode and the cathode does not equal the electron and ion currents in the quasi-neutral beam. In fact, the current to the anode is almost entirely composed of electrons – both the electrons from the electron source, and secondary electrons generated during ionization. The excess electron generation from the cathode is usually sufficient to current-neutralize the ion beam with electron emission from the anode when the cathode current is set to the anode current [66]. However, the cathode current is usually set in excess of this current.

3.1.1.3. Electric Discharge and Voltage-Current Characteristics

Since, typical thin film processing/modification pressure ranges from $10^{-5}$ Torr and $5 \times 10^{-3}$ Torr (Figure 3-5), ion sources must be operated in this region for ion assist modifications. Ion beam operation
in this pressure range exists in primarily two modes of operation, a) a self-sustained discharge and b) a non-self-sustained discharge.

Figure 3-5. Voltage-current characteristics (between $5 \times 10^{-5}$ Torr and $1 \times 10^{-3}$ Torr) of an end-Hall ion source showing the various regimes of operation of its plasma discharge. The two curves show the voltage-current characteristics for a discharge current of $I_d = 5A$ and corresponding ion beam current of $I_i$ [61].

In the self-sustained discharge region, the plasma is comprised of ions generated by the ion source and a number of electrons equaling the total positive charge in the plasma [61]. These electrons are generated when a plasma discharge voltage of $V_d \geq 300 - 350V$ is applied between the anode (ion source) and any surface that can act as a conductor in the vacuum chamber (chamber walls, flanges, bellows, etc.). These surfaces can potentially serve as the cathode materials from which a sufficient number of electrons can be generated from the ion bombardment. Because no external source of electrons is needed, this discharge is known as a self-sustained discharge [61].

However, at discharge voltages between 50 V and 300 V, the discharge voltage by itself does not produce enough secondary electrons to sustain the discharge [61] [64]. This region of operation is called a non-self-sustained discharge. In this type of discharge, an external source of electrons is needed to
generate and maintain stable plasma. The source of electrons is either a hot filament (HF) or a hollow cathode (HC), and will be discussed in the next section.

The voltage-current characteristics of a Hall current ion source depend on several factors such as [61]:

1. Pressure at the discharge channel. Higher the pressure, the lower the discharge voltage.
2. Magnetic field value atop the gas reflector at the anode. In general, the lower the magnetic field, the lower the ignition voltage (However, this leads to a lower ion beam current)
3. System geometry, anode dimensions and the relative placement of the electron source.

3.1.1.4. The Ion Source for the LANS BTD System

For this thesis, an ion source is required which produces ions with energy less than the sputter threshold of all material in the deposition chamber. In addition, the ion source required must be rugged for operation in a reactive environment. The EH-1000 compares well to the required specifications (Figure 3-6). Some key features are as follows [63] [67]:

1. A hollow cathode was used to as the source of electrons to prevent cathode oxidation during reactive sputtering.
2. The anode was grooved (Figure 3-7) to prevent the oxidation/deposition of dielectric materials that prevents operation at a low discharge voltage.
3. The discharge voltage was reduced to < 30 V by reducing the strength of the magnetic field in the discharge chamber. The ion source was custom designed for this sputter system by Kauffman and Robinson Inc. The magnetic field was established using an Alnico magnet whose field intensity was reduced to 400 G (versus the original 550 G) atop the gas reflector. This reduced magnetic field strength was measured using a F.W. Bell Gaussmeter, Model 410 and compared with quoted values from the product technical specifications.
A retarding potential analysis (Figure 3-7) at different angles at a distance of 30 cm from the center of the ion source shows that > 90% of the ions have energies < 30 eV, which is less than the sputter threshold of most materials in the deposition chamber [61] [68].

Because End-Hall ion sources are unfocussed ion generators, it is important to understand the angular distribution of ion current produced by the ion sources. Figure 3-8 shows the angular distribution of ion current density at a distance of 30 cm from the ion source, as is the case in the 4Wave’s LANS System [68].
Figure 3-8. Spherical ion current density profiles for the KRI End-Hall 1000 ion source with the source at the center of the sphere. Source to target distance is 30 cm (12 in.). The working gas was 70 sccm of argon and the discharge characteristics were 10 A and 45 V [68].

3.1.2. Hollow Cathode Electron Source

As discussed in the previous sections, the ion source’s mode of operation of interest lies in the non-self-sustained discharge region: an external source of electrons is required for ionization and neutralization of the space charge region of the ion beam. Without an external source of neutralization the plasma is under neutralized and extracts electrons from any object in the deposition chamber [61] [64]. This extraction manifests itself in the form of an undesirable arc that could last for a several microseconds and whose frequency of occurrence may depend on the degree of under neutralization of the ion beam.

In an ion source, the discharge current is given by the sum of the ion current and electron current produced by the discharge:

\[ I_d = I_{i,d} + I_{e,d} \]

In the case of a non-self-sustained discharge \( I_{i,d} > I_{e,d} \), the neutralizer should provide the difference between the ion and electron current such that the net neutralization electron current is equal to the ion current. This can be expressed as:
Therefore the ion source discharge current is the sum of ion current and the current from an electron neutralizer as:

\[ I_d = I_n = I_{e,n} + I_{e,d} \]

Typically the electron current from the neutralizer is set to a value higher than the ion source discharge current. The effect of under neutralization can be seen in Figure 3-9. The additional electron current not only facilitates the ionization of the working gas in the ion source discharge chamber but also to produce an ion beam with low energy spread as shown in Figure 3-9.

![Figure 3-9. Typical ion beam energy distribution for different neutralization ratios: \( I_{em} \) (3.8 A) < \( I_d \) (4 A), \( I_{em} \) = \( I_d \) = 4 A and \( I_{em} \) (6 A) > \( I_d \) (4 A) [61].](image)

There are two main types of electron sources: the Hot Filament type (HF) or the Hollow Cathode type (HC). Due to the limited lifetime of HF type source in thin film deposition, HC type electron sources are commonly used.

Hollow cathode electron sources are an alternative source of electrons that overcome the shortcomings of the hot filament type electron sources as described in the previous section. In these types
of sources, a refractory metal tube is negatively biased to create argon ion bombardment. This ion bombardment causes significant heating, leading to thermionic emission from the refractory metal (tungsten) [61] [66] [69]. Because of the low pressure regime of operation of these electron sources (< 0.01 Torr), these electron sources are operated by maintaining a constant flow of ionizable gas through them [61] [66] [69].

Thermoelectron emission current of a material with a square potential barrier is governed by the Richardson-Dushman equation [70]. For a conductor at temperature T and a work function φ, the emission current density is given by:

\[ j = A_0 T^2 e^{-\phi/kT} \]  

Equation 18 [70]

Where \( A_0 = 4\pi me^2/h^3 = 120.4 \text{ A/cm}^2\text{K}^2 \) is the Richardson constant for a tungsten filament, T is the temperature of operation (≈2500K) for the tungsten filament and \( k \) is the Boltzmann’s constant, \( k = 1.38 \times 10^{-23} \text{ J/K} \). This gives a typical current density of ≈ 0.65A/cm² [61].

One of the first hollow cathode assemblies was described by [69]. Since its invention, there have been numerous performance improvements. One such iteration of the hollow cathode is manufactured by the Kaufman & Robinson Inc. and is shown in Figure 3-10 [71].

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Figure 3-10. Schematic and electrical circuit diagram of a hollow cathode assembly [71].
The operation of this Hollow Cathode is described in reference [68] and is summarized here:

Prior to operation of the hollow cathode, a flow > 75 sccm is maintained to purge contamination physisorbed in the hollow cathode assembly. This high flow rate also produces a large number of electrons which facilitates to strike the plasma. To start the hollow cathode electron source (HCES), a voltage of several hundred volts is applied between the keeper 17 and the cathode body 90. Once a plasma is established between the keeper and the cathode, the keeper current should rise to ≈ 1.5 A (for LHC-1000). At this point sufficient argon ions are generated in the gas 13, flowing out of the cathode tip to start thermionic emission of the cathode. A current controlled power supply can be used to supply a positive to the assembly body with respect to the tip 90, thereby causing ion bombardment of the tip.

Figure 3-11. An end-Hall ion source showing the typical placement of a filament [68].

The hollow cathode design adopted in the biased target deposition of this work is one of such iterations detailed in a 2010 patent filing (Figure 3-11) and realized as LHC-1000 electron source by Kaufman & Robinson Inc. [71]. Figure 3-11 shows the ion and electron source assembly. The electron source is physically mounted on the ion source in the direction of the ion beam propagation.
3.1.3. Summary of Operation of 4Wave LANS system

The key points that are to be kept in mind when setting parameters for deposition in this tool are:

1. The ultimate requirement for deposition with this technique is obtaining a large flux of ions in the deposition chamber
2. A large amount of electrons must be generated for igniting and sustaining ion generation of low energies
3. The gridless type ion source produces ions with different energies whose distribution is angular. The ions along the axis carry the highest energy and can be used for secondary conditioning of the growing thin film with minimal contamination from the sputtering of the chamber walls.

Table 2 gives the parameters used for deposition in this work. The only variable in this work is the partial pressure of oxygen whose values will be given in subsequent chapters in which VO₅ thin films were deposited.

<table>
<thead>
<tr>
<th>Hollow cathode (HC) Ar flow rate: 10 sccm</th>
<th>Ion Source Ar flow rate: 60 sccm</th>
<th>O₂ flow: Varies based on oxygen partial pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Keeper V/I: 20 V/1.5 A</td>
<td>Bias V/I: 23 V/8 A</td>
<td>Source V/I: 38 V/8 A</td>
</tr>
<tr>
<td>Target Material(s)/Gun(s):</td>
<td>V/I (Gun 1): –800 V +5 V</td>
<td>V/I (Gun 3): –800 V +5 V</td>
</tr>
<tr>
<td>3 Vanadium Targets</td>
<td>V/I (Gun 5): –800 V +5 V</td>
<td></td>
</tr>
<tr>
<td>Spin: 20 rpm Time: 30 min.</td>
<td>V/I (Gun 1): 38 V/8 A</td>
<td></td>
</tr>
<tr>
<td>Pulse Width #1: 1 μsec</td>
<td>V/I (Gun 3): +5 V</td>
<td></td>
</tr>
<tr>
<td>Period #1: 100 μsec</td>
<td>V/I (Gun 5): +5 V</td>
<td></td>
</tr>
<tr>
<td>Pulse Width # 3: 1 μsec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pulse # 3: 100 μsec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pulse Width # 5: 1 μsec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pulse # 5: 100 μsec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P_total: 5.8 × 10⁻⁴ Torr</td>
<td>P_{H₂O}: &lt; 2 × 10⁻⁷ Torr</td>
<td>P_{O₂}: Varies</td>
</tr>
<tr>
<td></td>
<td>P_{H₂}: &lt; 1 × 10⁻⁷ Torr</td>
<td></td>
</tr>
</tbody>
</table>

3.2. Deposition of Hydrogenated Ge:H Thin Films

Thin films of Hydrogenated Germanium (Ge:H) were deposited using a research scale Plasma Enhanced Chemical Vapor Deposition (PECVD) with 13.56 MHz RF power capacitively coupled to the two electrodes of the system as can be seen in Figure 3-12. The thin films of Ge:H were deposited using
the carrier gas germane (GeH₄) gas diluted with hydrogen before entering the deposition chamber by a variable dilution ratio. The dilution ratio \( R = \frac{[H₂]}{([H₂]+[GeH₄])} \) is programmable by means of varying the gas flow using a mass flow controller. The dilution can be increased or decreased by increasing the amount of GeH₄ to H₂.

The deposition of thin films of hydrogenated SiGe alloys using PECVD is well known with numerous reports detailing the process. In CVD, a source gas is used in the fluid-flow regime (High pressure). Briefly, during the PECVD deposition of Ge:H, the carrier gas for germanium (GeH₄) is mixed with hydrogen gas (H₂) and introduced into the chamber through a mass flow controller (MFC) which is programmable for a certain dilution ratio. Figure 3-12 shows a schematic of the chamber used in this work. A low power plasma is struck with a power density of 0.08 W/cm², using an inductively coupled plasma at a radio frequency of \( f=13.56 \text{ MHz} \). By electron impact ionization, the gas disassociates to produce a variety of ions, radical species and primary and secondary electrons. These species may adsorb onto the surface of the substrate to form a thin film.

![Figure 3-12. Schematic of the plasma enhanced CVD deposition chamber used to deposit the Ge:H of this work as it appears in ref. [72].](image-url)
The spacing between the electrodes and the plasma power density are used to control the plasma kinetics and therefore the ion bombardment experienced by the growing thin film. The substrate temperature on the other hand influences the surface reactions on the substrate. Low substrate temperatures may lead to a low density, disordered thin film because the impinging species may not have enough adatom mobility. On the other hand a high substrate temperature may lead to reduced hydrogen incorporation, which may leave the growing thin film unpassivated.

The total flow rates and the pressure for deposition, as with any vapor deposition, decides the residence time of the disassociated species created during the RF plasma. Large deposition pressures may lead to significantly reduced mean free paths. This in turn leads to an increase in gas phase reactions and produces an undesirable number of particles.

In the case of Si:H deposition, the process parameters have been optimized for infrared imaging applications over many years of active investigation [53] [73]. The substrate temperature is usually kept at 200 °C, the total pressure is around 0.5 Torr and a low plasma is applied (plasma density ~0.08 W/cm²) [74]. These deposition conditions optimized for Si:H were found to be non-optimal for thin films of Ge:H [53].

3.3. Characterization Techniques

3.3.1. Grazing Incidence X-Ray Diffraction (GIXRD)

Grazing-incidence diffraction is a scattering geometry that combines the Bragg condition with the conditions for x-ray total external reflection from crystal planes to identify the crystalline phase of the sample under investigation. This configuration uses small angle of incidence and reduces the penetration depth of the x-ray by up to three orders of magnitude (typically from 1-10μm to 10-100Å).

Grazing incidence x-ray diffraction was done using a PANAnalytical PRO X’Pert MPD with Cu Kα1 (λ = 1.54 Å) radiation, to analyze the structure of the deposited thin film. Diffraction patterns were
collected for 20 values between 10° and 85°, and a scan rate of 1.2°/min. The crystalline phase and the lattice parameter were determined by comparing the peak positions and intensities and their corresponding 20 values to that of standards using MDI Jade V9.0 software.

3.3.2. Atomic Force Microscopy (AFM)

Atomic force microscopy is a form of surface probe microscopy technique in which a sharp tip is scanned across the surface and the surface-tip interaction force is used to infer the surface morphology of the surface under investigation. For measurements in this work, measurements were made using the peak force tapping method using instrumentation developed by Bruker Icon.

3.3.3. Rutherford Backscattering Spectroscopy (RBS)

Rutherford Backscattering Spectroscopy (RBS) was used in this work to determine the stoichiometry of the VOₓ thin films. RBS operates on a phenomenon that occurs when a high-energy primary ion (typically He⁺⁺ ions with energies in the order of MeV) is elastically scattered. The energy and angle of the scattered ion yields information about the mass of the scattering atom in the sample. This information then can be used for composition analysis of thin films.

All RBS data described herein was collected at the Tandem Accelerator at the Department of Physics and Astronomy, Rutgers University. Figure 3-1 describes the schematic used for RBS experiments. All measurements were performed in a standard RBS chamber under a vacuum of 10⁻⁶ Torr using two Si surface barrier detectors: one at 155° and other at 100° scattering angle. A 2 MeV He⁺⁺ ions from a 1.7MV tandem accelerator was used with an ion current of 2-3nA and a beam spot of 2mm.
Figure 3-13. Schematic of a typical RBS experimental setup.

This ion beam is deflected into the RBS chamber, where it is incident on the sample under investigation. The He$^{++}$ ions that are deflected are backscattered and their intensity is measured at a Si surface barrier detector which is placed at a 17° angle with respect to the incoming incident radiation. Data was collected till 10μC of charge was accumulated (typically 15 minutes).

3.3.4. Ellipsometry

Ellipsometry is a non-destructive technique which measures the change in polarization of a reflected beam of light from the sample’s surface with a known polarization state. The change in the polarization of light depends on the structural and the optical properties of the sample under investigation. Unlike x-ray diffraction, ellipsometry is a portable and versatile tool that can be used to monitor in-situ the growth of thin films. Ellipsometric measurements in this work were conducted by using a J.A. Woollam Co. M2000-DUV variable angle rotating-compensator multichannel spectroscopic ellipsometer. This instrument uses two light sources, a Xe lamp and a Quartz Tungsten Halogen (QTH) lamp. By combining both light sources, the attainable ellipsometric spectra from the RC2 instrument spans over the
0.73 – 5.15 eV (1700 – 240 nm) spectral range. Figure 3-14 shows the schematic of an ellipsometry configuration. The measured response is a function of optical properties and the thickness of the thin film.

Figure 3-14. Typical configuration for ellipsometry measurement [75].

3.3.5. Resistivity Measurements

The resistivity of the thin film deposited was measured by using the Transmission Line Method (TLM). The TLM method gives a complete characterization of the contact by providing the sheet resistance, the contact resistance, and the specific contact resistivity. For top contacts to VO\textsubscript{x} thin films, 100 nm of titanium was deposited using the Perkin Elmer sputtering system. Bottom contacts to VO\textsubscript{x} thin films were obtained using only Vanadium as the bottom metal. The mask set used to extract resistivity values is shown in Figure 3-15. It consists of two layers, the first of which is used to isolated the TCR material so as obtain an accurate value of resistivity; the second layer consists of a series of five electrodes for metal contacts with spacings of 10\textmu m, 30\textmu m, 70\textmu m, 150\textmu m and 310\textmu m respectively.
Current–Voltage (I-V) characteristics were measured by sweeping voltage using a Hewlett Packard 4140B pA meter/ DC Voltage source and the resistance was recorded. A LabVIEW program was used to collect the data. The I–V curves were examined to ensure ohmic behavior of the resistance structures. The resistance of each resistor with different spacing is plotted as in Figure 3-16. The Y–intercept gives the contact resistance. Apart from ensuring ohmic contacts, another important parameter used to assess the quality of the contacts is the transfer length [76]. Half the value of the x–intercept gives a value known as the transfer length. Transfer length is defined as the distance at which ‘1/e’ of the current is transferred from the semiconductor to the electrode or vice-versa. Thus an accurate measurement of the transfer length helps to determine the minimum spacing of electrodes (therefore the size of the active region) that can be used to extract electrical properties from the sample with minimal contribution from the contact resistance itself.
3.3.6. Temperature Coefficient of Resistance Measurements

As a prerequisite to measurements, electrical contacts must be made to the thin films. Metal contacts were deposited onto the thin film whose electrical properties need to be measured. Prior to the measurement, the contact quality is evaluated to ensure that Ohmic contacts were obtained. An HP4141B picoammeter was set up to source voltage and bias the thin film resistors, the resultant current was recorded. These I-V measurements were performed as a function of temperature from 30°C to 65°C at 5 °C increments. The data was exported into excel and the resistance was calculated at each temperature step. The resistivity is a function of temperature and can be described by:

\[ \rho = \rho_0 e^{\frac{-E_a}{k_B T}} \]  

\[ \text{Or,} \quad \ln(\rho) = \frac{-E_a}{k_B T^2} + \ln(\rho_0) \]  

Equation 19

Equation 20

Figure 3-16. Plot of total resistance as a function of electrode spacing.
A Ln resistance versus ‘1/k_B T^2’ plot was used to extract the activation energy of the thin film. The TCR can be calculated using the formula:

\[ TCR = \frac{-E_a}{k_B T^2} \]

Equation 21

3.3.7. Measurement of Electrical Noise in Thin Films

As discussed in the previous chapter, the electrical noise in a thin film is comprised of two components, the Johnson noise and the 1/f noise. While the Johnson noise is a function of device resistance and the bandwidth of the measurement, the origin of 1/f noise, on the other hand, depends on the value of Hooge’s parameter and the carrier concentration. Therefore in order to compare the inherent noise of a thin film the parameter ‘\(\alpha_{1/f}/n\)’ or the normalized Hooge’s parameter must be extracted.

If the parameters \(V, f, I_{bias}\) and the measured \(I_{noise}\) are known, ‘\(\alpha_{1/f}/n\)’ (units: cm^3) can be extracted from Equation 14 such that:

\[ \frac{\alpha_H}{n} = \frac{I_{noise}^2}{I_{bias}^2} \cdot V \cdot f \]

Equation 22

Thin film samples were patterned using lithographic processing to define a known volume of the sample area. Figure 3-17 shows the patterns used to extract the normalized Hooge’s parameter.
Each pattern in Figure 3-17 contains three distinct volumes; however for each volume the aspect ratio (width to length ratio) is maintained the same. The similar aspect ratios allows for interrogation of different volumes while the thin film resistance remains the same. The lithographic patterning was done as shown in Figure 3-18 using Shipley Microposit 1800 series photoresist and Poly(methyl methacrylate) or PMMA C4. The active material was patterned using either dry or wet etching techniques. Thin films of Ge:H were etched in 30% hydrogen peroxide (H₂O₂). Since VOₓ thin films are etched in the developer we used (Microposit 351), a double layer lithography technique was used with PMMA C4, as the underlying layer since PMMA can be developed in toluene (non-aqueous) post-exposure. A dry etch recipe was developed which utilized CF₄/O₂ plasma (100 mTorr, 25 sccm CF₄: 100 sccm O₂, -100V self-bias at a power of 100W). For high resistivity VOₓ the etch rate was found to be ≈ 40 nm/min.

It was observed that for vertically integrated (through film) devices of VOₓ, dry etching caused the thin film of VOₓ to break down, and so, a wet etch recipe was also developed. High resistivity thin films of VOₓ were found to be etched in dilute solutions of H₂O₂ (1:2,000) at an etch rate of 200 nm/min.
Once the sensing material was patterned, top electrodes were deposited using double layer lithography using PMMA as the liftoff resist. 100 nm of chromium was sputtered for making ohmic contacts to Ge:H, while 100 nm of sputtered titanium was used for thin films of VO$_x$.

Since the 1/f noise of a device also depends on the electrical contacts to the semiconductor [77], it is necessary to evaluate the contact quality and choose a device geometry from which the contribution from the contact resistance is minimized. Transmission line measurements were made and the contact resistance was evaluated. Electrical noise measurements were made on patterns such that the total contact resistance was significantly less than the total device resistance under consideration.

Previous attempts to measure 1/f noise of thin films have been through the measurement of a current power spectral density (PSD) at a specific volume and a reference frequency using a known electrical bias. Such spot measurements are performed using a dynamic signal analyzer (DSA) and are limited to one data point per measurement and could include unwanted contributions from the system noise, Joule heating, temperature fluctuation of ambient air, etc.

In this work, a custom-built 1/f noise measurement system and the technique to extract the 1/f noise of thin films is described which isolates the material 1/f noise from other noise sources. Some advantages to this noise measurement system over previous methods include: a large frequency range of
data acquisition obtained in a single measurement, a user controlled bandwidth of measurement, digitization of the output signal for post-measurement processing of the data, and low frequency drift correction for accurate measurement of the thin film’s 1/f noise. The construction of the automated 1/f noise measurement system was mostly developed by Myung-Yoon Lee from the department of Electrical Engineering at the Penn State University.

3.3.7.1. Effect of Low Frequency Drift for the Measurement of Noise in Thin Film Resistors

Some devices consist of materials sensitive to temperature fluctuations while others may contain materials whose electrical property drifts due to physical changes associated with aging of the thin film in ambient. In such devices, the electrical output of the film drifts over time [77]. During their regular operation, these thin films are isolated from the ambient by vacuum packaging and temperature stabilization. However, these provisions are not feasible in laboratory desktop style measurement system in which numerous samples deposited using various deposition conditions need to be analyzed. Figure 3-19 shows the drift in resistance of a thin film of vanadium oxide measured over more than 45 hours. The resistance change manifests itself in the measured current as a sinusoidal variation with a long, non-constant time period (low frequency) which we refer to herein as ‘drift’.
This drift causes variations in the measurement which makes both data acquisition and data analysis difficult. When the acquired time–signal data is converted into the frequency domain, this low frequency drift causes a shift in the acquired spectra and makes the $1/f$ noise of the thin film appear larger than its true intrinsic value.

In the measurement system developed for this work; the low frequency drift has been subtracted using post-measurement data processing. A voltage offset correction has been applied whose value as calculated as follows. Let us assume that the measured voltage output can be modeled by a sinusoidal signal using the following equation:

$$ V = X \sin(2\pi ft) + Y + Z t $$  \hspace{1cm} \text{Equation 23}

Where $V$ is the voltage noise modeled, $X$ is the amplitude of $1/f$ noise, $Y$ is a constant voltage offset required to subtract the bias voltage, $Z$ is the magnitude of the drift effect, $f$ is the frequency and $t$ is the discrete time which has a range of 0 sec to 10 sec for our measurement, so that the lower limit of frequency is 0.1Hz. When the acquired time domain signal is converted into frequency domain, the
obtained result is shown in Figure 3-20. The noise PSD displays a sharp peak given by the $1/f$ noise at a frequency $f$ of 10Hz.

![Figure 3-20](image)

**Figure 3-20.** The recorded PSD for varying magnitudes of drift, C. The peak represents the $1/f$ noise (in this case at 10 Hz).

We observed no change in the spectra with the magnitude of the offset value used; which implies that the $1/f$ noise is not affected by the offset of the amplifier at least when offset value is within the input range of filter or the measurement instrument. On the other hand, the plotted $1/f$ noise spectrum of Figure 3-20 is strongly affected by the drift of resistance of the thin film which appears as a linear function of $f^2$. As the drift effect gets larger, the $Z$ value increases and can even surpasses the $1/f$ noise of the material seen here for large values of C and for smaller frequencies. However, due to the difference in their frequency dependence, (Drift has a $1/f^2$ dependence, while flicker noise has a $1/f$ dependence), they can be separated from each other. The noise measurement system developed in this work consists of drift filtering to obtain a noise spectral density that follows Hooge’s empirical relationship and has a slope of negative unity. It must be noted that since we attribute this drift to self-heating of the resistor, we find this drift particularly large when a large power is dissipated across the resistor.
A schematic flow chart of the noise measurement setup is shown in **Error! Not a valid bookmark self-reference**. The setup consists of three instruments: A low noise current amplifier (A Stanford Research SR-570), a dual channel filter and a data acquisition hardware (DAQ) card which is controlled by a personal computer. The SR570 contains an RS-232 interface which allows for remote control of each operation eliminating the need for a manual operation. The SR570 has internal batteries capable of providing up to 5V. To avoid the need for microscope and a probe station, and hence make the setup simpler, a 16 pin ceramic dip package is used along with Al or Au wires. The dip package is placed in a faraday’s cage (a small Al box) to isolate the sample from the environment. A second shielding box is used to isolate the entire shielding assembly and the measurement setup from the environment. In addition, all assemblies and instruments are grounded using a heavy copper wire. A floating table is used to isolate the measurement assembly from all sources of vibration.

Using a LabVIEW program, the wire bonded samples were biased using the internal battery of the SR-570. Prior to data acquisition at the DAQ, proper filtering of the acquired data is necessary. A low pass filter of 1 KHz is used as an anti-aliasing filter to remove the high frequency signal, which may affect the lower frequency noise. In addition a high pass filter of frequency 0.1 Hz is necessary to remove any low frequency drift which leads to erroneous values of the thin film’s inherent 1/f noise. To avoid aliasing, sampling rate of DAQ should exceed Nyquist rate. We have used a sampling rate of 20 KHz, much higher than the Nyquist rate which is set to be \( \approx 2 \text{ KHz} \) \((2 \times (1 \text{ KHz} – 0.1 \text{ Hz}))\). The acquisition time is set to 20 seconds, and the data collected is averaged over 20 samples. At the end of the measurement, the sample is connected to a pico ammeter (HP 4140B DC SMU) and the current bias is recorded.
Figure 3-21. A schematic showing the process flow used in this work for the measurement of 1/f noise in thin films.
The averaged time varying signal is converted into the frequency domain using the LabVIEW’s Fast Fourier transform function. This power spectral density (PSD) is plotted as a function of frequency such that the resultant graph resembles the schematic representation of Figure 3-22.

![Power Spectral Density Plot]

Figure 3-22. Frequency spectra representation of collected spectra showing two distinct regions: a) Region with 1/f noise and, b) Region with Johnson noise

If the magnitude of the 1/f noise is higher than the Johnson noise, the lower frequency of the spectra is a line with a slope of ‘–1’ in a log-log scale. An anti-aliasing filter roll off appears at 1 KHz which is associated with the low pass filter; whereas the Johnson noise appears as ‘white’ because it is independent of the frequency. Depending on which noise contributor is larger the recorded power spectral density (PSD) may differ from Figure 3-22. If the device resistance is high, the total noise may be dominated by Johnson noise and the 1/f noise may be suppressed in the spectra. In such a case, an alternate aspect ratio of the same thin film must be measured such that the device resistance and therefore the Johnson noise is lower than the thin film’s 1/f noise.
In summary, In order to successfully extract the normalized Hooge’s parameter it is necessary to ensure that the Hooge–Vandamme relationship is satisfied. As Equation 22 suggests, the intensity of the recorded PSD depends on:

1. $f^{-1}$ (1/frequency) of measurement,
2. The volume of the device interrogated,
3. The bias applied to the device and
4. The normalized Hooge’s parameter or ‘$\alpha_{n}$/n’.

Thus if everything but normalized Hooge’s parameter is known, it can be estimated by using Equation 22. Figure 3-23 shows the acquired PSD of the data with decreasing volume of the sample and with increasing bias applied to the sample. If the sample bias is kept constant and the volume is halved, the magnitude of PSD is doubled. Similarly if the volume is kept constant and the applied bias is doubled, the magnitude of PSD is also doubled.

![Figure 3-23. Acquired Power Spectral Densities of a thin film for different volumes and different biases to confirm the Hooge–Vandamme relation and extract the materials normalized Hooge’s parameter.](image-url)
Chapter 3 has provided an overview of the experimental methods used in this work. Detailed specifics are added in the following chapters as necessary. Having discussed all the tools required for characterization of thin films, the next chapter looks at high TCR thin films of VO$_x$ and Ge:H for use as infrared imaging materials.
Chapter 4. High TCR Thin Films of Vanadium Oxide (VO_x) and Hydrogenated Germanium (Ge:H)

4.1. Introduction

Thin films of vanadium oxide (VO_x) and hydrogenated amorphous silicon (a-Si:H) are the two dominant material systems used in resistive infrared radiation detectors for sensing wavelengths in the 8–14 μm range. While thin films of VO_x (x < 2) currently used in the bolometer industry have a magnitude of temperature coefficient of resistance (TCR) between 2%/K – 3%/K, the magnitude of TCR of a-Si:H thin films lies between 3%/K to 4%/K [8] [30] [78] [18]. Although high TCR materials are desired, increasing the activation energy (and therefore the effective band gap) results in a decrease in the mobile carrier concentration and therefore resistivity [30]. This is evident from the fact that while VO_x thin films have a resistivity between 0.1 – 1 Ω–cm, a-Si:H used in microbolometers have a resistivity between 200 and 2,500 Ω–cm [8] [30] [78] [18].

Unfortunately high resistivity materials are also associated with high Johnson and 1/f noise. The fundamental differences in resistivity and electrical noise of VO_x and a-Si:H thin films used in the microbolometer industry requires two distinctly different read out circuits [18] [60] [59]. To overcome the higher noise in a-Si:H thin films, the devices are constantly biased and averaged numerous times at the pixel level so that the noise contribution can be reduced dramatically [18] [60]. Such a constant bias readout circuit cannot be used for thin films of VO_x due to the self-heating associated with their lower resistivity [30] [59]. In 2013, Jin et al. demonstrated thin films of VO_x having the lowest resistivity of all previously deposited VO_x thin films with |TCR| > 5 %/K (Figure 4-1) [79]. In this work, thin films with |TCR| as high as −5%/K were deposited having resistivity as low as 10,000 Ω–cm [79]. The obtained TCR and resistivity values made these films potential candidates for use in constant bias bolometer systems. However, this work did not contain any information on the inherent 1/f noise properties of these thin films.
Another material system, consisting of amorphous silicon germanium alloys ($\text{Si}_{1-x}\text{Ge}_x\text{:H}$) has also been investigated for use in constant bias bolometer systems. The motivation for its use stems from a considerable research effort in the development of thin films of amorphous hydrogenated silicon ($\text{a-Si:H}$) for use in large area devices such as solar cells and as transistors in liquid crystal displays and for use as sensing material for infrared detection [73] [80] [50]. An analogous material system consisting of amorphous hydrogenated germanium ($\text{a-Ge:H}$) was also investigated for use in those applications [51] [52] [53] [54] [55], however, these thin films could not compete with the performance of $\text{Si:H}/\text{SiGe:H}$ systems for the stated applications. While $\text{Si:H}$ and $\text{SiGe:H}$ have a dark resistivity between $10^{11} - 10^{13}$ $\Omega$–cm with light resistivities as low as $10^{4}$ $\Omega$–cm, $\text{Ge:H}$ thin films show smaller dark resistivity of $10^{7}$ $\Omega$–cm with smaller thermal activation [80] [52] [53] [54] [55]. However, the lower resistivity of $\text{Ge:H}$ thin films make them good candidates for further investigation as infrared imaging materials.

Figure 4-1. Deposition rate, resistivity and TCR of thin films of $\text{VO}_x$ deposited by Biased Target Ion Beam Deposition as a function of partial pressure of $\text{O}_2$ as it appears in ref. [79].
Further improvement in the resistivity – TCR tradeoff was necessary, even at these levels. In addition to the high Johnson noise imposed by high resistivity, these films have fairly high 1/f noise with the ‘α/n’ values around $10^{-18}$ cm$^3$ [51].

It was hypothesized that in the case of Ge:H thin films a reduction in the resistivity was possible by incorporation of nanocrystallites. Saint-John found that for PECVD deposited Ge:H, nanocrystallites can nucleate as early as 10–15 nm for a dilution ration ([H$_2$]/[GeH$_4$]) of 400 [74]. Furthermore, it was suggested that the nucleation density of germanium crystallites can be increased by lowering the substrate temperature from the previously investigated value of 200 °C to 170 °C [74]. Indeed it was seen that by incorporating a small fraction of nanocrystallites, thin films with resistivity of around 1,500 Ω–cm were deposited having a |TCR| value of 3.7 %/K [57].

To further the development of high TCR materials, this work investigates high TCR VO$_x$ and Ge:H thin films for use in constant bias systems. For thin films of VO$_x$, normalized Hooge’s parameter values are measured. To improve the electrical properties of Ge:H, in this work thin films of Ge:H have been deposited at a very high dilution ratio of GeH$_4$ in H$_2$ to incorporate germanium nanocrystallites. Electrical properties of mixed phase amorphous + nanocrystalline thin films are investigated by varying the crystal fraction of germanium; this was achieved by changing the total thicknesses of the deposited thin films.

4.2. High TCR VO$_x$ Thin Films

4.2.1. Experimental Details

High resistivity VO$_x$ thin films were deposited using a Biased Target Ion Beam Deposition System (BTIBD), the details of which were described in the previous chapter. Thin films ~ 85 nm thick were deposited having a thickness uniformity >±3% for a 4” wafer. A three-target configuration was used to deposit the thin films. A series of films was deposited as a function of oxygen partial pressure, pO$_2$, ranging from $1 \times 10^6$ Torr to $2.1 \times 10^6$ Torr, corresponding to oxygen flow 5 % – 8.5% of argon. Detailed
values for the parameters used can be found in Table 2. The total flow was kept at 70 sccm, corresponding to a deposition pressure of \(\approx 5.8 \times 10^{-4}\) Torr. A pulsed waveform was used to increase the sputter yield and prevent arcing of the target. The sputtering voltage was kept at \(-800\) V while the duty cycle was set to 99%, for all three targets yielding a film growth rate of \(\approx 0.5\) Å/second.

Thin films were patterned using lithographic processing steps described in Section 3.3.7. The thickness of the deposited VO\(_x\) and Ge:H thin films was measured using an AFM, Dimension Icon made by (Bruker Corporation) on a lithographically patterned step in the thin films. The roughness of the thin films was evaluated using the same AFM. The crystal structure of the thin films was evaluated by grazing incidence XRD using a PANalytical’s X’Pert Pro MPD. The composition of the VO\(_x\) films was evaluated by Rutherford back scattering (RBS) using a 2.275 MeV He\(^{2+}\) ion beam with the detector set at a backscattering angle of 160° [81].

4.2.2. Results and Discussion

Thin films of VO\(_x\) were deposited by BTIBD as a function of pO\(_2\), from \(1 \times 10^{-6}\) Torr to \(2.1 \times 10^{-6}\) Torr. The films were found to be 85 nm thick. All the thin films deposited were found to be amorphous and a characteristic plot of the grazing incidence x-ray data is shown in Figure 4-2. The angle of incidence for the measurement was 0.8°. The x-value in ‘VO\(_x\)’ (obtained from the RBS analysis) increased linearly from 2.2 to 2.4 as the partial pressure was increased from \(1 \times 10^{-6}\) Torr to \(2.1 \times 10^{-6}\) Torr.
The samples show a temperature dependent resistivity which is dominated by thermal activation at least in the range of 300 K – 350 K. The magnitude of TCR was calculated by extracting the thermal activation energy ($E_a$) from material’s resistivity-temperature plots.

The room temperature TCR is given by:

$$ TCR = -\frac{E_a}{k_bT^2} $$  \hspace{1cm} \text{Equation 24} 

Where $E_a$ is the extracted activation energy, $k_b$ is the Boltzmann’s constant in eV and $T$ is room temperature or 300 K.

The 1/f noise of the thin film samples was evaluated using Hooge-Vandamme relation given by [28] [82]:

Figure 4-2. Grazing incidence X-ray diffraction spectra associated with high resistivity VO$_x$ thin films deposited at high partial pressure of oxygen using Biased Target Ion Beam Deposition showing amorphous thin film structure.
\[ \frac{S_I(f)}{I_{bias}^2} = \frac{\alpha_H}{nVf} \]

where \( S_I(f) \) is the spectral current density, \( I_{bias} \) is the sample current, \( V \) is the volume of the sample, \( f \) is frequency range of measurement, \( \alpha_H \) is Hooge’s parameter and \( n \) is the carrier concentration. Since the volume and the bandwidth of measurement are chosen on a system level, the parameter ‘\( \alpha_H/n \)’ (normalized Hooge’s parameter) was used as a measure of evaluating the 1/f noise of the samples.

To extract the normalized Hooge’s parameter, samples having different volumes were lithographically patterned and biased using a voltage source in a shielded iron box. A low noise current amplifier (Stanford Research Model –570) was used to amplify the measured current power spectral density (PSD) which was recorded using a National Instruments data acquisition card (NI-6036E). The recorded PSD was converted into the frequency domain by using the Fast Fourier Transform function in LabVIEW. When normalized for volume, the three curves overlap as predicted by the Hooge-Vandamme relation. Similarly, the bias dependence of noise was also confirmed by applying three distinct biases. The measured power spectral density at 1 Hz (also referred to as the k-value) was multiplied by the sample volume to determine the normalized Hooge’s parameter, or ‘\( \alpha/n \)’ value of our thin film.

Figure 4-3 shows the resistivity, TCR and \( \alpha_H/n \) (1/f noise) of the deposited films as a function of \( pO_2 \). As expected, the resistivity of the deposited films increases as a function of \( pO_2 \), reaching 20,000 Ω-cm at an oxygen partial pressure of \( 2.1 \times 10^{-6} \) Torr. The TCR and the characteristic 1/f noise of the films followed no particular trend. The |TCR| was a maximum of 5%/K for the film deposited with a \( pO_2 \) of \( 1 \times 10^{-6} \) Torr while the \( \alpha_H/n \) was found to be the lowest for the film deposited at \( 1.79 \times 10^{-6} \) Torr with a value of \( 1 \times 10^{-20} \) cm³. In other material systems, the inherent noise of the amorphous semiconductor has been associated with the short-range order in the amorphous material [83].
Figure 4-3. Resistivity, temperature coefficient of resistance (TCR) and the normalized Hooge’s parameter (α_H/n) of VO_x thin films deposited by BTIBD as a function of partial pressure of oxygen (pO_2).

Regardless of the deposition conditions, the lowest resistivity deposited for the high TCR material was > 10,000 Ω·cm. This limitation put the high resistivity VO_x material system at a disadvantage when compared with Si:H thin films even for use in constant bias readout integrated circuit bolometer cameras. Although this resistivity is higher than the currently used a–Si:H thin films for constant bias bolometer devices, their α_H/n values are on the order of those of Si:H thin films. A reduction in the total device resistance might make these thin films competitive for use in constant bias bolometer devices.

None the less, the magnitude of TCR, noise and resistivity observed for some of the thin films of VO_x of this work are better than other materials with high TCR such as spinels based on transition metal oxides and hydrogenated silicon as manufactured by L–3 communications [18] and ULIS [84].
4.3. High TCR Mixed Phase Thin Films of Ge:H

4.3.1. Experimental Details

A series of mixed phase amorphous + nanocrystalline hydrogenated germanium ((a+nc)-Ge:H) thin films having different thicknesses (thickness = 7 nm – 200 nm) were deposited onto c-Si wafers coated with 20 nm of silicon nitride by RF (f= 13.56 MHz) plasma enhanced chemical vapor deposition (PECVD) using ultra-high purity germane (GeH₄) gas diluted with H₂. A number of deposition parameters were fixed along the lines of previous work including: a total pressure of ∼ 93 Pa and a low plasma power density of 0.08 W/cm². The background pressure was typically < 5 × 10⁻⁵ Pa. To facilitate an earlier nucleation of crystallites, the substrate was held at a temperature of 170°C and the hydrogen-to-reactive gas dilution ratio was maintained at R = [H₂]/[GeH₄] = [160 sccm]/[0.4 sccm] = 400.

To extract the electrical properties such as resistivity and TCR, the deposited thin films of Ge:H were patterned by photolithography and wet etching in a 30% H₂O₂ solution. Top contacts were made to the patterned thin films by photolithography and lift-off of sputter deposited chromium. The thickness of deposited Ge:H thin films were measured using an AFM, Dimension Icon made by (Bruker Corporation) on lithographically patterned steps in the thin films. The contact quality and thin film resistivity was evaluated using transmission line measurements on these lithographically patterned samples. As expected, the samples show a temperature dependent resistivity which is dominated by thermal activation at least in the range of 300 K – 350 K. The magnitude of TCR was calculated by extracting the thermal activation energy (E_a) as described in the previous sub-section.

4.3.2. Results and Discussion

Figure 4-4 shows the measured resistivity and TCR of the samples as a function of thickness. Starting with the 7 nm thin film, the magnitude of TCR increased from 1.8%/K to a value of about 6.5%/K for the 100 nm thick film. The 200 nm film showed a decrease in |TCR| from the 100 nm film to about 3.6%/K. The resistivity of these films was between 750 Ω–cm and 4,500 Ω-cm. Of particular interest are the films of thicknesses 50 nm and 100 nm which show a |TCR| of 5%/K and 6.5 %/K for a
resistivity of about 1,500 Ω-cm and 2,250 Ω-cm respectively. To verify reproducibility of results, the 100 nm thin film was re-deposited with similar results, as can be seen in the graph.

The 1/f noise of the thin film samples was evaluated using the Hooge-Vandamme relation as described in the previous sub-section. Figure 4-5 shows the measured power spectral density (PSD) of a 100 nm film of Ge:H deposited in this work, confirming the volume dependence in these thin films. The y–intercept at a frequency of 1 Hz can then be used to extract $\alpha_{1f}/n$ value of that thin film.
Figure 4-5. The measured Power Spectral Density (PSD), $S_f^2/I_{\text{bias}}^2$, for the 100 nm sample of Ge:H showing volume dependence as predicted by the Hooge-Vandamme relation.

Figure 4-6 shows the extracted $\alpha_{\text{H}}/n$ values for the thin films of this work as a function of thickness. The 1/f noise for the 7 nm thick film could not be measured accurately. One possible reason for this high drift might be a resistance variation due to Joule heating of the thin film having a small cross-section for current flow (due to extremely small thickness). These values were found to be between $10^{18}$ cm$^3$ (25 nm) and $10^{20}$ cm$^3$ (100 nm) for the films. Interestingly, the film having the largest TCR value was also found to have the lowest $\alpha_{\text{H}}/n$ value, while the film with the lowest TCR was also found to have the highest $\alpha_{\text{H}}/n$ value.
Figure 4-6. Extracted normalized Hooge’s parameter ‘α_H/n’ as a function of thickness for thin films of Ge:H each annotated with their corresponding value of TCR. Note that the 100 nm thin film had the lowest α/n value while having the highest TCR.

In an effort to explain the combination of high TCR and low 1/f noise in thin films of PECVD deposited Ge:H, the thin film microstructure was characterized. The crystal structure of the thin films was evaluated by grazing incidence X–ray diffraction using PANalytical’s X’Pert Pro MPD with a step size of 0.026 degrees at a rate of 1.2 degrees/minute. Figure 4-7 shows the x-ray diffraction spectra for the 200 nm thin film grown on thermally oxidized silicon wafers. The data shows the presence of diamond cubic structure as expected from high dilution ratio during PECVD.
To extract a first order estimate of the crystal size of the grains, the diffraction pattern for the 200 nm thick film was also collected in the Bragg-Brentano geometry, as shown in Figure 4-8. The first order grain size of the crystallites present in the films can be obtained using Scherrer’s formula [85]:

$$D_p = \frac{0.94 \lambda}{\beta_{1/2} \cos \theta}$$  \hspace{1cm} \text{Equation 26}$$

where $D_p$ is the calculated grain size, $\lambda$ is the wavelength of radiation used for diffraction (1.54 Å); $\beta_{1/2}$ is the angle at full width half maximum. Using the Scherrer’s formula, the grain size for this film was calculated to be about 140 nm.
An even thicker sample (250 nm) was deposited on a native oxide coated c-Si wafer for TEM analyses (albeit at a slightly higher temperature of 200 °C as opposed to 170 °C). Figure 4-9 shows a dark field TEM micrograph collected by Dr. David Saint John from the sample which shows that initial layers of the Ge:H are amorphous [19].

![Graph showing Bragg-Brentano x-ray diffraction pattern of the 200 nm thin film of Ge:H used to extract the size of crystals to the first order using the Scherrer's formula.]

**Figure 4-8.** Bragg-Brentano x-ray diffraction pattern of the 200 nm thin film of Ge:H used to extract the size of crystals to the first order using the Scherrer’s formula.

Around a bulk thickness of 10–15 nm, nanocrystallites start to nucleate and continue to grow segregated in an amorphous matrix until a thickness of around 100 –120 nm when these grains coalesce. In addition, the planar twinning defects with a regular periodicity of ~ 1nm were also observed in this thin film.
Figure 4-9. Dark field TEM micrograph of a 250 nm thin film of Ge:H showing the presence of nanocrystallites in an amorphous matrix [published previously in ref. [19].

The roughness of the thin films as a function of thickness was evaluated using an AFM. Figure 4-10 shows AFM micrographs of thin film of Ge:H with a thickness of 7 nm and 200 nm. Figure 4-10a shows the 7 nm thin film having a peak-to-valley height of 7 nm having small island sizes. In contrast Figure 4-10b shows the 200 nm film has a much larger grains (> 50 nm).

Figure 4-12 illustrates the surface roughness evolution (both RMS and peak-to-valley) of the thin films of varying thicknesses deposited in this work. The RMS roughness of the films < 25 nm thick is around 8 Å – 9 Å. The 100 nm thin film shows the highest roughness with an RMS value of 2.7 nm and a peak-to-valley height of 15 nm. Such a texture evolution is typical of growing crystallites and has been noted in Ge:H and Si:H/SiGe:H material system by numerous authors [15] [86] [72] [87]. For a film thickness of 200 nm, the RMS roughness decreases to about 2 nm (peak-to-valley height of 12 nm).
Figure 4-10. AFM micrographs of Ge:H thin films with thicknesses a) 7 nm and b) 200 nm presenting differences in surface morphology. The 200 nm thin film shows larger grains and larger peak-to-valley heights than the 7 nm thin film.

Figure 4-11 shows a detailed three-dimensional image obtained for the 100 nm thin film of Ge:H. Clusters of Ge:H can be seen protruding from the surface of the film which results in the larger peak-to-valley height of 15 nm as measured for this thin film.
Figure 4-11. A three dimensional AFM micrograph showing the peak-to-valley roughness associated with the large grain sizes for the 100 nm thin film of Ge:H deposited in this work.

The presence of nanocrystallites in films thicker than 20 nm is confirmed in the electrical resistivity data. While amorphous germanium has been observed to have a resistivity as high as $10^7$ Ω·cm, crystalline germanium has a resistivity of about 50 Ω·cm. The incorporation of these nanocrystallites lowers the resistivity of the films to about 750 Ω·cm for the films in which the crystalline grains have coalesced. The increase in the resistivity and TCR for increasing film thicknesses is unexpected especially because the fraction of nanocrystallites is increasing with increasing thickness. To shed light on this behavior, the thin films were characterized using Real Time Spectroscopic Ellipsometry or RTSE.
RTSE data was analyzed by a global $\Sigma\sigma$-minimization procedure to obtain optical properties, in the form of the complex dielectric function spectra $\varepsilon = \varepsilon_1 + i\varepsilon_2$, and the time dependence of the bulk layer ($d_b$) and surface roughness ($d_s$) thicknesses while the film remains in the amorphous growth regime [88]. After crystallite nucleation occurs, virtual interface analysis (VIA) is used to obtain $\varepsilon$ for the nanocrystalline phase and the time dependence of $d_s$ and the nanocrystallite fraction ($f_{nc}$) [89] [90] [91]. In the mixed-phase amorphous + nanocrystalline (a + nc) regime, VIA uses a Bruggeman effective medium approximation [92] consisting of variable fractions of $\varepsilon$ for a-Ge:H and nc-Ge:H to represent the optical response of the outermost 1.5 nm of the growing film. From this information, a depth profile in crystallinity can be determined.
Figure 4-13. Surface roughness and nanocrystallite fraction for Ge:H on native oxide coated crystalline silicon (c-Si) obtained from two layer modeling and virtual interface analysis (VIA) of real time spectroscopic ellipsometry (RTSE) measurements. The nanocrystallite fraction profile used in the analysis of the single final set of spectra is also shown.

The RTSE data analysis was led by Dr. Nikolas Podraza from The University of Toledo, Toledo, Ohio. The results of RTSE data analysis are provided in Figure 4-13. The film remains amorphous with $d_s$ initially decreasing and reaching a minimum value prior to the amorphous-to-mixed-phase transition [$a \rightarrow (a + nc)$] at $d_b = 13$ nm. Afterward, $f_{nc}$ and $d_s$ begin to increase as nanocrystallites grow preferentially over the surrounding amorphous phase. Coalescence of nanocrystalline clusters occurs when $f_{nc} = 1$, $d_s$ reaches a maximum, and is called the mixed-phase-to-single-phase nanocrystalline transition [(a + nc) $\rightarrow$ nc] at $d_b = 100$ nm. After the (a + nc) $\rightarrow$ nc transition, $d_s$ decreases slightly as a single phase nanocrystalline layer is formed and protruding clusters coalesce.

The initial decrease in resistivity could be due to the initial nucleation of crystallites or enhanced ordering of protocrystalline Ge:H prior to the onset of crystallinity. Resistivity, however, subsequently
increase with \( f_{nc} \), reaches a maximum near the \((a + nc) \rightarrow nc\) transition, then decreases. An explanation for this behavior is that while the initial appearance of more conductive crystallites reduces film resistivity, subsequent evolution introduces a significant amount of grain boundary material into the film. Poorly passivated grain boundaries can act as oxidation pathways post-deposition as the films are exposed to atmosphere, thereby increasing resistivity.

Figure 4-14 shows variations in \( \varepsilon \) for the nanocrystalline component of the nominally 100 nm film deposited on native oxide coated c-Si, monitored by RTSE, and characterized by ex situ spectroscopic ellipsometry post-deposition. RTSE VIA analysis yielded \( a \rightarrow (a + nc) \) and \((a + nc) \rightarrow nc\) transition thicknesses as well as \( \varepsilon \) for a-Ge:H and nc-Ge:H in vacuum at the deposition temperature. The final RTSE data point was analyzed using these reference \( \varepsilon \) and a structural model incorporating a 13 nm continuous amorphous layer, a 93 nm mixed-phase layer with nanocrystallite fraction incorporating an exponential gradient shown in Figure 4-13, a 15 nm continuous nanocrystalline layer, and a 12 nm surface roughness layer in accord with VIA results. These structural parameters were fixed and used to fit ex situ ellipsometric spectra collected from the same sample after exposure to atmosphere at room temperature. The temperature dependence of \( \varepsilon \) for c-Si is known [93] and that for a-Ge:H was obtained from in situ spectroscopic ellipsometric monitoring of a 60 nm thick film prepared at \( T = 200 \, ^\circ\text{C} \) and \( R = 120 \) which remains amorphous.

Spectra in \( \varepsilon \) for a-Ge:H can be described using the Cody-Lorentz oscillator model [94] in terms of energy independent parameters including an amplitude \( (A) \), broadening \( (\Gamma) \), resonance energy \( (E_0) \), band gap \( (E_g) \), partition energy \( (E_p) \), and constant additive term to \( \varepsilon_1 \) \( (\varepsilon_\infty = 1) \). The temperature dependence of \( E_g, E_p, \) and \( A \) were identified as \( dE_g/dT = -0.000411 \, \text{eV/K} \), \( dE_p/dT = -0.000617 \, \text{eV/K} \), and \( dA/dT = -0.02 \, \text{eV/K} \), respectively, which are in reasonable agreement with previous results for hydrogenated amorphous silicon germanium alloys [95]. Variations in \( \Gamma \) and \( E_0 \) were not greater than the error limits within this temperature range and were fixed. Spectra in \( \varepsilon \) obtained from RTSE for \( R = 400 \) a-Ge:H prior to
nanocrystallite nucleation was parameterized using the Cody-Lorentz model at the deposition temperature, and room temperature $\varepsilon$ was simulated using these temperature dependencies to assist in fitting ex situ ellipsometric measurements of nominally 100 and 200 nm thick films. Spectra in $\varepsilon$ for nc-Ge:H were parameterized using three Tauc-Lorentz oscillators [96] [97].

**Figure 4-14.** Complex dielectric function, $\varepsilon = \varepsilon_1 + i\varepsilon_2$, spectra for nanocrystalline Ge:H (nc–Ge:H) obtained in situ, in vacuum from virtual interface analysis of RTSE data and obtained ex situ, after atmospheric exposure.

Differences in $\varepsilon$ for nc-Ge:H are expected with differences in temperature, although typically critical point broadening decreases and amplitude increases at lower temperatures for crystalline materials [93]. The data shown in Figure 4-14 shows an increase in broadening and decrease in amplitude, particularly for the higher energy critical point feature. This variation is consistent with partial oxidation of a material. When $\varepsilon$ obtained at room temperature after atmospheric exposure is treated as a pseudodielectric function and fit to a structural model consisting of un-oxidized nc-Ge:H, in this case $\varepsilon$ obtained in vacuum, and an overlayer of germanium oxide [98], the overlayer thickness is 1.5 nm thick.
This model is a gross simplification, but it also seems to indicate that some fraction of the nc-Ge:H has oxidized.

Figure 4-15. Spectra in $\varepsilon$ for nc-Ge:H obtained ex situ, after atmospheric exposure for nominally 100 and 200 nm thick films.

Figure 4-15 shows a comparison of $\varepsilon$ obtained at room temperature, after atmospheric exposure for the nominally 100 nm film on native oxide coated c-Si and nominally 200 nm film on SiN$_x$ coated c-Si. Although the substrate is different, previous studies indicate that the $a \rightarrow (a + nc)$ transition occurs as at similar thicknesses for Ge:H, as given in Figure 4-15. The features in $\varepsilon$ for the thicker film are sharper and have a larger magnitude, indicating that grain size has increased, the amount of grain boundary material has been reduced, and/or a smaller fraction of the film has oxidized.

A proposed model describing the variations in resistivity and TCR relies on nanocrystalline grain boundaries acting as oxidation pathways. At low crystalline content as in the nominally 25 nm thick film, improved ordering in the amorphous phase may lower film resistivity. When crystallites begin to occupy a significant fraction of the surface, as in the 50 and 100 nm films, resistivity increases as the grain
boundaries enable the internal crystallite surfaces to oxidize. At greater thickness, the increase in grain size and reduction in grain boundaries again reduce resistivity since more interconnecting pathways between un-oxidized grains exist. The highest TCR is observed for the highest resistivity, which potentially consists of crystallites encompassed by higher resistivity germanium oxide material. This structure is similar to that of microbolometer device grade vanadium oxide, where low resistivity, small defective face centered cubic vanadium monoxide nanocrystallites are surrounded by higher oxygen content, higher resistivity material [43]. Similar vanadium oxide thin films also exhibit significant post-deposition oxidation as monitored by RTSE [99].

4.4. Conclusion

Thin films of VOₓ and Ge:H having TCR values larger than 4%/K have been deposited in this work. Thin films of VOₓ were found to be amorphous, with resistivity around 10,000 Ω–cm to 21,000 Ω–cm with α_H/n values between ~ 10⁻¹⁸ and 10⁻²⁰ cm³. Ge:H thin films were found to be amorphous or amorphous + nanocrystalline depending on thickness and dilution ratio of the film deposited. When deposited by PECVD using high dilutions of GeH₄ in H₂, these films show nanocrystalline grain growth in an amorphous matrix. At thicknesses optimized for a dilution ratio, R of [H₂]/[GeH₄], films 50 – 100 nm thick exhibit large TCR (5%/K – 6.4%/K) with reasonable values of α/n (~ 10⁻¹⁹ and 10⁻²⁰ cm³) having low resistivity (1,500 Ω–cm and 2,250 Ω–cm). This superior combination of electrical properties was attributed to the incorporation of an optimal value of grain size and the crystal fraction of the germanium nanocrystallites.

Figure 4-16 shows a comparison of TCR versus resistivity of various thin films reported in the literature in comparison with thin films deposited in this work. Both Ge:H and VOₓ thin films show the highest reported TCR for the resistivity < 10,000 Ω–cm. However, thin films of (nc+a)-Ge:H show a superior resistivity to TCR tradeoff in comparison with thin films of VOₓ deposited in this work.
However a more comprehensive figure of merit is the NETD which was discussed in Chapter 2. For a given material, the theoretical NETD is given by:

\[
NETD \propto \frac{I_{\text{noise, total}}}{I_{\text{bias}} \times TCR}
\]

Or,

\[
NETD \propto \sqrt{\left(\frac{\alpha_H}{nVf}\right)} \times \frac{1}{TCR}
\]

Equation 27

Equation 28
From equation 2 it is evident that a metric of comparison between different materials is the ratio of \( \frac{\alpha_H}{n} \) to TCR\(^2\). Figure 4-17 gives a comparison of the normalized Hooge’s parameter ‘\( \frac{\alpha_H}{n} \)’ versus TCR for different materials reported in the literature with the materials deposited in this work.

![Figure 4-17](image.png)

**Figure 4-17.** A comparison of the Normalized Hooge’s parameter of some common materials reported in literature vs. thin films deposited in this work. The dotted line represents a constant ratio of \( \frac{\alpha_H}{n} : \text{TCR}^2 \).

The dotted line is a curve of an equipotential NETD\(^2\). This curve shows the values which would give the same NETD regardless of the fact that the TCR and noise values are changing between the thin films. This line shows that although films with higher TCR are deposited in this work, the tradeoff of TCR and noise is not sufficient for use of these high TCR materials in a pulsed bias geometry as with the VO\(_x\) microbolometer system. The real advantage in using these thin films can be obtained by advanced circuits such as those used in constant bias bolometer devices existing at L-3 communications. Both the
hydrogenated nanocrystalline + amorphous (nc+a)–Ge:H thin films and possibly VOₓ thin films are potential candidates for high TCR materials used in constant bias microbolometers.
Chapter 5. High TCR Bolometers Using Vertically Integrated Thin Film Resistors

5.1. Introduction

Vanadium oxide (VO$_x$) and hydrogenated silicon germanium (SiGe:H) having a magnitude of TCR between 2%/K and 4%/K form an integral part of a majority of infrared imaging systems manufactured today [8] [30] [78] [100] [18]. The material properties of these thin films used in making uncooled infrared focal plane arrays has remained largely unchanged since some of the first investigations detailing their properties [8] [100] [18]. Most of the performance improvement in these devices has come from the evolution of the underlying read out circuits [101] [59] [102] [103] [104].

The current pixel structure in infrared imaging sensors employs a lateral resistor configuration with a square geometry in which the length and width of the sensing material is 17 μm × 17 μm. To minimize thermal mass and optimize performance, the thickness is kept to ≈ 50nm to 100 nm. Though high TCR materials are preferred, these materials typically have higher resistivities and therefore higher device resistance and noise associated with them. Existing materials with large values of TCR (~ 4%/K) typically have a resistivity larger than 2,000 Ω–cm with higher TCR materials having even larger resistivities. Although higher TCR materials exist, they have not been incorporated into infrared focal plane arrays due to their higher resistivity (> 100 MΩ) and higher noise in the lateral resistor configuration [50] [42] [51] [105] [106] [84].

To overcome this limitation, in this work, an alternate pixel design is investigated in which a high TCR material is incorporated into a structure having low device resistance. In the literature, only a few other device configurations have been explored which use the out of plane conductivity (or through film) as opposed to the lateral conductivity currently used in resistive bolometers. Figure 5-1 shows the three bolometer pixel designs explored by Unewisse et al. which are detailed in ref. [107]. These through film devices can have resistance values up to $10^5$ smaller for the sandwich–gap type structures [107] [108].
Figure 5-1. The three bolometer design structures explored by Unewisse et al. in ref. [107].

where $\rho$ is the resistivity of the semiconductor material, $g$ is the gap length, $d$ is the thickness of the detector, $w$ is the width of the detector, and $l_1$ and $l_2$ are the lengths of the bottom contact in the sandwich and the sandwich-gap type detectors, respectively.

Figure 5-1(a) shows the standard device structure used by the resistive microbolometer industry, whereas the detector of Figure 5-1(b) shows the sandwich type configuration in which the current flows in the transverse direction [107]. This greatly reduces the length to area ratio of the detector and hence the resistance. Due to the change in the length over area ratio associated with the through-film configuration, the resistance of a bolometer can be lowered ($R_{\text{in-plane}}/R_{\text{out-of-plane}}$) by up to a factor of $10^4$ for 10 $\mu$m $\times$ 10 $\mu$m pixel dimensions in a sandwich type configuration [107]. Another type of detector was named the Sandwich-gap type detector, as shown in Fig. 2(c), which essentially consists of two sandwich type elements in series, separated by a small gap [107]. In this configuration, the resistance could be reduced up to a factor of $10^5$ [107]. Although these alternate designs were investigated at the Defense Science and Technology Organisation, in Australia from 1995 through 2003, there is a limited body of literature detailing the performance of microbolometers in this configuration [107].

In 2010, Moreno et al. investigated the electrical performance of the sandwich type structure and detailed it in ref. [108]. It was found that the sandwich structures had a higher level of electrical noise (5
orders of magnitude higher, $10^{-11} \text{A}/\sqrt{\text{Hz}}$ vs. $10^{-16} \text{A}/\sqrt{\text{Hz}}$) [108]. However, no reason was attributed to this higher level of noise. One possibility for the higher level of noise could be the higher electric field to which the sensing thin films were subjected. Indeed the ‘$I$–$V$’ curves of the biased sandwich structures was found to be nonlinear [108].

This work looks to fabricate sandwich type detector elements (referred to here as the through film device structures) with high TCR and high resistivity materials. The resultant through films structures are characterized for their electrical properties, especially their electrical noise. Thin films of high TCR hydrogenated germanium (Ge:H, |TCR| > 6%/K) and vanadium oxide (VO$_x$, TCR > 5%/K) were integrated in lateral and through film configuration. The Johnson noise of the devices was reduced significantly while maintaining the same contribution of $1/f$ noise.

5.2. Theoretical Performance of Through Film Structures

An important figure of merit is the Noise Equivalent Temperature Difference or NETD, which can be used to compare the theoretical performance of the through film structures. The NETD is the measure of the sensitivity of an IR imaging system. It is defined as, “….the change in the temperature of a black body of infinite lateral extent which, when viewed by the thermal imaging system, causes a change in the signal-to-noise ratio of unity in the electrical output of the pixels of a focal plane array….” [17]. The NETD of a detector is given by [30] [17]:

\[
\text{NETD} \propto \frac{I_{\text{noise, total}}}{I_{\text{bias}} \times TCR}
\]

Where $I_{\text{noise, total}}$ is the resultant total electrical noise from an electrical bias $I_{\text{bias}}$. The ratio of $I_{\text{bias}}/I_{\text{noise, total}}$ is also known as the Signal to Noise Ratio or ‘SNR’.

When a microbolometer, dominated by the material noise, is biased using a voltage source, $V_{\text{bias}}$, producing a current $I_{\text{bias}}$ the obtained SNR is given by [30]:
SNR = \frac{I_{bias}}{\sqrt{4\kappa T(f_2 - f_1) + \frac{I_{bias}^2}{R_b} \frac{\alpha_H}{nV} \ln \left( \frac{f_2}{f_1} \right)}} \quad \text{Equation 30}

where \(\kappa\) is the Boltzmann’s constant, \(T\) is the temperature of operation, \(f_1\) and \(f_2\) is the range of frequency for the bandwidth of operation, \(R_b\) is the electrical resistance of the bolometer, \(\alpha_H\) is Hooge’s parameter, \(n\) is the carrier concentration and \(V\) is the volume of the sample.

From Equation 29 and Equation 30, the NETD can be written as:

\[
\text{NETD} \propto \frac{1}{SNR \times TCR}
\]

To compare the electrical performance of the lateral and through film configurations, their TCR and the SNR must be evaluated. While the TCR of a device is an inherent property of the particular device and depends on the activation energy of the material used, the SNR depends on the electrical bias, applied to the device.

From Equation 30, the SNR increases for increasing bias values and reach a maximum when the device is operated at a bias level such that the 1/f noise dominates the total noise contribution. On the other hand the power dissipated across the thin film (\(V_{bias}I_{bias}\)) increases with increasing bias which may cause Joule heating of the thin film.

Due to these conflicting factors, a new set of bias conditions must be considered for this new configuration. Figure 5-2 shows calculated values of the power dissipated and the theoretical maximum values of SNR which can be achieved for the through film configuration with the following values:
Table 3. Parameters and their values used for estimating the SNR ratio and the power dissipated as a function of applied bias.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\kappa_b$</td>
<td>$1.38 \times 10^{-23}$ m$^2$.kg.s$^{-2}$.K$^{-1}$</td>
</tr>
<tr>
<td>$T$</td>
<td>300 K</td>
</tr>
<tr>
<td>$f_1$</td>
<td>0.001 Hz</td>
</tr>
<tr>
<td>$f_2$</td>
<td>1 KHz</td>
</tr>
<tr>
<td>$\rho$</td>
<td>20,000 $\Omega$.cm</td>
</tr>
<tr>
<td>$\alpha_{\mu}$/nV (k)</td>
<td>$1 \times 10^{11}$</td>
</tr>
<tr>
<td>Dimensions</td>
<td>17 $\mu$m $\times$ 17 $\mu$m</td>
</tr>
</tbody>
</table>

A large voltage bias, similar to the one in the lateral configuration produces a bias current value $10^4$ times larger than in the lateral configuration, causing a significant heating of the bolometer material leading to low frequency drift of the signal current [77].

The increase in the temperature of the bolometer due to the bias current is given by $\Delta T$ [30]:

$$\Delta T = \frac{V_{bias}I_{bias}}{c}$$  \hspace{1cm} \text{Equation 32}

where $\Delta T$ is the rise in temperature of the bolometer per second of applied bias, and $c$ is the heat capacitance of the bolometer material [30].
Figure 5-2. Graph showing theoretically calculated values of the power dissipated (red) and the maximum attainable signal to noise ratio (black) for a thin film device with a resistivity of 20,000 $\Omega\cdot$cm and dimensions of 17 $\mu$m $\times$ 17$\mu$m.

While a large bias voltage may cause bias–induced heating, a low voltage bias may result in a low SNR (from Equation 30). Figure 5-2 shows that the SNR increases dramatically for low bias values; which is also when the total noise is dominated by the Johnson noise of the bolometer. The SNR reached a maximum when the 1/f noise contribution in equation 1 dominates. Thus it can be concluded that for the vertically integrated thin films the optimal bias level is the lowest voltage value for which the SNR has reached a maximum OR the bias value should be high enough so that the device performance is dominated by the 1/f noise and not by Johnson noise.

5.3. Experimental Details

To compare the electrical performance of the through film structures to the conventional lateral configuration, both the lateral and through film configurations have been fabricated using thin films of
high resistivity and high TCR VO\textsubscript{x} and Ge:H. The process steps for fabricating the devices are described in the following subsections.

5.3.1. Lithographic Fabrication of Device Structures

Figure 5-3 shows a schematic for the two device structures whose electrical properties were compared. The lateral resistors devices were processed as described in Section 3.3.7. Briefly: thin films having a high TCR were first deposited onto an electrically insulating substrates (100 nm thermally grown SiO\textsubscript{2} on c-Si for VO\textsubscript{x} thin films and 20 nm LPCVD SiN\textsubscript{x} for Ge:H). The TCR material is then isolated in a 1:2,000 H\textsubscript{2}O\textsubscript{2}:H\textsubscript{2}O solution, following which top electrodes are deposited by liftoff (titanium for VO\textsubscript{x} and chromium for Ge:H).

![Diagram](image.png)

Figure 5-3. Cross-sectional schematic of bolometer configurations explored: a) In-plane conductivity (top); b) Out-of-plane conductivity (bottom).
Figure 5-4. Lithographic processing for through film resistance structures.

Figure 5-5. Top view of a mask set showing the different layers used for fabricating the out of plane resistance structure.

The processing of through-film structures was performed by using a mask set as shown in Figure 5-5. The processing was done following the process flow diagram of Figure 5-4 as follows: Bottom
contacts were deposited onto insulating substrates by a liftoff method. 100 nm of chromium was used for bottom contacts to Ge:H, whereas bottom contacts to high TCR VO<sub>x</sub> thin films were obtained using either 100 nm of Titanium or Vanadium. Following the patterning of bottom contacts, 100 nm of SiO<sub>2</sub> was sputtered using magnetron sputtering of a 3–inch silicon target (99.999% pure) with 20% of O<sub>2</sub> flow in argon. A bias of −120 Volts was applied to improve the leakage current density through the sputtered SiO<sub>2</sub>. Following this step, vias were etched using (6:1) BOE solution down to the bottom metal. High TCR VO<sub>x</sub> deposited using Biased Target Ion Beam Sputtering (BTIBD) while Ge:H was deposited using Plasma Enhanced Chemical Vapor Deposition (PECVD) whose deposition will be described in the following subsection.

5.3.2. Deposition of High TCR, High Resistivity VO<sub>x</sub> Thin Films

Previously, it has been shown that high TCR VO<sub>x</sub> thin films can be deposited using a novel deposition technique of Biased Target Ion Beam Deposition (BTIBD) that incorporates precise control of the oxygen content of the thin film using a residual gas analyzer (RGA). The details of this deposition technique have been discussed in a previous chapter. Detailed values for the parameters used can be found in Table 2.

Briefly, a three-target configuration was used to deposit the thin films. A series of films was made with increasing partial pressure of oxygen, pO<sub>2</sub>, ranging from 1×10<sup>-6</sup> to 3×10<sup>-6</sup> Torr, corresponding to an oxygen flow of 3 sccm–5 sccm. Total flow was kept at 70sccm, corresponding to a deposition pressure of ≈5.8×10<sup>-4</sup> Torr. A pulsed waveform was used to increase the sputter yield of the target in the oxidized regime. The target voltage was pulsed between −800V and +5 V to help minimize arcing during reactive sputtering. The voltage was pulsed at 10 KHz with a pulse width of 1 μsec. Thin films were deposited to a thickness of ≈100 nm (at a deposition rate of 0.17 Å/sec) and were found to be highly uniform in thickness and its electrical properties (better than ±3%).
5.3.3. Deposition of High TCR, High Resistivity Ge:H Thin Films

Thin films of Ge:H having thicknesses of 25 nm to 100 nm were deposited by RF (f = 13.56 MHz) plasma enhanced chemical vapor deposition (PECVD) using ultra-high purity germane (GeH₄) gas diluted with H₂. The deposition conditions were similar to the experiment described in Section 3.2. A number of deposition parameters were fixed along the lines of previous work including: a total pressure of ~ 0.7 Torr and a low plasma power density of 0.08 W/cm². The substrate was held at a temperature of 170°C and the hydrogen-to-reactive gas dilution ratio was maintained at \( R = \frac{[\text{H}_2]}{[\text{GeH}_4]} = \frac{[160 \text{ sccm}]}{[0.4 \text{ sccm}]} = 400. \)

5.3.4. Electrical Characterization of Device Performance

The three most important electrical properties for thin film microbolometer materials are: room temperature resistivity, temperature coefficient of resistivity (TCR) and the electrical noise given by the normalized Hooge parameter (\( \alpha_H/n \)). The lateral resistivity of the thin films was extracted using the Transmission Length Method (TLM) patterns. Figure 5-6 shows a graph of resistance versus spacing of electrodes for a thin film of VOₓ deposited using the BTIBD system at a partial pressure pO₂ of 3×10⁻⁶ Torr. The other deposition conditions are described in section 5.3.2. The contact resistance is given by the y–intercept (here 2×10⁷ Ω), the transfer length is given by half the value of the x–intercept (here 2 μm) and the resistivity is given by multiplying the slope of the graph with the cross-sectional area of the thin film (here \( \rho = 21,000 \text{ Ω} \cdot \text{cm} \)). The transfer length is used to decide on the minimum feature size which can be used to extract accurate thin film properties.
Figure 5-6. Resistance vs. electrode spacing measurements used to extract the sensing material's resistivity and the contact resistance.

Since the thickness of thin film is constant, the resistivity of the through film structures could not be measured using the TLM method. Instead, the effective through film resistivity was determined by patterning through film structures having different areas as can be seen in Figure 5-7.

Figure 5-7. Top view of the mask set used for the measurement of resistivity of the sensing material.

I–V measurements were made on each through film resistor VO₃ thin film and is evaluated in Figure 5-6. The resulting resistance values are plotted as a function of ‘1/contact area’ and are shown in
Figure 5-8. The slope of the graph can be used to obtain the ‘effective resistivity’ of the thin film such that:

$$\rho = \frac{1}{\text{thickness}} \times \frac{R_B}{1/\text{Area}} = \frac{\text{Slope}}{\text{Thickness}}$$

Equation 33

Similarly, the contact resistance can be calculated using the x–intercept of the curve. The x–intercept gives a point in which the area of the device interrogated is infinitely large. Such a point would contain only the contact resistance and have a relatively insignificant contribution from the thin film itself.

The TCR measurements were made using a heated stage and a computer controlled HP4140B pA/DC voltage source. The TCR value was extracted from the activation energy after measuring the temperature dependence of resistance at incremental temperatures between 20-60°C using the relation given by:
where $E_a$ is the activation energy of the thin film material, $\kappa_b$ is the Boltzmann’s constant and $T$ is the temperature at which the TCR value is to be calculated.

The $1/f$ noise of the material is evaluated by the Hooge-Vandamme relation given by:

$$
\log \left( \frac{S_I(f)}{I_{bias}^2} \right) = \log \left( \frac{\alpha_n}{n \times V} \right) - \beta \log(f)
$$

Equation 35

where, $S_I(f)$ is the spectral current density, $I_{bias}$ is the sample current, $V$ is the volume of the sample, $f$ is the frequency of measurement, $\alpha_n$ is the Hooge’s parameter, $\beta$ is the slope of $1/f$ noise measurement and $n$ is the carrier concentration in the samples. To evaluate the properties of the films, ‘$\alpha_{H}/n$’ was treated as the property of interest as described previously. The instrumentation for this measurement has also been described in a previous chapter.

5.4. Experimental Results

Since in the through film configuration, the thickness of the thin film is used as the length of the resistor, small electrical biases can result in large electric fields across the thin film. These large electric fields can result in nonlinear I--V characteristics of the through film resistors. The result of which can be seen in Figure 5-9.
To ensure accurate measurements, the through film sensors were biased in the linear region of Figure 5-9. Figure 5-10 shows the lateral and through film resistivities of thin films of Ge:H deposited in this work. The lateral resistors of Ge:H have been investigated in Chapter 4. The lateral resistivity depends on the thickness of the thin film investigated. The through film resistivity varies significantly from its lateral resistivity: from 1,000 Ω·cm for the 200 nm (750 Ω·cm for the lateral configuration) to 28.6 MΩ·cm for the 25 nm thin film (1,250 Ω·cm for the lateral configuration). The disparity in the resistivity between the lateral and the through film configuration suggests that there exists an insulating layer at the interface of the bottom metal and the resistive thin film, or the initial layers of the resistive thin film have an extremely high resistivity.
Figure 5-10. Lateral and through film resistivity of thin films of Ge:H thin films as a function of thickness. The through film resistivity increases dramatically for smaller thicknesses, indicating an insulating interfacial layer between the bottom metal and the resistive thin film.

Figure 5-11 shows a plot of natural log of resistance versus temperature of thin films of VOₓ and Ge:H in the lateral and through–film configurations. For the through film measurements, the resistors were biased by using small electric fields such that the ‘I–V’ data collected were linear. It was found that though the lateral and through film resistance values are orders of magnitude different, the TCR values remained the same. These results are a strong proof of concept for the proposed through film resistor design.
Having measured the resistance and the TCR values of the through film resistors, the 1/f noise of the thin films was evaluated using methods described in Chapter 3. Figure 5-12 shows the acquired Power Spectral Density of thin films of Ge:H, 50-nm-thick with different volumes of the thin film resistors in both lateral and through film configurations. As expected, the 1/f noise increases with decreasing device volume. When normalized for volumes, the magnitude of the 1/f noise was found to be the same as can be seen from the inset of Figure 5-12.
Figure 5-12. The measured Power Spectral Density (PSD), \( \frac{S^2_{II}}{I_{bias}^2} \) for a 50 nm thin film of Ge:H showing good agreement with the Hooge’s relationship, independent of device structure used (lateral or through film). Inset shows the PSD normalized for volume.

Table 4 compares the TCR and ‘\( \alpha_{II}/n' \) of thin films in lateral and through film configuration deposited in this work. Within the margin of error of measurement, the TCR and the ‘\( \alpha_{II}/n' \) of thin films was found to be the same.

Table 4. Summary of TCR and ‘\( \alpha_{II}/n' \) values in lateral and through film configuration deposited in this work.

<table>
<thead>
<tr>
<th>Sample</th>
<th>TCR Lateral (%/K)</th>
<th>TCR Through Film (%/K)</th>
<th>‘( \alpha_{II}/n' ) Lateral (cm(^3))</th>
<th>‘( \alpha_{II}/n' ) Through Film (cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>VO(_x)</td>
<td>-4.1</td>
<td>-4</td>
<td>(1.4 \times 10^{-19})</td>
<td>(1 \times 10^{19})</td>
</tr>
<tr>
<td>VO(_x)</td>
<td>-4.5</td>
<td>-4.6</td>
<td>(1.5 \times 10^{-18})</td>
<td>(1 \times 10^{18})</td>
</tr>
<tr>
<td>VO(_x)</td>
<td>-4.7</td>
<td>-5</td>
<td>(4 \times 10^{-18})</td>
<td>(1 \times 10^{18})</td>
</tr>
<tr>
<td>VO(_x)</td>
<td>-5</td>
<td>-5.3</td>
<td>(3 \times 10^{-18})</td>
<td>(1 \times 10^{18})</td>
</tr>
<tr>
<td>Ge:H</td>
<td>-3.4</td>
<td>-3.6</td>
<td>(7 \times 10^{-19})</td>
<td>(5 \times 10^{19})</td>
</tr>
<tr>
<td>Ge:H</td>
<td>-5</td>
<td>-5.1</td>
<td>(1.7 \times 10^{-19})</td>
<td>(1 \times 10^{19})</td>
</tr>
<tr>
<td>Ge:H</td>
<td>-6.2</td>
<td>-6.5</td>
<td>(8 \times 10^{-20})</td>
<td>(1.2 \times 10^{-20})</td>
</tr>
</tbody>
</table>
Similar values of TCR and $a_{\mu}/n$ shows that in going from the lateral device configuration to a through film configuration there is no change in the signal to noise ratio (SNR). However, as it was noted in section 5.2, the vertically integrated devices can sustain larger signal currents due to their lower device resistance without any change in self heating. This means that although the SNR has not changed with device configuration, the magnitude of the signal is larger. As was seen in Section 2.3.4.2, a higher signal current implies reduced integration time on the readout capacitor, thereby increasing the available frame rate of the sensing array.

5.5. Conclusion

Vertically integrated VO$_x$ thin films structures have been demonstrated as potentially viable alternative to the conventional lateral-pixel configuration used in uncooled imaging bolometers, for next generation IR FPAs that employ higher TCR and higher resistivity material. In this work, high VO$_x$ thin films were obtained using BTIBD and high TCR Ge:H thin films were obtained by PECVD of GeH$_4$ at high dilution ratios.

Using lithographically patterned structures it has been shown that through film conductivity measurements make it possible to reduce the device resistance of high TCR materials. The SNR for the through film structures equal to those obtained using lateral conductivity.
Chapter 6. Resistive Temperature Sensing Arrays

6.1. Introduction

Microbolometer arrays have been at the forefront non-contact thermometry. These devices have high sensitivity (NETD < 30 mK) and high frame rates (> 30 Hz) [15] [18]. However, these systems are based on the principle of differential temperature and are not designed for use in the contact mode or for mapping the absolute temperature of the object under investigation. In addition, these sensors are based on materials with |TCR| < 4 %/K in the lateral configurations where the device impedance is relatively high (>100 MΩ). In this work, thin films of VOₓ with TCR ≈ 4.4 %/K were used to fabricated one dimensional arrays of vertically integrated temperature sensors on glass substrates. The performance of these arrays is compared with those of the lateral resistors. In addition, discrete devices are fabricated with integrated ZnO based transistors to allow multiplexing of two dimensional arrays.

6.2. Array Design and Fabrication

6.2.1. Design and Processing of 1-D VOₓ Sensor Array

Figure 6-2 shows the schematic of the 1×8 1-D temperature sensing array fabricated in this work. The total length of the sensor was kept at 2 cm, while the head of the sensor was fabricated to be 500 μm wide. The narrow width of the top of the sensor was designed to reduce the thermal mass, and facilitate implant for in-vivo testing. The base of the sensor was kept wide to facilitate bonding to the pads. The bonding pads had a width of 250 μm with a pitch of 500 μm.
Figure 6-1. Schematic showing the 1x8 temperature sensing array fabricated in this work. The top of the sensor is kept narrow while the leads are spread out to facilitate wire bonding.

Figure 6-2 shows a close up view of the top of the sensor array. Although the array shown in this figure consists of vertically integrated thin films of VO$_x$, lateral configurations were also investigated in this work. Vertically integrated resistors had dimensions of 10 $\mu$m $\times$ 10 $\mu$m, while the lateral configuration had a W/L ratio of 10 $\mu$m/50 $\mu$m, both with a film thickness of 85 nm. To avoid catastrophic failure of the vertically integrated sensors due to electrostatic discharge during handling, all leads were connected using a shunt. Once the array was ready for measurement, the shunt connection could be cut by using micromanipulators on a probe station.

To improve the sensitivity of the fabricated array, glass substrates ($\approx$ 2 mm thick) were used due to their low thermal conductivity and therefore a lower ability to transport heat away from the sensors. The fabrication of the vertically integrated resistor array was done using a procedure described in section 5.3 and Figure 5-4, while the lateral configuration resistors were fabricated using the procedure detailed in Figure 3-18. The thin films of VO$_x$ were deposited using the BTIBD system using a three target configuration. The deposition parameters can be found in Table 5. The obtained resistivity of the thin films was $\approx$ 12,000 $\Omega$–cm.
Figure 6-2. Schematic of the top of the 1x8 vertically integrated VO<sub>x</sub> sensor array fabricated in this work. All through film sensors were electrically shorted during fabrication, dicing and wire bonding. Prior to measurement they were cut using a micromanipulator.

Table 5. Typical parameters used for the deposition of VO<sub>x</sub> thin films for this chapter

<table>
<thead>
<tr>
<th>Deposition Conditions</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Pressure</td>
<td>&lt; 1 x 10&lt;sup&gt;-7&lt;/sup&gt; Torr</td>
</tr>
<tr>
<td>Targets</td>
<td>3, Vanadium (99.99% pure)</td>
</tr>
<tr>
<td>Total pressure</td>
<td>5.8 x 10&lt;sup&gt;-4&lt;/sup&gt; Torr</td>
</tr>
<tr>
<td>O&lt;sub&gt;2&lt;/sub&gt; Partial pressure</td>
<td>3 x 10&lt;sup&gt;-8&lt;/sup&gt; Torr (4–5 sccm)</td>
</tr>
<tr>
<td>H&lt;sub&gt;2&lt;/sub&gt;O partial pressure</td>
<td>&lt; 2 x 10&lt;sup&gt;-7&lt;/sup&gt; Torr</td>
</tr>
<tr>
<td>Voltage pulse</td>
<td>99 µsec/1 µsec (–800V/+5 V) = 100 µsec</td>
</tr>
<tr>
<td>Target current</td>
<td>285 mA</td>
</tr>
<tr>
<td>HC parameters</td>
<td>10 sccm Ar flow, 1.5 A, 20 V</td>
</tr>
<tr>
<td>Ion source parameters</td>
<td>60 sccm Ar flow, 7.5 A, 38 V</td>
</tr>
<tr>
<td>Pre-sputter time</td>
<td>10 minutes</td>
</tr>
<tr>
<td>Stage spin</td>
<td>20 rpm</td>
</tr>
<tr>
<td>Time</td>
<td>30 minutes (85 nm thick)</td>
</tr>
</tbody>
</table>

An equivalent circuit diagram of a second design consisting of a ZnO based transistor/switch is shown in Figure 6-3. The VO<sub>x</sub> sensor/resistor is placed on the drain of the transistor. The current (and therefore impedance) is read between the drain and the source terminals when the transistor is turned on.
and a bias is applied using a voltage source. Devices were fabricated using both the vertically integrated and the lateral resistance structures.

![Equivalent circuit diagram (Figure 6-3)](image)

**Figure 6-3. Equivalent circuit diagram showing VO\textsubscript{x} resistors integrated with ZnO transistors as fabricated in this work.**

To reduce the resistance of the lateral VO\textsubscript{x} structures, interdigitated electrodes were fabricated as can be seen in Figure 6-4. The resistor’s W/L ratio was designed to be 160 \(\mu\text{m}/5 \mu\text{m}\). For high TCR VO\textsubscript{x} thin films (85 nm thick) having resistivity of \(\approx 10,000 \Omega\text{–cm}\), this implied a resistance value of \(\approx 35 \text{ M}\Omega\) for the lateral devices and \(\approx 1.5 \text{ M}\Omega\) for the vertically integrated devices Figure 6-5. The W/L ratio of the transistor was chosen so that the TFT’s contribution to the total impedance is minimal. This ratio is especially critical for the vertically integrated transistors where the resistance of the VO\textsubscript{x} structure is of the order of 1 M\Omega. For these TFTs, interdigitated source drain contacts were deposited having a W/L ratio of 470 \(\mu\text{m}/5 \mu\text{m}\) for TFT on resistance of \(\approx 1,500 \Omega\).
Figure 6-4. Schematic of lateral VO\textsubscript{x} sensors integrated with ZnO based transistors. To allow for low impedance, the width to length ratio of 200\textmu m/5\textmu m was designed for both the VO\textsubscript{x} resistors (100 M\textohm) and the ZnO transistors (4,000 \textohm).

Processing of these TFT integrated VO\textsubscript{x} thin films was done using a 6 layer mask set as follows. Bottom gate metal (100 nm of Ti) was patterned by using sputter deposition and liftoff techniques. The thin film of VO\textsubscript{x} was deposited using parameters shown in Table 5. Following this the VO\textsubscript{x} thin films were patterned and etched using double layer lithography in a 1:2,000 solution of H\textsubscript{2}O\textsubscript{2} and H\textsubscript{2}O. A second layer of Ti metal was sputter deposited onto the VO\textsubscript{x} thin films to serve as electrodes for the thin film.

The gate oxide and ZnO layers were deposited using plasma enhanced atomic layer deposition (PEALD) with help from Yiyang Gong from the department of Electrical Engineering. A 32 nm of Al\textsubscript{2}O\textsubscript{3} was deposited from trimethylaluminum (TMA) and CO\textsubscript{2}, and a 10 nm ZnO film was deposited from diethylzinc and N\textsubscript{2}O, both at 200 °C. After the PEALD step the ZnO was patterned by wet etching in diluted HCl (1:4000), and vias were etched in the Al\textsubscript{2}O\textsubscript{3} down to the bottom metal and the second metal contacts, by wet etching in hot (80 °C) phosphoric acid. Finally the source drain contacts were sputter deposited and patterned by liftoff.
Figure 6-5. Schematic of vertically integrated VOₓ sensors with ZnO based transistors. The through film resistors had dimensions of 10 μm × 10μm × 100 nm (1.3 MΩ), while the ZnO transistors were fabricated with a width to length ratio of 450 μm/ 5μm (1,500 Ω).

Figure 6-5 shows the schematic of vertically integrated VOₓ resistors. For these structures, the bottom metal used as the gate metal for the ZnO TFT, was used as the bottom electrode for VOₓ thin films.

6.2.2. One Dimensional Testing Setup

One dimensional arrays were fabricated using both lateral and through film configurations using high TCR VOₓ thin films. Figure 6-6 shows the equivalent circuit diagram of the design used for the fabrication of the array. Resistors R₁ through R₈ are resistors of VOₓ thin films which could be fabricated using either lateral or vertically integrated configurations. Switches S₁ through S₈ are relays of the Keithley 7075 general purpose multiplex card which can be used to select the resistor to be interrogated. Each switch of the Keithley 7075 general purpose multiplex card is connected to an HP 414B DC source/monitor unit (SMU) which sources voltage and measures current through each resistor. Since 4 SMUs are available on an HP 4141B, up to four resistors could be read simultaneously to achieve a higher array refresh rate. The 1-D array was measured by bonding the array to a PC Board using anisotropic conductive film (ACF) bonding. Leads from the board were connected to individual relays of
the Keithley 7075 multiplex card. Figure 6-7 shows an array bonded to a PC Board and the setup used for measurement of the 1-D array.

For temperature stabilization, the entire array was placed on a VWR heater block which had a temperature stability of ± 0.1 °C and a surface temperature variability of ± 0.1 °C. The entire setup was insulated from the environment using a foam construction as seen in Figure 6-7.
Figure 6-7. Test setup for the measurement of the 1-D arrays. ACF bonded samples on a VWR heater block (left) and the sensor biasing hardware including the HP4141B DC SMU and the Keithley 707A mainframe consisting of the Keithley 7075 general purpose multiplex card (right).

Figure 6-8. Screenshot of the C++ based program written to readout the 1D array by remote operation of the HP4141B DC SMU and the Keithley 7075 general purpose multiplex card. The program sets parameters for the array voltage bias, current measurement, the number of spot measurements to be performed and the wait time between each measurement.

The testing of the array was performed using a C++ program (screenshot in Figure 6-8) which provided computerized control and automation of the measurement. Appropriate bias, the integration time
for the HP 4141B DC SMU as well as the number of iterations the array needed to be measured can be remotely selected. The program triggered a spot measurement of the HP4141B after the selected bias was applied. Since two sensors are needed to be measured by one SMU, each device was left unbiased while the other device was being measured. Using a long integration time, the array had a refresh time of 3.5 seconds.

6.3. Results and Discussion

The 1-D array consisting of lateral VO\textsubscript{x} resistors was biased using a voltage of 1 V. The output current for all the 1×8 sensors was recorded over a 24 hour time period at a temperature of 35 °C. The current of one of the sensors is shown in Figure 6-9. The output current has an RMS noise of ≈ 160 mK and is found to drift by ≈ 0.25 °C over the 24 hour time period. In addition, the output current shows periodic oscillations with a frequency of 4 × 10^{-4} Hz.

![Current output for a lateral VO\textsubscript{x} sensor with a resistance of ≈ 50 MΩ at 35 °C measured over 24 hours. The periodicity arises from the attempts of the heater block to stabilize to a set point of 35 °C. The measured RMS noise was found to be 160 mK.](image-url)

Figure 6-9.
Figure 6-10 shows the output current from all 8 sensors of the lateral 1×8 sensor array. It can be seen that all sensors have similar values of resistance (≈ 1 %). All sensors have similar drift in the long term performance of 0.2 °C. The low frequency oscillations observed appears at the same time for all sensors. This implies that the observed periodic fluctuation in the output current at a frequency of 4×10^{-4} Hz is due to oscillations in the temperature of the heater block.

The RMS noise observed in all 8 sensors is ≈ 160 mK. Table 6 gives the parameters used as well as the calculated and observed signal to noise ratio (SNR) of thin film sensors using lateral resistance structures. The experimentally observed noise is ≈ 40 times higher than the theoretical calculations.
estimated using Equation 30. The higher noise can be attributed to the cables used in the setup and leakage through the Keithley switch matrix card. When open circuit measurements were performed with the setup, current leakage of ± 40 pA was observed.

Table 6. Assumed parameters for SNR calculation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Lateral</th>
<th>Vertically Integrated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistance</td>
<td>50 MΩ</td>
<td>1.5 MΩ</td>
</tr>
<tr>
<td>$V_{\text{bias}}$</td>
<td>1 V</td>
<td>0.1 V</td>
</tr>
<tr>
<td>N. Hooge’s parameter ($\alpha_H/n$)</td>
<td>$10^{-18}$ cm$^3$</td>
<td>$10^{-18}$ cm$^3$</td>
</tr>
<tr>
<td>$f_2$</td>
<td>714 Hz (1.4 ms measurement)</td>
<td>714 Hz (1.4 ms measurement)</td>
</tr>
<tr>
<td>$f_1$</td>
<td>1 Hz</td>
<td>1 Hz</td>
</tr>
<tr>
<td>Samples averaged (based on integration time)</td>
<td>256</td>
<td>256</td>
</tr>
<tr>
<td>TCR</td>
<td>$-4.4 %/K$</td>
<td>$-4.4 %/K$</td>
</tr>
<tr>
<td>$T$</td>
<td>308 K</td>
<td>308 K</td>
</tr>
<tr>
<td>Volume</td>
<td>$10 \mu m \times 50 \mu m \times 85 \text{ nm}$</td>
<td>$10 \mu m \times 10 \mu m \times 85 \text{ nm}$</td>
</tr>
<tr>
<td>Calculated SNR from Equation 30</td>
<td>10,000</td>
<td>7,200</td>
</tr>
<tr>
<td>Observed SNR</td>
<td>$\approx 280$</td>
<td>$\approx 1,500$</td>
</tr>
</tbody>
</table>

Figure 6-11 shows the current response of a vertically integrated sensor from a 1×8 temperature sensing array. These devices were biased with 0.1 V with other relevant measurement parameters as shown in Table 1. The observed SNR ratio was 1,500 compared with 280 for the lateral resistance structures as calculated from Equation 30. The recorded RMS noise for these sensors was $\approx 35$ mK compared with 160 mK for the lateral devices. The periodic fluctuation in the temperature of the heater block can be seen much clearly for these vertically integrated sensors.

Figure 6-12 shows a plot of log resistance versus temperature for a lateral device. The TCR from the measurement was found to be $-4.4 \%/K$ which was similar to that of vertically integrated thin films.
Figure 6-11. Output current of vertically integrated VO\textsubscript{x} sensors 10 \(\mu\text{m} \times 10 \mu\text{m} \times 85\text{ nm} \) having a resistance of 1.3 M\(\Omega\) at a temperature of 35 °C. The resistor was biased with a voltage of 100 mV. The measured RMS noise was found to be \(\approx 35\text{ mK}\).

Figure 6-12. A plot of natural log of resistance as a function of temperature for lateral thin film resistors of VO\textsubscript{x} having dimensions of 10 \(\mu\text{m} \times 50 \mu\text{m} \times 85\text{ nm} \). The TCR extracted using activation energy was found to be \(-4.4 \%/\text{K}\).
Although there is a significant decrease in the electrical noise for the vertically integrated devices in comparison with the lateral devices (from 160 mK to 35 mK), the noise is significantly higher than the theoretical calculation of electrical noise of 8 mK. One such reason for the disparity in the electrical noise could be elevated levels of electrical noise due to the ribbon cables used in the test setup. Figure 6-13 shows the measured current noise of the measurement system over duration of 8 hours with the VO\textsubscript{x} sensors disconnected. This open circuit measurement gives the lower limit of the current signal current which can be measured and hence sets a lower limit on the achievable sensitivity. This current noise was found to be of the order of 40 pA.

![Figure 6-13. A plot of leakage current through the ribbon cables used during measurement.](image)
In order to measure the sensitivity of the sensor, a lower noise test setup was used. The sensors were measured without the use of the ribbon cables on a probe station using the HP 4141B DC SMU. Figure 6-14 shows the electrical output of the temperature sensors measured on the probe station without the ribbon cables. These sensors have sensitivity of 12 mK, a significant improvement from 35 mK measured using ribbon cables.

![Graph showing vertically integrated resistor output](image)

Figure 6-14. Output current of vertically integrated VO₃ sensors 10 μm × 10 μm × 85 nm having a resistance of 1.3 MΩ at a temperature of 35 °C measured without a switch matrix using the HP4141B DC SMU. The resistor was biased with a voltage of 100 mV. The measured RMS noise was found to be ≈ 12 mK.

To evaluate the performance of the TFT integrated temperature sensors, the TFT performance was characterized to extract the threshold voltage (Vₜ), the resistance of the TFT when turned on (R_{on}) and its mobility. Figure 6-15 (black curve) shows a plot of the Log (I_D) versus gate voltage (V_G) for a transistor with a width to length ratio (W/L) of 400 μm/15 μm. The extracted differential mobility (red
curve) was found to be $\approx 10 \text{ cm}^2/\text{V} \cdot \text{s}$ at a $V_{\text{DS}}$ of 1 V at a $V_{\text{GS}}$ of 12 V. $R_{\text{on}}$ of $\approx 4,000 \ \Omega$ was achieved for this W/L.

Figure 6-15. Linear region log ($I_D$) versus $V_{\text{GS}}$ of a ZnO based TFT with a linear region differential mobility of $10 \text{ cm}^2/\text{V} \cdot \text{s}$ for a $V_{\text{DS}}$ of 1 V. The TFT dimensions are $W/L = 400 \mu\text{m}/15 \mu\text{m}$ and $t_{\text{ox}} = 32 \text{ nm}$. The $R_{\text{on}}$ for this W/L ratio was $\approx 4,000 \ \Omega$ for a $V_{\text{GS}}$ of 10 V.

Figure 6-16 shows the $I_D$ versus $V_G$ for a ZnO TFT ($W/L = 400 \mu\text{m}/5\mu\text{m}$) integrated with vertical VO$_x$ resistors ($\approx 1.5 \text{ M}\Omega$) for a $V_{\text{DS}}$ of 75 mV. The expected $R_{\text{on}}$ for these TFTs is $\approx 1,500 \ \Omega$. It can be seen that the transistor is off at $-1 \text{ V}$ and is on at 4 V.
Figure 6-16. Log (I_D) versus V_G for a ZnO based TFT with a vertically integrated VO_x resistor (1.6 MΩ at room temperature) on the transistor drain for a V_DS of 75 mV (W/L = 450 μm/5 μm, t_ox = 32 nm).

Figure 6-17 shows the drain current of the TFT integrated with vertical VO_x thin films structures. As the gate voltage is increased from a V_G of −1 V to +5 V, the resistor is swept from a bias of −0.1 V to 0.1 V (V_DS) and the current is recorded. When the transistor is off, the impedance of the integrated sensor is 10^{11} Ω, when the transistor is turned on the resistance is 1.3 MΩ (~ the resistance of the VO_x resistor) for V_GS higher than 4 V.
Figure 6-17. $I_D$ versus $V_{GS}$ of a ZnO based TFT with a vertically integrated with vertically integrated VO$_x$ resistor (1.6 MΩ at room temperature) on the transistor drain as a function of $V_{GS}$ from -1 V to 5 V (W/L = 450 μm/5 μm, $t_{ox}$ = 32 nm). $I_D$–$V_D$ at $V_{GS}$ of 4 V and 5 V are similar indicating a small impedance contribution from the ZnO TFT.

Figure 6-18 shows the $I_D$–$V_D$ curve of a vertically integrated VO$_x$ sensor incorporated with a ZnO TFT as a function of temperature. The drain voltage is swept from −0.1 V to +0.1 V and the resultant current is recorded. During this measurement, the $V_{GS}$ of the TFT is held at +4 V so that the transistor is turned on. After each measurement the temperature was increased and the $I_D$–$V_D$ sweep was collected at this new elevated temperature. The resistance of this vertical device along with the TFT was plotted at various temperatures in a Ln (resistance) versus temperature plot and can be seen in Figure 6-19. The extracted TCR from the activation energy was found to be −4.4 %/K.
Figure 6-18. $I_D$ versus $V_{GS}$ of a ZnO based TFT with a vertically integrated VO$_x$ resistor (1.6 MΩ at room temperature) on the TFT drain as a function of temperature for a $V_{GS}$ of 5 V ($W/L = 450 \mu m/5 \mu m$, $t_{ox} = 32$ nm). Increasing temperature of the device leads to lower resistance of the VO$_x$ thin film at higher temperatures which is evident from the increasing slopes of the $I_D-V_D$ characteristics.

Figure 6-19. Ln (Resistance) versus temperature of vertically integrated VO$_x$ thin film resistors (1.6 MΩ at room temperature) integrated with ZnO TFT at a $V_{GS}$ of 5 V ($W/L = 450 \mu m/5 \mu m$, $t_{ox} = 32$ nm). The TCR extracted from the activation energy was found to be $-4.4 \%/K$. 

TCR = $-4.4 \%/K$
6.4. Conclusion

$1 \times 8$ temperature sensing arrays are fabricated using lateral and vertically integrated thin films of high TCR VO$_x$ thin films. Custom hardware and software was built using a DC SMU and a switch matrix to perform the measurements. Thin film TCR of $-4.4 \, \%K$ was maintained in both lateral and vertically integrated structures. The hardware setup allowed for the arrays to have a refresh time of 3.5 seconds. While the arrays using lateral resistance structures have an RMS noise of 160 mK, due to limitations imposed by the hardware setup. The vertically integrated resistors have higher sensitivity due to increased magnitude of the signal. The RMS noise of the vertically integrated structures was found to be 35 mK.

Both the lateral and vertically integrated resistance structures were incorporated along with a ZnO based TFT for device level multiplexing of the sensor arrays. The bias and therefore the current from the resistor could be turned on or off depending on the gate voltage applied to the transistor. Temperature dependent measurements confirmed that the TCR of the thin film material was maintained in these TFT integrated resistance temperature sensors.
Chapter 7. Summary and Future Work

7.1. Summary

High TCR Thin films of VO\(_x\) and Ge:H

Resistance based microbolometers are based on two distinct types of materials: materials with |TCR| of 2–2.5 %/K, resistivity between 0.1–1 Ω–cm and those with |TCR| between 3–4 %/K with resistivity between 200–2,000 Ω–cm. This work investigates thin films of Ge:H and VO\(_x\) having |TCR| > 4 %/K.

For thin films of undoped Ge:H deposited by PECVD, it was found that significant improvement in the resistivity, TCR and electrical noise could be achieved for a thickness of \(\approx 100\) nm by increasing the dilution ratio of [GeH\(_4\)] in [H\(_2\)] to extremely large values (R = 400). Thin films having TCR of \(\approx 6.5\) %/K were repeatedly deposited having a resistivity of 2,250 Ω–cm and a normalized Hooge’s parameter of \(\approx 10^{-20}\) cm\(^3\). In combination with prior work it is concluded that the thickness of thin film showing superior TCR-noise-resistivity tradeoffs could be achieved by could be changed by changing the dilution ratio used during deposition. The combination of electrical properties for thin films of Ge:H deposited in this work are the best in comparison with other materials reported in the literature to date.

Electrical noise in thin films of VO\(_x\) with \(x > 2\) and |TCR| > 3%/K was largely uninvestigated prior to this work. Thin films having high TCR (|TCR| of 4–5 %/K) were deposited by Biased Target Ion Beam Deposition. These thin films had very high resistivities (between 10,000 to 20,000 Ω–cm), much higher than those of Ge:H. Their normalized Hooge’s parameter values were around \(10^{18}\) to \(10^{20}\) cm\(^3\).
Vertically Integrated High TCR Thin Films

The use of large TCR values in microbolometer applications has been limited by large values of normalized Hooge’s parameter and the enormous resistivities (10,000 Ω–cm) associated with these materials. High resistivity leads to high pixel resistance in a lateral resistor configuration. In this work, an alternate pixel structure using vertically integrated high TCR films was demonstrated and its electrical performance was characterized.

This configuration resulted in much lower pixel resistance than the lateral configuration with no change in the signal to noise ratio. In addition, TCR and the normalized Hooge’s parameter of the vertically integrated configuration were found to be similar to resistors fabricated in the lateral configuration. The reduced pixel resistance allows for sustaining higher bias currents without an increase in Joule heating and therefore operation in a constant bias mode. In addition, the constant SNR and larger signal current might allow for reduced current integration times and therefore higher imaging frame rates.

Temperature Sensor Arrays

Combination of large TCR materials and low impedance devices developed in this work positions the thermistor based contact based temperature sensor at a distinct advantage over existing micro fabricated temperature sensors. 1-D temperature sensing arrays using thin films having large values of TCR lateral and vertically integrated configurations were fabricated on to glass substrates. 1×8 sensor array using the lateral configuration was demonstrated have an RMS noise value of 160 mK, while vertically integrated resistors showed a much lower RMS noise of 35 mK. To allow for measurement of 2-D arrays, sensors were integrated with ZnO based transistors to turn the current through the resistor on or off depending on the gate voltage applied to the bias with no degradation in the sensor performance.

7.2. Future Work

This dissertation investigated several areas of temperature sensing materials and devices. As a result many questions arose which may be answered by future work. One main area of future work would
be to publish the results on the high TCR materials, the through film structure, the noise measurement system and results, and the temperature sensing array.

**High TCR thin films of VO\(_x\):**

Thin films of VO\(_x\) deposited by BTIBD were grown having high resistivity (>10,000 \(\Omega\)–cm) and moderate values of Hooge’s parameter (\(10^{-20}\) to \(10^{-18}\) cm\(^3\)). These electrical properties are too large for thin films of high TCR VO\(_x\) to be incorporated in existing pulse bias, lateral resistor, read out circuits. There appears to be a large gap in terms of resistivity (from 1 \(\Omega\)–cm to 10,000 \(\Omega\)–cm), and Hooge’s parameter (from \(10^{-24}\) to \(10^{-20}\) cm\(^3\)) where thin films of VO\(_x\) have not been successfully deposited. It is worth attempting to deposit films in this region to see if the electrical properties of these films are superior films of SiGe:H which are currently used in the constant bias bolometer market.

VO\(_x\) thin films with |TCR| > 2.5 %/K and reasonably low resistivity (< 0.1 \(\Omega\)–cm) have been deposited by reactive pulsed DC magnetron sputtering of a vanadium target with oxygen [37]. Basantani et al. showed that when thin films of VO\(_x\) were deposited using pulsed DC magnetron sputtering, an increase in the oxygen content during sputtering led to an increase in the thin film |TCR|. The |TCR| increased from 1.2 %/K at 4 % of O\(_2\)/Ar mixture to 2.1 %/K at 11 % O\(_2\)/Ar at a substrate bias of −250 V and a sputtering pressure of 2.5 mTorr [44]. In that work, a further increase in the oxygen content led to an abrupt increase in the TCR and resistivity of the thin films. At an O\(_2\)/Ar ratio of 12 %, the resistivity increased to 10 \(\Omega\)–cm and the TCR was 3 %/K [44].

In that work, a further increase in the oxygen content was not investigated because thin films having large resistivity were not of interest for that work. However, high TCR and high resistivity thin films are of interest for the constant bias bolometers. Further investigation of the parameter space not covered in ref. [44] could be performed with the deposition of thin films with oxygen percentage in argon from 12 % to 20 %. Some other deposition parameters that were held constant: the total pressure was maintained at 5 mTorr, the substrate bias was kept at −250 V and the total gas flow was held at 18 sccm.
Figure 7-1 shows that a further increase in the percentage of oxygen used during deposition led to an increase in the resistivity and the magnitude of TCR for the deposited thin films of VO\textsubscript{x}. For the thin film deposited with an oxygen content of 20\%, the resistivity was found to be \( \approx 6,000 \, \Omega \text{–cm} \) and the |TCR| was found to be \( \approx 3.8 \, \%/K \). The resistivity and TCR of these thin films are similar to the thin films of VO\textsubscript{x} deposited using BTIBD.

Future work for thin films deposited using pulsed DC magnetron sputtering of VO\textsubscript{x} thin films should involve investigation of thin films having even higher TCR values. An increase in the oxygen percentage during deposition beyond the 20\% investigated in this work may result in thin films with even
higher TCR values. In addition, the normalized Hooge’s parameter must be investigated for thin films of VO$_x$ deposited using pulsed DC magnetron sputtering.

**Hydrogenated Germanium Thin Films:**

Thin films of Ge:H were deposited with superior electrical properties of TCR (> 6 %/K), resistivity (2,250 Ω–cm), and normalized Hooge’s parameter (10$^{-20}$ cm$^3$). In combination with prior work it is concluded that the thickness of thin films showing superior TCR-noise-resistivity tradeoffs can be adjusted by changing the dilution ratio used during deposition. Through years of investigation, F. N. Hooge concluded that 1/f noise of thin films is strongly inversely proportional to the number of carriers available for transport [29]. Therefore, similar to the work done for SiGe:H systems, it is worth investigating if the material properties can be improved by doping of the thin films to increase the available carriers for transport. In the case of SiGe:H thin films, Ajmera et al. and Saint John et al. have demonstrated a decrease in the normalized Hooge’s parameter ‘\(\alpha_H/n\)’ with little change in the TCR of the thin films [18] [19].

**Vertically Integrated Thin Films of VO$_x$:**

Vertically integrated thin films of VO$_x$ were demonstrated as an alternate to lateral resistor configurations. A few key questions arose as a result of this work:

1. It was observed that there exists a barrier layer at the interface of the sensing material for both VO$_x$ and Ge:H based devices. Further investigation must be done to identify this barrier layer which may involve the use of a noble metal.

2. Pin hole defect density for large area out of plane devices is a concern which must be addressed to prove manufacturability of vertically integrated bolometer structures.

3. The performance of the vertically integrated sensor has been evaluated by measuring the electrical response as a function of change in temperature. However, the spot temperature
measurements may require additional information on heat transfer between the substrate, the active thin film material as well as the top and bottom electrodes. This information can be obtained by means of Finite Element Analysis (FEA).

4. The true test of this architecture would be to evaluate a pixel incorporated into a free-standing MEMs structure including a full evaluation of the optical and thermal properties of the device.

**Temperature Sensor Array:**

One dimensional temperature sensing arrays were demonstrated in this work by means of a Keithley 707A general purpose multiplex card. The ability to switch each sensor ON/OFF was also demonstrated by means of integrated ZnO TFTs.

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**Figure 7-2.** A schematic showing the layout of an 8 x 8 temperature sensor array with integrated TFTs for two dimensional temperature sensing applications.
Future work for the one-dimensional sensor array would be to fabricate them on a flexible, biocompatible substrate for insertion into the body, particularly the brain. This would require temporary attachment of the device on a stiff but small cross-sectional area probe that would allow insertion, dissolution of the adhesive and removal of the probe.

Future work for the temperature sensor array includes measurement of two dimensional temperature sensor arrays with integrated TFTs. Figure 7-2 shows a layout of two dimensional temperature sensor arrays with integrated TFTs for in pixel switching. Hardware test setup must be developed for appropriate biasing of these temperature sensor arrays.
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Vita
Hitesh Basantani

Hitesh Arjun Basantani was born in Kuwait on April 24th, 1987, to parents of Indian decent Meena Basantani and Arjun Basantani. They moved to New Delhi, India for a few years during the Gulf War of 1990 before returning back to Kuwait in 1992. Hitesh graduated from the Carmel School of Kuwait in 2004. He moved to The Penn State University for his B.S. and M.S. degree in Engineering Science and Mechanics, which he obtained in 2009 and 2011 respectively. His Master’s thesis was on the development of vanadium oxide thin films for microbolometer application.

Following the obtaining of his M.S. degree, he stayed at Penn State for his Ph.D and was the first Super User for the Penn State Nanofabrication Facility. Here he worked on magnetron and low energy ion beam deposition systems. His Ph.D. work was on temperature sensing materials and devices.

In July of 2014, Hitesh accepted a position at Intel within the Intel Mask Operations Unit and will be moving to Hillsboro, OR with his fiancée Jennifer Ober.