Measurement of Low Temperature Thermal Properties of Microcalorimeters Using Johnson Noise Thermometry

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MEASUREMENT OF LOW TEMPERATURE THERMAL PROPERTIES OF MICROCALORIMETERS USING JOHNSON NOISE THERMOMETRY

A DISSERTATION
PRESENTED TO THE FACULTY
OF NATURAL SCIENCES AND MATHEMATICS
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IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE
OF DOCTOR OF PHILOSOPHY

BY
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Abstract

Calorimetric measurements and thermal transport measurements through a Si$_x$N$_y$ patterned and suspended membranes have been performed to further the understanding of low temperature reduced dimensional transport, and understand the interesting physics present within an amorphous dielectric material. The thermal properties of several calorimeters have been measured to understand the geometric dependence on transport phenomenon. We analyze and compare our results with observations of previous measurements, and with those indicating quantum limited thermal transport. We see strong indications of changes to the physical mechanism transporting heat through these materials as temperature changes from 50 mK to 2 K. We have also developed a relaxation calorimetric measurement for use with Johnson Noise Thermometry.
Acknowledgements

I have come to understand a great deal more about the world, and my place within over the last 5-plus years. I set out to be in a position to understand and observe phenomenon that had only been observed by a few people ever, and to possibly discover something new. I have benefited greatly from the efforts of other scientists, people I am familiar with, and those that I have never, and never will meet. Each and every person that seeks to discover, and understand new things has laid something valuable on the altar of science.

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Chapter 1

Introduction

1.1 Introduction

Further developing the understanding of physics in a number of materials is often probed through understanding how matter interacts with energy in a variety of circumstances. This often necessitates the measurement and understanding of the thermal properties of a system. Specifically, how heat is passed through a material (thermal conductance $G$), and how it interacts with energy (heat capacity $C$). These measurements are indications of physical changes within the material, and are tools that shed light onto the physics of matter. [4] [15]

While much is understood about how the interaction between matter and energy under many circumstances, much more remains to be described if we are to achieve the advances in understanding and technology that facilitate improvements in our daily lives. It is with this aim that we have studied the thermal properties of matter temperatures below 4K and in constrained dimension. The overwhelming trend in technology development since the advent of the micro-processor is to achieve vast performance increases within an ever decreasing footprint. This drive has lead to advances and discoveries seemed impossible only a few decades ago. The consequences of this drive have lead to discoveries of physical phe-
nomenon that have pushed the boundaries of science and forced the development of entire fields of science.

The desire to shrink functional devices to the diffraction limit of light itself has generated numerous questions, and answers, about the interaction of matter and energy as the scale of a device approaches fundamental physical length-scales. These questions have often forced answers that challenge current understanding and force adaptations to models. As science furthers technology, technology furthers science.

We are currently realizing many technological advances that will further enable scientific exploration and understanding as the associated length-scales further decrease. One such technological advance is the establishment of Transition Edge Sensing (TES) calorimeters.

1.2 Transition Edge Sensors

The idea behind this technology is using the steep $R(T)$ of superconducting materials around the transition temperature ($T_c$) as a highly sensitive thermometer. The sensitivity of a thermometer is given by

$$S = \frac{d\Theta}{dT} \frac{T}{\Theta}$$

(1.1)

Where $\Theta$ represents some physical observable that has a temperature dependence. Using the steep change in resistance as a superconductor passes through its $T_c$ as a thermometer is very high. Typically low $T_c$ superconductors are metals, and resistance of metals should not change as a function of temperature at $T \lesssim 5K$, and is 0 for all temperatures below $T_c$. The implications of this are that within a narrow range of operational temperature, a superconducting material is a highly sensitive thermometer.

$$S_{sc} = \frac{dR(T_c)}{T} \frac{T_c}{R_{measured}}$$

(1.2)

Using a superconductor as a thermometer requires special considerations to ensure a
Figure 1.1: Model of a TES calorimeter with absorber coupled to thermometer, and weak link to bath. An array of $\gamma$-ray detecting TES micro-calorimeters fabricated by collaborators at NIST Boulder [6].

stable temperature in within the superconducting transition. We can instead use the dramatic changes in superconducting critical current $I_c(T)$ as the thermometer. The Critical current of a superconductor is defined to be the maximum current a superconducting material can support before undergoing a phase transition back to a normal metal.

Figure 1.2: Qualitative plot of $R(T)$ of a normal metal and Superconductor showing relative changes in $\frac{dR(T)}{dT}$ [18].
The properties of a superconducting film as a thermometer dictate that it be used to measure very small temperature excursions from a narrow operational window in $T$. These constraints and advantages lend superconducting thermometers for application as a calorimeter, where the small changes in temperature correspond to small dissipations of energy in the thermometer, but the changes in temperature are small compared to the bath temperature. The operational temperature of a superconducting thermometer can be reduced to slightly below $T_c$ and by establishing a DC current passing through the superconducting material, slight changes in its temperature will cause the material to undergo a phase transition and the resistance of the superconductor will increase dramatically with temperature.

TES’s were not realized, despite their initial demonstration Andrews et al. in 1941, and conceptualization three years prior by the same group[1][2]. The proof of concept involved observing the superconducting to normal transition induced by shining infrared light on a superconducting wire. Several limiting technological factors prevented implementing TES’s as detectors. These include difficulty impedance matching the signal in the cryostat to room temperature amplifiers, and operating these sensors within the narrow superconducting-to-normal transition requires extremely careful regulation of the bath temperature. Advances made in cryogenic amplification, understanding the proximity effect in superconductors, and altering the bias scheme have allowed TES’s to become detectors used in photon spectroscopy from the infra red to gamma ray energies [7][63].

1.2.1 TES Microcalorimeters

By developing TES’s and adapting them for use as microcalorimeters has allowed development of spectrometers whose resolution is orders of magnitude better than current spectrometers. Advances in technology this fundamental provide a tool to investigate aspects of science that were previously unavailable.

Examples of TES microcalorimeters being implemented in fundamental science missions exist from cosmological studies investigating the nature of dark matter, to implemen-
tation in sensitive material characterization tools in SEM’s.

Further understanding of limitations to microcalorimeter resolutions (like the low temperature thermal properties) will lead to further advances in the resolution of these devices. This will have further implications in advancing science in countless other avenues.

1.2.2 High Resolution Spectroscopy

The principle application for TES’s is in improving the resolution of microcalorimeters as spectrometers. The traditional spectrometer relies on semi-conducting materials for resolving the energy of an incident photon. The energy of an incident photon is inferred from the current generated when a photon excites a valence electron into the conduction band. Many such electrons can be generated, and the amount of current generated by an absorption event is an indication of the energy of the photon.

This type of detector can be made for use across the energy spectrum by introducing dopants, to engineer the energy gap, and electronic structure of the detector. Limits to this type of detector’s resolution result from interactions between charge carriers and the lattice. It is important to very quickly collect charge carriers generated from a photon absorption before the carrier interacts or recombines. For this reason, the most sensitive spectrometers are made from high purity Ge, Si detectors, and are typically operated at cryogenic temperatures.

1.2.3 Nuclear Forensics

Perhaps the most illustrative example of real-world impact these TES microcalorimeters is in performing Non-destructive isotopic analysis measurements of radioactive material. Observing the spectrum of emitted photons from radioactive material indicates the parent decay process that generated an emitted photon. By observing the various decays generated from a radioactive object will indicate the objects composition, this valuable information not only identifies what elements are present within the material, but also identifies the
abundances of these elements.

Comparing the relative abundance of different isotopes of particularly important material (Pu) indicates the level of enrichment needed to generate the material, as well as allowing comparison from reference measurements to identify where and when a material was created. The ability to accurately resolve the isotopic ratios of radioactive material is a critically important measurement that has implications in current and future political environment surrounding nuclear programs around the world [24].

![Figure 1.3: Observed decay spectrum of actual radioactive material. This sample generating this spectrum was generated to simulated spent nuclear material, and the observed energy resolution of the \( \gamma \)-ray microcalorimeter array of 256 detectors was 86 eV FWHM at 106 keV. The data in black was observed using the microcalorimeter array, while the red data was collected using a planar High Purity Ge detector, representing the current state-of-the-art. Measurements performed by A.S. Hoover et.al in conjunction with LANL and NIST [24].](image)

The obvious improvement in spectral resolution between HPGe and micrcalorimeter detection techniques for high energy photon measurement is clearly evident in these data, and the significant motivation to implement this detection technique as a complementary non-destructive analysis measurement. Adapting a TES to perform a corollary measurement of total energy dispersive spectroscopic (Q-Spec)measurement will address the some limitations associated with bulk absorber. Then, the total energy dissipated within the absorber can be calculated, and further improvements on energy resolution can be achieved.

The spectral resolution of these detectors, while already impressive, diminish as a result of grouping them into large arrays. Because each calorimeter is subtly different than the others, and is highly sensitive to operational parameters, operation of large detector arrays
is hardly a trivial matter. Much of the limitation in resolution for large arrays is due to slightly different behavior of the thermometer in the superconducting transition. This leads to a spread in the energy resolution of detectors. For large arrays of TES calorimeters, each detector needs to be individually biased for optimized energy resolution and performance.

It is possible to limit the effect different R(T) curves for thermometers by individually biasing each detector to an operating point within the superconducting transition. Voltage biasing a TES microcalorimeters in the superconducting transition allows negative electro-thermal feedback, which was an important development to establishing TES calorimeters as a viable detection technique. Negative electro-thermal feedback facilitates stable biasing of an individual detector within the transition despite small fluctuations in base temperature, as well as improving the counting rate of these sensors. This biasing scheme automatically decreases the power dissipated in the thermometer as the temperature rises [25]. Realizing optimal performance of a TES relies on accurately predicting the thermal properties of the fully assembled detector, creating a predictable superconducting transition for thermometry, and operating the detector at optimal conditions. Currently, the discrepancies between predicted behavior and actual thermal properties is a substantial limitation to the real performance of operational TES detectors. Improving the description of thermal transport at low temperatures in amorphous dielectric films like a-Si$_x$N$_y$ membranes. These membranes provide thermal isolation and are the foundational element of high energy TES detectors. Typically these membrane geometries are thin (100’s of nm thick) and operated at temperatures at which the transport phenomenon are affected by finite size effects. Under these circumstances thermal transport is highly sensitive of the defining geometry, and surface condition of the transport medium.

1.3 Reduced Dimensionality Transport

Advancing our understanding of transport processes as dimensions become smaller and can be first understood at low temperatures. Low temperatures allow the long mean free
paths necessary to understand how transport phenomenon are altered by restrictions in dimension. One area of interest is in how phonon transport is altered by a reduction in thickness such that common assumptions made about the number of available modes are no longer valid. These small influences can shed light into the behavior of different carriers. Geometric restrictions to electronic and thermal transport phenomenon have applications in surprising areas of science and technology.

As the transistor, the fundamental element of a computer, becomes smaller and smaller, performance of modern computers is bottlenecked by limited thermal transport from the transistor to thermal bath. It seems likely that Moore’s law will soon be broken because of the significant hurdles to be overcome in realizing further reductions in size. Particularly when it comes to moving such a large thermal flux efficiently to bath. The ability to separately control the electronic and thermal transport phenomenon can translate into dramatically improved thermoelectric devices, and improved efficiencies in many devices.

Electronic transport properties are well understood, and considerably easier to measure, but as the dimensions of transport become limited, assumptions such as the Sommerfeld value fail to accurately capture the relationship of electrical and thermal transport. In these conditions it is necessary to measure both thermal and electrical transport values. This is of particular importance to advancing development of highly structured thermoelectric materials. Research into thermoelectric materials over the last 10 years has shifted to thin film and “nano-scale” regime specifically to exploit larger differences between thermal and electrical conductance.

### 1.4 Amorphous Material Thermal Properties

Thermal transport in insulating materials is well described by the Debye model. At low temperatures, an amorphous material has thermal transport also dictated by phonon populations, however, the observed thermal conductivity for a number of amorphous materials at low temperatures deviates from predictions using the Debye model in which $\kappa \propto T^3[61]$. 
Figure 1.4: Classical observation amorphous structure affecting thermal properties. Zeller and Pohl observed a difference in heat capacity of SiO$_2$ between an amorphous structure (glass), and crystal structure (Quartz) [62].

Bulk amorphous materials at low temperatures obey $\kappa \propto T^{(2-\delta)}$, with measured exponents falling between 1.7 and 1.9 [45]. Because of the amorphous nature of Si$_x$N$_y$ membranes, we can address the influence of frustration on transport phenomenon. One question that has eluded a satisfying quantitative description is how and why a variety of different material have strikingly similar transport phenomenon across a relatively large temperature range when amorphous. Measurements of insulating amorphous materials with very different Debye Temperatures ($\Theta_D$) and sound speeds should have a transport phenomenon that are comparable. Zeller and Pohl observed that the amorphous nature of a material has a strong influence on transport within the medium [62]. The observation that amorphous materials have transport properties that vary as $\propto T^2$ with comparable amplitudes is an interesting observation.
Chapter 2

Johnson Noise Thermometry

2.1 Introduction

Selecting a thermometer for use at low temperatures is a design parameter dictated by the ease of use, sensitivity, precision, and the measurement requirements. Thermometers for use at $T < 1 \text{ K}$ typically are unsuitable for use above 5 K. This is the case because dominant interactions at high temperatures tend to change as temperature approaches 0 K. For instance, typical thermometers used in the temperature range from 200 K to 77 K such as simple resistive element can be used as a thermometer. We can easily create a thermometer by measuring the resistance of a conductive film as a function of temperature, or by measuring the voltage generated by a thermoelectric material. As the temperature decreases below 10 K, the sensitivity of such a thermometer approaches zero, rendering a simple metallic film ineffective as a thermometer. The sensitivity of a thermometer is given by eq. 2.1

This happens because the dominant scattering shifts from electron-phonon interactions to electron-impurity scattering. Platinum thermometers also suffer from a dramatic decrease in sensitivity as the relative thermal excitations decrease. As matter is cooled, the physics of materials change. These changes can render a thermometer ineffective as temperature...
drops below 4 K.

\[ S \propto \frac{d\Theta}{dT} \]  

(2.1)

\[ \lim_{T \to 0} \frac{dR_{metal}}{dT} = 0 \]  

(2.2)

For measuring temperatures below 10 K, we rely on different physical processes to observe temperature. Low temperature thermometers make use of similar observables; like resistance, noise amplitude, voltages/currents developed across material junctions (NIS), and magnetic moments of materials [17] [46]. Resistance thermometers come in many forms including doped Ge and Si, and rely on thermally excited conduction. At low temperatures thermally excited charge carriers within these doped semiconductors exhibit Variable Range Hopping [5]. Thus, electrons flow through a semiconductor at low temperature, and the resistance of the material increases as temperature decreases. While the materials may be similar, and the observables are the same, the physics governing the temperature response of resistive thermometers are very different. The resistance of this type of material is describe in eq. 2.3.

\[ R(T) = R_0 e^{\left(\frac{T_0}{T}\right)^p} \]  

(2.3)

We see that resistance thermometers suitable for low temperature thermometry relies on different physical phenomenon than resistance thermometers suitable for higher temperatures. This type of thermometer is highly sensitive to the fabrication techniques used, and the dopant homogeneity. Typical thermometers of this class include Neutron Transmutation Doped Ge (NTD Ge) thermometers, Ruthenium Oxide thermometer (RuOx), and Silicon/Ge films doped with metallic elements. Low temperature resistive thermometers have very high sensitivities, but can become challenging to measure as \( R_{therm} > 1 M \text{ } \Omega \). The high sensitivity of a resistive thermometer is appealing, but can represent problems measuring such large resistances at low temperature. Resistance thermometry is perhaps
the most common technique used at low temperatures, but can become problematic for conducting measurements of thermal properties because the likelihood of stray heating or self heating by virtue of measuring the thermometer.

Johnson Noise thermometry (JNT) is a technique to determine the temperature of a resistive element by measuring the noise amplitude generated by the resistor. This technique has a significant advantage over more commonplace resistance thermometry in that it is a non-dissipative technique [42]. Measuring the temperature does not introduce self heating or stray heating effects. All other low temperature thermometry involves exciting or actively probing the thermometer, while JNT passively observes generation of small voltage (current) fluctuations existing about a resistive element. Since fabrication involves creating a low resistance conductor, and electrical leads, the process is quite simple, and lends itself well to creating thermometers with a small form factor that is more easily reproduced than doped semiconductors, with better thermal contact to the membrane [50].

In order to understand the source of Johnson-Nyquist noise we look to the original observation and description of this noise [26][43]. If we consider a conductor at low temperature, the electrons all lie on the Fermi surface and are described as a free electron gas. Slight perturbations or vibrations within the ionic lattice result in corresponding perturbations of the free electrons. These perturbations generate very small fluctuations in the position of electrons throughout the conductor giving rise to small, but measurable fluctuations in voltage across the resistor. Small fluctuations in location of electrons give rise to currents within the material as electrons are perturbed from their equilibrium position. We see that the voltage generated across a material.

The voltage noise developed across a resistive element is described by the Johnson-Nyquist relation, where the voltage noise is given by eq. 2.4, and the current noise is given by eq. 2.5. This classical description of the noise amplitude is independent of frequency. We should observe a constant power spectral density from DC to many GHz, where corrections due to relativistic quantum mechanics become relevant.
Since any resistive element within the circuit generates a signal governed by the same law, we observe that a loop including two resistive elements, with different values, at different temperatures (such is the case for our thermometer connected by normal metal wires), the observed noise amplitude will be given by eq. 2.7. It is obvious that in order to use this phenomenon to measure the temperature of a resistor at low temperatures, any secondary resistance at higher temperature must be extremely small compared to the thermometer resistance, and low noise amplification is needed to actually observe this signal at low temperatures regardless of the presence of a stray resistance [50].

\[ S_{V_{\text{total}}} = 4k_{B}(T_t R_t + T_s R_s) \]  
\[ S_{I_{\text{total}}} = 4k_{B}\left(\frac{T_t R_t + T_s R_s}{R_t + R_s}\right) \]

So the observed power of the thermometer loop is dominated for \( R_{\text{stray}} << R_t \). This is realized experimentally by minimizing the resistance in series with the thermometer element. This is accomplished by using a material that is superconducting in the temperature range of interest, or by making the resistance in series much smaller than the thermometer resistance.

The challenge in using Johnson Noise Thermometry at low temperatures is amplifying the small signal generated at low temperature so that it is not overwhelmed by other noise sources at room temperature. The small amplitude signals generated by a resistor at low temperature is challenging to measure using typical measurement techniques considering that a 1 Ω resistor at 100 mK will generate a voltage signal that is \( \sim 1 \frac{mV}{\sqrt{Hz}} \), with the corre-
sponding current noise $\sim 1 \frac{pA}{\sqrt{Hz}}$. Off-the-shelf low noise amplifiers introduce noise signals $\sim 4 \frac{nV}{\sqrt{Hz}}$, which will overwhelm the signal we are trying to measure. It is necessary to employ a sufficiently low noise amplification chain to measure the small signal generated by a resistor at low temperatures.

2.2 Adiabatic Demagnetization Refrigeration

To observe the low temperature phenomenon we must first reach low temperatures. We do this by using a liquid helium cooled Janis Research Adiabatic Demagnetization Refrigerator (ADR) cryostat. This cryostat uses a cooling cycle where a paramagnetic material is exposed to an increasing external magnetic field. While the magnetic field is increased, the paramagnet is held in thermal contact with a liquid He bath (4.2 K). As the magnetic field is increased, the spins in the paramagnet are forced to orient with the magnetic field. In the absence of a magnetic field, these spins are free to orient randomly within the material. Constraining the spins to align represents a restriction to a degree of freedom, which forces the entropy of the paramagnet to decrease. This decrease in entropy gives rise to a net heat flow from the paramagnet to the heat bath.

Raising the magnetic field to sufficiently high strength (3 T) for an extended period of time (1 hr) results minimizes the entropy of the system, while maintaining a constant temperature. By then isolating the paramagnet from bath, and decreasing the external magnetic field, the aligned spins are allowed to relax, causing an increase in the spin entropy. By allowing spins to reorient as the magnetic field is decreased, the kinetic energy of the lattice is transferred into reorienting spins. This results in a decrease in temperature of the paramagnetic material. Each of these phases in the magnetic cooling cycle can be observed in fig. 2.1.

By using two separate paramagnetic salts, Ferric ammonium alum (FAA) and Gadolinium Gallium Garnet (GGG) with magnetic ordering temperatures near 1 K and 50mK respectively, we are able to reduce the temperature of our experimental stages to below 1 K.
Figure 2.1: The magnetic strength and temperature of experimental stage plotted against time. Magnetic field is increased to a maximum with the GGG pill in thermal contact with liquid He bath. After the temperature of the experimental stage reaches equilibrium with bath, we isolate the paramagnetic salt from bath, and reduce the magnetic field strength. Base temperatures reached using GGG paramagnets typically reach $\sim 50$ mK.

Typical base temperatures observed in our cryostat are between 45 mK and 55 mK. From this base temperature, we are able to control and regulate the temperature of our experimental platform by controlling the magnetic field the paramagnetic materials are exposed to. This amounts controlling the temperature of our experimental platform by regulating the current flowing through the superconducting magnet.

Properly tuning the feed-back loop between magnet current and temperature of the experimental platform allows regulation within $\sim 100$ $\mu$K. At base temperature this error represents an uncertainty in temperature of one half percent of the base temperature. We are able to regulate the temperature of our experimental platform from nearly 55 mK up to slightly below bath temperature (4.2 K).
2.3 Noise Amplitude Measurement

Johnson Noise Thermometry relies on the ability to accurately measure the noise amplitude of a system. The two methods adopted for measuring the noise amplitude generated by our thermometer for these experiments will be discussed as follows. We will either report noise amplitudes that are measured from determining the “White Noise Value” in units of $\frac{V}{\sqrt{Hz}}$. We will also measure the noise amplitude as a voltage variance $V^2$ that is determined by calculating the variance in the output signal for a defined bandwidth corresponding to a “white” level.

Determining the “White Noise Value” (WNV) within spectral composition of a noise source can be determined by measuring the value for a sufficiently long time at a sampling rate that dictates the bandwidth of the measurement. By then taking observed $V(t)$ and converting this to frequency space by calculating the Power Spectral Density of the function, we are able to plot the relative signal composition as a function of frequency. The PSD of the measured signal is determined by first performing an FFT to project it into frequency space, then multiplying the magnitudes of the real and imaginary parts of the FFT. By performing these operations we are able to analyze signal composition in terms of frequency dependence. Typical PSD measurements of our connected thermometer are shown in fig 2.2 and indicate signal contributions not due to the thermometer at lower frequencies. We also see a very “white” composition from 1 kHz to 100 kHz. This indicates that the signal observed in this bandwidth is dominated by the Johnson noise of our thermometer. There is a clear shift in noise amplitude as the temperature of the cryostat is changed shown in 2.2. This indicates that the noise we observe is not dominated by another resistance at constant temperature.

Measuring the amplitude of signal contribution as a function of frequency allows to visualization of signal composition. Identifying and eliminating any extraneous signals that might compromise the integrity of the measurement can easily be implemented on the PSD data. We do this by identifying the amplitude of signal that best corresponds to a WNV,
and assume this amplitude is generated by our thermometer, excluding contributions from extraneous signals.

Figure 2.2: Power Spectral Densities of SQUID output at 2 base temperatures, by identifying the WNV of the spectrum and using this as the noise amplitude generated by the Johnson Noise Thermometer we can compare with calibration curve data to determine temperature.

The bandwidth of our measurement capability is determined by the sampling rate and duration of observation. With an infinite sampling rate, we would be able to accurately describe the spectral contributions to an infinite frequency. For a finite sampling rate we are constrained to an upper bound in frequency resolution dictated by the Nyquist frequency. This frequency is determined by the Nyquist theorem that sets the highest frequency resolved by a finite sampling period $T_{sample}$ is given by eq. 2.1 and is a consequence of a finite measurement rate.

$$f_{Nyquist} = \frac{1}{2T_{sample}} \quad (2.1)$$

The inability to resolve spectral contributions from frequencies higher than $f_{Nyquist}$ gives rise to aliasing effects. Aliasing effects occur where contributions from signals whose frequency is in excess of $f_{Nyquist}$ will appear as additional amplitude to frequencies below
$f_{Nyquist}$. Typically measurements of spectral contribution include some filtering to prevent aliasing effects from altering the measured values. We employ an analog Low-Pass 100 kHz filter to mitigate aliasing effects in our measurements.

![Figure 2.3: Signals of three frequencies are plotted against sampling number. While the frequencies are different, measurements of all three signals would be identical and result in identical FFT results.](image)

Alternatively, one can measure the noise amplitude by defining a bandwidth of measurement, making a large number of voltage measurements in time, and compute the mean voltage variance of N measurements of the voltage, given by eq 2.2. The variance of the voltage signal is related to the noise associated with the voltage measurement. In this case we are not interested in the mean value of voltage, although it should be $\sim 0$ V in our measurements. The uncertainty in temperature in this measurement is associated with the noise of the voltage variance. That is to say that our uncertainty in temperature is related to the noise of the noise of the voltage measurement.

$$\bar{\sigma}_{V} = N^{-1} \sum_{i=1}^{N} (V_i - \bar{V})^2$$  \hspace{1cm} (2.2)
The result of this type of measurement is equivalent to analyzing the spectral composition and determining the “White noise” level, and multiplying square of the white noise amplitude by the measurement bandwidth. These two methods are essentially the same, with the exception that one step utilizes Digital Signal Processing (DSP) techniques to facilitate signal analysis. This method has the advantage that it is not as computationally demanding, but suffers from any extraneous signal contributions. Spectral measurements to date have been performed on a Stanford Research Systems 770 network analyzer that has limited processing power, which has prompted us to migrate the measurement setup to a data acquisition (DAQ) card. I will maintain the convention that data analyzed using a spectral analysis will report values of noise amplitude in $\frac{V}{\sqrt{Hz}}$, and those measurements of noise amplitude made by observing the mean variance of the voltage signal will be reported in units of $V^2$. Performing DSP functions on the computer allows much faster processing times, resulting in less time per noise amplitude measurement. By first verifying that the measurement bandwidth is free of any extraneous signal we are able to quickly measure the noise amplitude of the thermometer without analyzing the spectral composition.

The noise amplitude determined by measuring the variance of the voltage for a defined bandwidth, or identifying the “white” noise amplitude within the spectrum amount to an equivalent measurement. We have observed the noise amplitude using both techniques, and adjusted the voltage variance data to reflect the bandwidth of measurement. Comparing observations of the noise amplitude using both methods we see that the noise amplitude observed in either measurement technique results in an overlapping data set, as is indicated in fig. 2.4. With the ability to accurately measure the amplitude of noise generated by the thermometer we are able to interpret the temperature of the sample to be measured. JNT is an observable that depends only on the resistance, and temperature of the resistor as variables, and as a result is compatible as a primary thermometer.

Utilizing JNT as a primary thermometer requires separate measurements of all aspects of the amplification chain, as well as the resistance of the thermometer at relevant tempera-
Figure 2.4: Measurements of the amplifier noise level using the “white” noise amplitude observed by the SRS 770 and by measuring the variance of the output voltage to determine the apparent WNV. The bandwidth of the measurement was 99 kHz (1 kHz-100 kHz).

Slight changes in the effective gain of the amplifier chain, variations in noise environment, and other factors can place unnecessarily burdensome constraints on the measurement process, and introduce unaccounted errors in measurement [44].

JNT is also compatible for use as a secondary thermometer. Using JNT as a secondary thermometer can lend the advantages of another thermometry technique to speed up the determination of the temperature of Johnson thermometer. This technique can speed up the process of determining parameters of the experimental thermometer, it also carries with it the systematic errors associated with the reference thermometer. This can implemented by connecting the experimental thermometer (Johnson Noise thermometer) at the same temperature as the reference thermometer and measuring the noise amplitude observed using JNT as a function of reference thermometer temperature. This process amounts to calibrating the our thermometer to a repeatable and stable thermometer. With observed noise
vs temperature $S_V(T)$, we are able to determine the temperature of the membrane as a function of observed voltage variance. This becomes critical when we need to measure the difference in temperature between our experiment and bath temperature.

### 2.4 SQUID Amplification Chain

The challenges faced by amplifying the small signal generated by our thermometer for observation at room temperature have largely been addressed by advances in DC Superconducting Quantum Interference Devices (SQUIDs). We have been able to couple the signal from the thermometer to the input of a two stage SQUID amplification chain of suitably low noise. Two stage SQUID amps, have been successfully applied to many applications requiring extremely sensitive measurement techniques [13].

A two stage SQUID amplifier consists of many individual SQUIDS connected to allow significant amplification of the signal, transduction of the input signal, and impedance matching to room temperature electronics. Amplification schemes involving SQUIDs come in many varieties, but all include a single (input) SQUID connected to many other SQUIDs (array). By using a two stage SQUID amplification chain we are able to match the low noise values of a DC Superconducting Quantum Interference Device (SQUID) $\sim \frac{2 \Phi_0}{\sqrt{Hz}}$ to the signal generated by our thermometer $\sim 1 \frac{pA}{\sqrt{Hz}}$.

#### 2.4.1 DC SQUID

Connected to the experimental thermometer is a DC SQUID referred to as the input SQUID. A DC SQUID is essentially a quantum limited magnetic flux to voltage transducer. Connecting the thermometer in series with a superconducting loop that is inductively coupled to the SQUID, we are able to directly couple the current in the superconducting circuit into the DC SQUID loop. The current flowing through the input loop of the SQUID directly couples the current into a flux by the Biot-Savart law where the current in a loop generates
a magnetic field. By measuring the noise amplitude of the Squid with the input coil open, and comparing that data with measurements with a thermometer $R_{\text{therm}} \approx 20 \text{ m} \Omega$.

![Conceptual diagram of a DC SQUID with a plot of the observed output voltage plotted as a function of incident flux on the superconducting loop [40].](image)

When the current passing through either Josephson Junction is slightly less than $I_c$, the application of a small magnetic field normal to the superconducting loop induces a change in the current flowing through either branch of the SQUID. One branch will have a current greater than the critical current, while the other has a suppression of current passing through the other junction. The branch with $I > I_c$ looses local superconductivity at the Josephson Junction and becomes resistive. At this instant, it is energetically favorable for the "superconducting" loop allow a flux quantum to be trapped within the center of the circle. This effect is disallowed for a truly superconducting loop. The inclusion of a $\Phi_0$ of flux to thread the superconducting loop causes a reversal of the induced super-current. Immediately following the inclusion of a $\Phi_0$ within the superconducting loop causes the induced super-current to flow in the opposite direction.

Further increases of the local magnetic field will then reduce the magnitude of the super-current. This is the source of the periodicity of $V_{\text{squid}}(\Phi)$ or the $V - \Phi$ characteristic, and establishes the mechanism by which a SQUID is able to behave as a quantum limited flux to voltage transducer. The difficulty in using a SQUID as an amplifier is that the $V-\Phi$ relationship is inherently periodic, and as such makes a non-linear amplifier. This improves linearity of the amplification chain, but it is possible to further improve the dynamic range.
and responsivity of our amplification chain[14].

Figure 2.6: \( V-\Phi \) characteristic of the input Squid showing period of 1 \( \Phi_0 \) periodicity. Small changes in current in the input coil cause shifts in flux density incident to the SQUID, resulting in a voltage developed across the SQUID.

The primary role of the input SQUID is to take the observed current circulating the thermometer loop and convert this signal into something that can be amplified. This is accomplished by fabricating an input coil over the top of the SQUID itself. By driving the current generated from the thermometer through the input loop, the local magnetic field normal to the SQUID loop is proportional to the current circulating the thermometry loop. This configuration is referred to as current sensing.

The small fluctuations in local magnetic field observed by the SQUID then affect the resistance of the Current biasing line. As the magnetic field increases, the effective resistance of the current biasing lines will change periodically to integer \( \Phi_0 \). By carefully choosing the input coil inductance, we can provide a very sensitive coupling between changes in resistance of the bias lines and the current circulating in the thermometer line while maintaining measurement bandwidth. The bandwidth of the thermometry circuit is determined by inductive filtering, and is determined by eq 2.1. For values of \( R_{therm} =40 \text{m} \Omega \) and \( L_{input} =1500 \text{ nH} \), the resulting bandwidth of 167 kHz. This matches our ability to measure, as the bandwidth of our measurement devices are limited to \( \sim 100 \text{kHz} \).
\[ BW = \frac{2\pi R_{\text{therm}}}{L_{\text{input}}} \] (2.1)

Voltage bias biasing the SQUID by placing a bias resistor in parallel with the SQUID forces changes in current passing through the Squid, as resistance of the Josephson Junctions change. This way we have coupled the very small changes in current on the thermometry loop to changes in current passing through the SQUID. The changes in current passing through the SQUID are larger than the small fluctuations passing through the input coil.

By carefully selecting the inductance of the input coil as well as the mutual inductance between SQUID and input coil, we are determining the sensitivity of the coil. The resistance of the SQUID bias line is on the order of 10’s of mΩ. The resistance of the Josephson Junctions will not vary greatly, so the sensitivity of the input SQUID is governed by the inductances. It is important that the input inductance is sufficiently high to allow the current noise from the resistor to generate changes in flux density on the SQUID to ensure we are not introducing other noise sources into our measurement at the lowest temperatures, but small enough to enable fast measurements of the temperature that are possible with a high bandwidth.

2.4.2 SQUID Array

By voltage biasing the first stage SQUID, and passing the input SQUID bias leads through the input coils of several other SQUIDS in series, the slight changes in the biasing current of the initial SQUID are then coupled into the input coils of an array of squids. By doing this, small changes in the first stage SQUIDs input coil cause fluctuations in the current passing through the input coils of the SQUID array.

Each SQUID in the array has a corresponding \( V \cdot \Phi \) response, which dictates the change in the voltage as a result of changes in current passing through the input coil of the array SQUIDS. Connecting the outputs of each of the array SQUIDS in such a way that the output voltages are additive, we are able to again amplify the initial current fluctuation into
a voltage changes large enough to be monitored by room temperature electronics.

The fully realized two stage SQUID amplification chain wiring diagram is shown below in figure 2.8, and suitably matches the low noise values of SQUID electronics to the low signal generated by the thermometer. This arrangement also suitably amplifies the signal to be observed and easily measured at room temperature.

Figure 2.8: Wiring diagram for SQUID amplification chain [9].

The Voltage measured at room temperature is then a product of the current circulating...
in the input loop, the input SQUID transfer coefficient that determines the current to flux quantum relation dictated by the squid amplification chain and the feedback resistor, as well as the gain setting on the warm electronics. Therefore the measured voltage is proportional to the small fluctuations of current circulating within the thermometer loop.

2.4.3 Flux Locked Loop

A two stage SQUID amplification chain is able to amplify the small signal observed at low temperatures to be measured at low temperatures, but this alone does not address the inherent nonlinearity of the input or array SQUID’s response. As is the case with many nonlinear amplifiers, we can improve the linearity of response, by using an active feedback loop. The purpose of a feedback loop is minimize the change in flux density around the SQUID. This can be accomplished by coupling a feedback inductor to the SQUID loop, and passing a current proportional to the current present in the input coil. A feedback loop of this type is referred to as a Flux Locked Loop (FLL) and has the advantage of dramatically improving the linearity of the $V - \Phi$ response while extending the range of SQUID response. By engaging the FLL at the most linear of the $V - \Phi$ response (typically the most sensitive point) small deviations in flux density normal to the SQUID cause proportional changes in
the observed voltage, which are then fed back to the SQUID via the feedback loop. We are able to observe the total change in flux density by measuring the current (voltage) in the feedback loop.

Generally speaking a feedback loop works to make a measurement, compare that with a target value, and introduce a change to the system you are measuring that is intended to minimize the difference between what you measured, and what you want. This is a sufficiently vague description that applies to many applications. Using a feedback loop to linearize the $V$-$\Phi$ response of the SQUID amplification chain behaves in a similar fashion. We select a point $V(\Phi)$ on the $V$-$\Phi$ response, and use this as our working point. Small changes in the flux environment of the SQUID result in small changes of the output voltage. The measured voltage is now different from the working point, so the response of the feedback loop is to make a proportional alteration to the flux environment of the input SQUID. This small change is intended to maintain a constant flux density across the SQUID loop.

A perfect flux locked loop would instantly respond to any change in current present in the input SQUID by generating current in the feedback loop that maintains a constant flux density across the SQUID loop despite changes in the current circulating through the input coil. FLL is engaged by selecting the point in the $V$ -- $\Phi$ response that will serve as the point of reference. Deviations in the flux environment observed by the input SQUID result in proportional currents being passed through the feedback coil to minimize the deviation.

The FLL electronics work by trying to zero the voltage measured from the output of the measurement chain to a reference point established by the user. By aligning the zero crossing of the $V$ -- $\Phi$ response will engage the feedback loop at this point. Typically we will attempt to null the effective flux at the working point, so that the SQUID is less likely to deviate a multiple of $\Phi_0$ and potentially shift to a different working point. If the FLL electronics temporarily loses lock on a working point it is possible that the feedback electronics will re-engage the lock point at $\frac{n\Phi_0}{2}$ with $n$ an integer. Due to the periodicity, re-locking at $n\Phi_0$ should result in no noticeable difference in effective gain, but locking on a
half integer shift in $\Phi_0$ would result in a change in effective gain of the SQUID amplification chain. This is clear by looking at the slope of the $V - \Phi$ characteristic. by examining the magnitude of Voltage transfer coefficient at -0.5 $\Phi_0$, 0 $\Phi_0$, +0.5 $\Phi_0$. We see that the difference in magnitude of voltage transfer coefficient for -0.5 $\Phi_0$ and +0.5 $\Phi_0$ are very close, while the difference between -0.5 $\Phi_0$ and 0 $\Phi_0$ is more substantial. This amounts to an asymmetry between the downward slope and upward slope of the $V - \Phi$ characteristic.

Using a FLL minimizes the deviation from the SQUID working point and extends the dynamic range of the input SQUID. Non-ideal feedback loop operation introduces feedback errors which are to be avoided. Non-ideal loop conditions include limiting the bandwidth of the feedback response, which causes the feedback mechanism to be too slow to respond to rapid changes of current in the input coil, as well as introducing nonlinear gain effects which are possible by too large of a feedback coupling. This can result in “ringing” or high frequency resonances being observed in the SQUID output. Proper tuning of the feedback loop retains operational bandwidth of the SQUID and improves the linearity of SQUID response. Carefully determining the appropriate values for $R_{f0}$ and integrating Capacitor is necessary for proper operation of the feedback loop, as these values determine the effective P and I terms in the feedback Loop. These values have impacts on the measurement bandwidth, and stability of the lock point.

Choosing the working/lock point for the feedback electronics is done by ”tuning” the SQUID amplification chain. Tuning the amplification chain amounts to adjusting the bias current and flux offset for both the input and array SQUIDs to reach a desired working point. The working point will contribute to defining the effective gain, stability of feedback electronics, and noise level of the amplifier chain. If the sensitivity/gain of the amplification chain is too high or poorly matched to the feedback electronics, maintaining a lock on the working point will become unstable. If the feedback electronics are unable to maintain a constant lock on the working point, it is possible to introduce shifts in working point.

Shifts in the working point can be difficult to observe, but would result in the $V-\Phi$ re-
sponse being shifted by some amount along the $\Phi$-axis. V-Phi shifts from one working point to another, the current to voltage conversion function of the amplifier chain can shift significantly. This has been observed a number of times during data acquisition as a calibration curve that will suddenly change slopes and offset by a multiplicative factor. Typically this value is small, but significant enough to cause problems in measurements. The noise amplitude of the thermometer has not changed; rather the effective gain of the amplifier chain has changed by some small amount. It is certainly possible to carefully tune the input and array SQuids, so that the V-$\Phi$ provides a stable working point, and the SQUID will not temporarily lose lock to regain at a slightly different working point indicated in fig 2.10. The two arrows indicate a shift in working point of $\Phi_0/2$, and have different magnitudes for slope at these two points.

Figure 2.10: The Flux Locked Loop will attempt to null the difference in voltage between the voltage output from the amplifier chain and zero. Two potential working points are indicated by arrows. The difference in slope of these two points represents a change in effective gain of the amplifier chain.

We have observed shifts in working point while measuring noise amplitude vs temperature. These events seem to be affected by the EM environment, as turning on and off electronics, or strong EM signals in the room can induce these shifts. Below, in fig 2.11 is an observation of the sudden change in noise amplitude. This observation was a result of making/breaking an electrical connection to room temperature electronics. Any transient
signal passed down to cryogenic temperatures may cause a sudden shift in FLL working point.

Figure 2.11: Observation of a "jump" in observed noise amplitude vs temperature. This behavior has been observed at seemingly random times at different temperatures. The slightly difference in slope before and after the jump is an indication of a shift in effective gain amplitudes of the SQUID amplification chain.

2.4.4 Details of Thermometry Connections

High purity Cu films were patterned using lithographic techniques to form resistive elements on the central membrane that serve as heater and thermometer. Measured resistances for the thermometer are between $40 \, \text{m} \Omega < R_t < 60 \, \text{m} \Omega$ corresponding to a thermally generated current noise of $9.59 \frac{\text{pA}}{\sqrt{\text{Hz}}} < I_{\text{noise}} < 11.7 \frac{\text{pA}}{\sqrt{\text{Hz}}}$ at 100 mK. We have measured the resistance of all heaters and thermometers down to $\sim 5$ K. Typical observations of $R(T)$ for heaters and thermometers is shown below in fig 2.12.

On chip electrical leads are made of a 400 nm Nb thin film that runs the length of the supporting Si$_x$N$_y$ legs to make electrical contact with heater and thermometer. Heaters and thermometers are formed from a 420 nm Cu film. Normal metals at low temperatures have an electrical resistance that decreases with temperature as the electron-phonon scattering rate decreases. Resistance at higher temperatures is dominated by electron-phonon interactions, but as the temperature decreases, the e-p scattering rate drops. In real metals this has
a significant implication that at a certain point the scattering rate, or the mean free path of charge carriers is then dictated by impurity scattering. When impurity scattering becomes the dominant scattering mechanism, the resistance is no longer dependent on temperature, and is instead determined by the purity of the film. This is the case with our Cu heaters and resistors.

We have measured the resistance as a function of temperature from room temperature down to 5 K, using a "quasi" four wire technique. By connecting current and voltage leads to the Nb bond pads and leads, we are able to make a 4 wire measurement of the resistance of the Nb, and Cu paths. What we have observed is that the resistance decreases until the Nb leads go superconducting. At this point the resistance suddenly drops, and we are left to measuring the resistance of the heater or thermometer only. As temperature is changed between 5 K and 7.9 K we see no significant change in resistance measurements.

Figure 2.12: Measured film resistance as a function of temperature. This measurement includes Nb lead resistance contributions for all $T > T_c$, which is $\sim 7.9$ K for Nb. The unexpected two step transition observed during the heater measurement is attributed to a discontinuous transition of the Nb leads on the thin legs becoming normal before the portion of the leads supported by the Si chip itself. This has been observed by generating relatively large excitation currents to accurately determine the resistance of the resistive films.

With the heater and thermometer resistance dominated by impurity scattering, we have created a resistive element that is insensitive to temperature changes. This is very impor-
tant for the application of JNT at low temperature. This allows the noise amplitude to be determined by the temperature alone at low temperatures.

Connecting our amplification chain to the actual device with thermometer fabricated out from 420 nm Cu film, and is connected to the input SQUID by Nb traces, and either Al or Nb bonding wire. Both Nb and Al are superconducting below $\sim 1.1$ K with Nb leads undergoing a superconducting transition at $\sim 7.9$ K. By creating a superconducting thermometry line, we are able to minimize extraneous noise signals. Connections between the input SQUID and the SQUID array are made using Cu/Ni coated Nb wires, further preserving the integrity of our signal from resistive losses. Connections between the experimental chip and wiring are made using a Pd/Sn coated standard Cu PCB. These traces remain superconducting until $\propto 2.8$ K. We observe superconducting transitions in our wiring as sharp discontinuities in observed noise amplitude vs temperature, and observe subtle shifts in the $V-\Phi$ curve as a result of changing resistances in the low temperature circuit. This gives us an effective temperature range from 50 mK to 2.8 K.

Measurements of the spectral composition of the observed signal as well as the noise amplitude vs temperature have been made with the input coils to the SQUID disconnected. This provides a baseline measurement indicating the noise generated by the SQUID itself. Comparing these measurements with those conducted with the thermometer connected to the input coil indicate a nearly 100x increase in noise amplitude. This tells us that the noise observed with thermometer attached is dominated by the signal generated by our thermometer.

We have compared the noise generated from the thermometer of several different devices, and have observed that, despite small differences in working points and thermometer resistances, we are able to generate very similar noise vs temperature curves. This tells us that we have a working thermometer, and that it is well described by the Johnson-Nyquist theorem. We achieve reliability between successive cool downs, both with cracking vacuum and within the same supply of liquid helium. Below in fig.2.14 we see that the observed
Figure 2.13: Directly comparing the observed noise amplitude of the amplification chain whose input coil is open to the same SQUID with the thermometer connected. We see that the noise amplitude observed with the thermometer connected is nearly 30x higher at the lowest temperatures.

noise amplitude vs temperature is observed over the course of 1 week. As long as the SQUID working point remains the same, the noise amplitude is a well defined and predictable function of bath temperature.

Figure 2.14: Comparing noise amplitude vs T over the course of several days from 50 mK < T < 3.2 K. Observed discontinuities in $V^2$ vs T result at T=1.1 K and 2.8 K corresponding to superconducting transitions of Al (bonding wire) and Pb/Sn solder (Circuit board).

If we compares the calibration curves taken for several devices with slightly different
working points for the amplification chain, and slight differences in the thermometer resistance, we see that the noise amplitude is highly repeatable, and is nearly the same for each device. Slight differences in slope are attributable to differences in the thermometer resistance, and small changes in the SQUID working point. The noise vs temperature curves of three different geometries and four devices is plotted in 2.15. The differences in geometry correspond to a nearly 200x difference in the thermal conductance of the isolation structure, and is an indication that the slight deviation from linearity at low temperature is not due to the presence of stray heating load.

Figure 2.15: Comparing the noise amplitude vs T behavior for three sample geometries. The low temperature deviation from linearity seems to be independent from sample geometry. This is an indication that the deviation is not due to unaccounted heat load on the sample.

Without measurements of the input sensitivity, it is difficult to calculate the theoretical voltage noise that should be observed at room temperature, but we can take the feedback current sensitivity and scale this value up by the ratio of turns to come up with an approximate input current sensitivity. By using this we can come up with an estimate of the theoretical noise amplitude to compare with the observed noise amplitude. Calculating the current circulating through the feedback loop during the process of tuning the SQUID, we can calculate the feedback mutual inductance. This value should be related to the input coil
mutual inductance by virtue of the constant geometries. The ratio of the feedback mutual inductance to input mutual inductance should be slightly less than the ratio of the turns in the input coil to the feedback coil. This should be the case because the geometry of the SQUID is identical, and the flux density generated due to current circulating the two loops should be proportional to the number of turns.

By calculating the feedback sensitivity and calculating an estimate for the input sensitivity, as well as observing the effective gain amplitude of the SQUID amplification chain we can generate an estimate of the voltage noise amplitude that should be observed as a result of the current noise circulating the thermometer loop.

In fig.2.15 the orange squares labeled 13-06 correspond to data collected in the presence of stray heating load that was corrected in the red data of the same name. Qualitatively these plots are very similar, however there is a more pronounced deviation from linear at the lowest temperatures. Because we are attempting to perform very sensitive measurements of the thermal conductance of these membranes it is critical that we know the heat load present on the sample.

By simulating the effect stray heating would have on these devices at low temperatures, we can determine whether the ”typical” deviation at low temperatures is due to the presence of stray heating, or whether it is due to some other effect. If we assume that the observed thermal conductance values measured for each of these devices is correct, we can calculate the temperature of the thermometer in the presence of a stray heating power of different magnitudes. Comparing the simulated noise amplitude vs temperature curves determined using this method for the three geometries, we should be able to determine whether the low temperature deviation is due to stray heating.

We can also compare the deviation from linearity between the three geometries and observe that the magnitude of deviation is similar throughout the temperature range for each of the devices, despite nearly 2 orders magnitude difference in thermal conductance. This also indicates that something other than stray heating is causing the low temperature
Figure 2.16: Noise amplitude vs T data observed for two geometries with curves simulated for the presence of stray heating power.

deviation from linearity observed in the noise amplitude vs temperature plots.

Our amplification chain is dominated by the noise on the input SQUID whose flux noise amplitude is \( \sim 1 \frac{\mu \Phi_0}{\sqrt{Hz}} \). This intrinsic SQUID noise is caused by shot noise across the Josephson Junctions as well as Johnson Noise generated by resistive shunts across the Josephson Junctions themselves. As the resistance of the junctions is typically quite small (\( \sim 10 \mu \Omega \)) the noise amplitude of these resistors should be very small compared to the signal we are measuring.

We realize a superconducting input loop with the thermometer by using Al or Nb wire to make connections between the SQUID input coil and thermometer. However, for Al, the \( T_c \) of nearly 1.1 K dictates that the wire will become another source of noise for higher bath temperatures. In order to account for this, we are able to model the input loop as two noise sources where the bonding wire is held at a bath temperature \( T_{bath} \), and the noise amplitude generated by the thermometer will change as a function of the membrane temperature. Therefore, the membrane temperature in the presence of a stray resistance is given by eq.2.2, with \( I_{noise} \) proportional to the voltage observed at room temperature, \( R_s \) and \( R_t \) representing the stray and thermometer resistances respectively.

\[
T_m = I_{noise}^2 \frac{(R_t + R_s)^2}{4k_B} - \frac{T_{bath}R_s}{R_t}
\] (2.2)

With a accurate measurement of the noise amplitude as a function of temperature before
and after the superconducting transition, we are able to determine $\frac{R_L}{R_s}$. This is accomplished by making an accurate measurement of the noise amplitude as a function of temperature below the superconducting $T_c$ of the bonding wire, and above the transition temperature with $T_m = T_b$, or with the membrane and low temperature stage at thermal equilibrium. The difference in the slope of $V_{\text{noise}}(T)$ above and below $T_c$ determines $\frac{R_L}{R_s}$. In the case that the connections between the input SQUID and other cryogenic electronics is altered by the same superconducting transition, the working point and tune conditions of the first stage SQUID will also be affected by the change in resistance. In this case it is necessary to tune the SQUID electronics above and below the $T_c$ so that the $V - \Phi$ characteristic is identical. If the working point of the SQUID is slightly altered by an increase in temperature, the observed $\frac{R_L}{R_s}$ will be systematically altered, causing a substantial error in the determination of $T_m$.

Another complication with the superconducting transition of Al bonding wire is that the relative resistance of the Squid biasing line will shift in relation to the bias resistor. This serves to alter the current flowing though the SQUID biasing circuit. This means that it is necessary to re-tune the input SQUID, to produce the exact $V - \Phi$ relationship that was observed for temperatures less than the superconducting transition temperature, otherwise, the effective SQUID gain $\frac{dI}{dV}$ will not be the same. This introduces a significant source of error to membrane temperature measurements above $T_c$. Here we see the expected drop in measured voltage variance as we pass through the superconducting transition temperature of the Al bonding wires. Although we are introducing an additional noise source, the amplitude of the overall noise signal will decrease because the existence of the additional resistance will dissipate some of the signal generated by the other noise source.

We determine the time necessary to establish equilibrium by monitoring the noise amplitude as a function of time during a shift in bath temperature, and fitting the temperature response of our thermometer to an exponential. By waiting a number of measured time constants beyond any change in $T_{\text{set}}$ we ensure that thermal equilibrium is established between
bath and thermometer. In practice the measurement of a calibration point is interspersed with various heat applications, and measurements are performed from 3 min to 40 min after perturbation of the temperature set point. Scatter on the points measured across such a large time is within the error of the measurement, and typical times needed to thermally equilibrate our sample are ~45 sec depending on the thermal contact between our device and the sample mount.
Chapter 3

Thermal Conductance Measurements

3.1 Introduction

Thermal conductance \( (G) \) is the measure of an object’s ability to conduct heat in the presence of a temperature gradient, and is dictated by the shape, composition, and nature of the temperature gradient within the object. Two objects of the same material and different geometries will have conductances that differ due to the geometric differences. For bulk materials with similar temperature gradients, the thermal conductance should be related by the material properties of the object. We can describe the material property dictating thermal conductance as the thermal conductivity \( (\kappa) \), which is given by eq. 3.1. This quantity represents the intrinsic material property governing the transport of heat through the material. Using the thermal conductivity we are able to accurately predict the conductance of two objects of similar composition that vary in geometry.

Measurements of thermal conductance are performed by observing the heating power needed to develop a given temperature difference through the medium. For a temperature gradient generated in one direction, the thermal conductance \( (G) \) is given by eq. 3.2, and
is typically determined by the properties of the material ($\kappa$) and the specific geometry of the object. It is then useful to measure the geometry independent quantity of thermal conductivity, which is given by eq. 3.3. These relations hold for nearly all experimental conditions.

\[
\kappa = -\frac{\vec{Q}}{\nabla T}
\]  

(3.1)

Predicting and controlling the thermal conductance through an object particular importance in the design of sensitive measurements at low temperatures. Understanding the transport properties of reduced dimensional systems at low temperatures is an important step in realized the tremendous resolving power of low temperature detectors. Studying thermal transport at low temperatures probes the nature of transport processes and improves the description of behaviors observed in TES-like devices. We shed light into the expected thermal conductance as lateral dimension approaches the dominant wavelength of a phonon, and better understand how heat is conducted in material at low temperature.

\[
G = \frac{\Delta P}{\Delta T}
\]  

(3.2)

\[
k = K \frac{L}{A_\perp}
\]  

(3.3)

All materials conduct heat, though the quantity and the mechanism of transfer vary depending on whether the material is a metal, superconductor, semiconductor, or an insulator, and the temperature of the material. Insulators conduct heat through lattice vibrations known as phonons. Phonons are quantized vibrational modes within the lattice, and a well defined density of states at a given temperature. Phonons also transfer heat in metallic samples as well, although the dominant heat carriers in metals are electrons. Thermal conductivity and its temperature dependence vary considerably across different materials and between different samples of the same material due to a variety of properties including crystalline structure, differing grain sizes in polycrystalline materials, the inclusion of de-
fects, alloy concentration, and scattering of the excitations responsible for heat transfer. This variation along with the importance of understanding $\kappa$ in thin films for technological applications such as low temperature detectors, make thermal transport at low temperatures an important topic of research.

3.1.1 Thermal Conductance of Bulk materials

Treating thermal transport of a material as though transport is a result of a kinetic gas, thermal conductance should be given by the amount of energy stored in a heat carrier the number of heat carriers, the rate at which the carriers travel, and the distance they travel. For bulk materials this is related to the thermal conductivity by eq. 3.4 where the mean free path ($l$) of the heat carriers, the specific heat of the material ($C$) and the group velocity ($v$) of the carriers. This equation accurately describes the thermal conductivity of metals, insulators, and semiconductors. This simple model is capable of describing heat transported through a material for very different carriers.

$$\kappa = \frac{cvl}{3}$$ (3.4)

Dominant heat carriers in different materials dictate the thermal conductivities of the material. For instance the thermal conductivity of a metal at at room temperature is very different from the thermal conductivity of an insulator at room temperature. This large difference in thermal conductance is a result of the differences in dominant heat carrier and mean free path of the carriers in the two materials. Electrons in metals are the dominant heat carrier, while phonons dictate heat propagation in insulators.

Thermal transport in bulk metals is described through the Wiedemann-Franz law [29]. Because heat is conducted by electrons in a metal, the thermal conductance is related to the electrical conductance, and is given by eq. 3.5. This description of thermal conductance in a metal is valid for bulk metals, and holds for many thin film metals.
For a bulk crystalline insulator near room temperature phonons propagate heat across along a thermal gradient. The conductance is dictated by the mean free path as indicated in eq. 3.4. Near room temperature the mean free path is limited by phonon-phonon Umklapp (inelastic phonon-phonon) scattering which results in a short mean free path. As the temperature of the insulator drops below \( \approx 20 \text{ K} \), phonon energies and populations decrease making these scattering events less likely increasing the mean free path within the material. Relative scattering rates of temperature dependent quantities will result in a temperature dependent \( G \), while scattering from surface defects, impurities, and lattice irregularities remains constant. 20 K is the region in which the phonon mean free path is limited by scattering from these defects, but the precise temperature at which this transition happens will depend on the purity and crystallinity of the object. For temperature much lower than the Debey temperature(\( \Theta_D \)) the heat capacity of an insulator is well described by the Debey model. The Debey temperature refers to the lowest temperature all available lattice excitations are active. This is a characteristic temperature for observing lattice properties of a material.

\[
G = \frac{\pi^2}{3} \left( \frac{k_B}{e} \right)^2 T \quad (3.5)
\]

\[
x = \frac{\hbar c_n}{2Lk_BT} \quad (3.6)
\]

\[
\frac{C_V}{Nk_B} = 9 \left( \frac{T}{\Theta_D} \right)^3 \int_0^{\Theta_D/T} \frac{x^4e^x}{(e^x-1)^2} dx \quad (3.7)
\]

\[
\frac{C_V}{Nk_B} \approx \frac{12\pi^4}{5} \left( \frac{T}{\Theta_D} \right)^3 \quad (3.8)
\]

This predicts that the heat capacity of an insulator varies as \( T^3 \) at low temperatures, and has been confirmed experimentally for a number of insulating materials. This accurately
describes the heat capacity and thermal conductance for a bulk phonon mediated processes at low temperatures. This picture will change as dominant scattering mechanisms change [57].

3.1.2 Amorphous Materials

For a dielectric material at low temperatures phonons dictate thermal transport properties, and predominantly scatter at lattice irregularities, and surfaces. Including additional lattice defects serves to shorten the mean free path of the phonon population. At low temperatures in amorphous materials, the existence of Two Level Systems (TLS) has been used to explain a number of unexpected behaviors. TLS’s have been very successful in explaining a linear dependence on heat capacity at low temperature, which is very different from the expectation that the heat capacity would go as $T^3$ from the Debey model. TLS’s interact with the phonon population at temperatures for which the energy carried by a vibrational mode is on the order of energy splitting of the TLS. In this model, TLS’s are coupled to the phonons, and are able to scatter, absorb and emit vibrational excitations within the lattice.

Simply speaking a two level system is a description of a system where the energy difference between two configurations are close in energy with only two available states. In amorphous materials, perturbations with energies that are close to the energy difference of a TLS can cause it to jump from configuration to the other. The classic TLS is a spin oriented either parallel or anti-parallel to a very weak external magnetic field. There are two configurations that are rather close in energy, but the spin is only allowed to reorient with a specific excitation energy. There is compelling evidence that TLS’s are present in Si$_x$N$_y$ and other amorphous materials. Slight configuration difference between two orientations of unsatisfied bonds within the “lattice of the amorphous materials, or local minima in the potential of the lattice can establish a foundation for TLS’s in amorphous insulators. Assuming these slight differences between two states acts as a TLS, we have introduced an alternative heat capacity within the system, which does not act as a heat carrier [32][45].
As these TLSs are sensitive only to a specific excitation energy, TLSs do not play a part in the heat capacity of SIN at higher temperatures, and will only come into play when there is a population of excitations with sufficiently low energy to interact with the TLSs. This can be the case as temperature is decreased and the energy carried in a lattice vibration drops with the temperature. If the TLS energy splitting is larger than the available excitation energies, TLS’s will not serve as a scattering site for lattice vibrations. By changing the temperature to allow phonons of sufficient energy TLS interaction and scattering is then opaque to certain Phonon modes, while remaining transparent to others [21][8].

3.1.3 Finite Size Effects on Transport

This is an oft encountered effect while working in low temperatures and in small structures. The expectation that the mean free path of any heat carrier will increase as the temperature decreases is based on the supposition that the carrier scattering rate decreases with temperature. While the scattering rate decreases with temperature under typical circumstances as populations and energy levels drop, other scattering rates are invariant in temperature. Obvious examples of temperature independent scattering sources include lattice imperfections (crystalline or defect) and the surfaces of the object itself [10].

As the dimension of an object of any composition (insulating, metallic, amorphous, etc.) the dimension of the object can play an important or dominant role in limiting conductance through a material. A bulk metal for instance will have a well described density of states, thermal conductivity and mean free path as a function of temperature. It may be the case that such a material has dimensions that are substantially larger than the mean free path at room temperature, but we decrease the temperature and the bulk mean free path is increased, the finite size of the sample can limit the actual mean free path of the heat carriers. In this way the reduced dimensionality of the sample can become a dominant scattering event for heat carriers propagating along the sample.

Such can also be the case in an insulating material, where the dominant heat carrier is
lattice vibrations. Bulk calculations for the mean free path can be many times the smallest dimension of the material, and can lead to scattering dominated by temperature independent scattering sites. Therefore at low enough temperatures and in geometries of finite dimension, we begin to see the reduction in geometry alter the transport properties of the material. In this case it is less useful to think of the observed thermal conductivity as a material property because the geometry of the sample is dictating the thermal conductance [19]. As the geometry becomes smaller, and the mean free path of carriers increases, we observe considerable discrepancies between observation and values predicted using eq. 3.3. This is a result of the so called “finite size effects”. In this regime the object will deviate significantly from predicted values as the object no longer behaves as its composition would indicate. The nature of the deviation from “normal” is an indication of how geometry is dictating transport through the object [12].

By substantially decreasing the dimension of the object, we influence whether the dominant scattering mechanism is material dependent at a given temperature. Observations of this effect have been made in thin films, nanowires, and quantum dots [48]. These special cases defy description by bulk values of identical material constitution. In these reduced dimensional systems, surface interactions and finite size effects become important and can dominate at different temperatures. The implication is that two films of identical composition and temperature gradient can often result in very different observed thermal conductivities because the geometric dependence is built into the dominant scattering mechanisms.

When the mean free path of a phonon in a bulk material is much larger than the smallest dimension it is not immediately clear what the implication is. Limiting the thickness of the material invalidates the assumption of a quasi continuous Density of state. By forcing discrete lattice excitations within the material, calculations of the Density of states changes. When the available excitations within direction are restricted, the dimensionality of the phonon gas is reduced from 3D to 2D. At this point lattice excitations no longer behave as bulk phonons, and this results in changes to the thermal conductance. For materials with
constrained geometries we describe the phonon population as reduced in dimensionality. Phonons within a bulk material are well understood, and behave unrestricted in three dimensions. We refer to this phonon population as a 3-D phonon gas. Phonons in a restricted geometry exhibit reductions in dimensionality to 2-D or 1-D [28].

3.1.4 Thin Amorphous Films

Much effort has been made in understanding the consequences in thermal properties when the dimensionality of phonon gas is influenced by restricting geometry [33][22][59]. Experimental observations of a-$\text{Si}_x\text{N}_y$ membrane transport phenomenon have been described by a number of models that can explain some of the thermal properties observed at low temperature [61][3]. Predictions for $G$ in thin films exhibiting 2D behavior indicate that $G \propto T^2$, but a more comprehensive study of transport including effects on coupling between shear, longitudinal and transverse phonon branches at low temperature indicate thermal conductivity should vary as $T^{1.5}$ [33]. This is a consequence of coupling between transverse and longitudinal phonon modes introduced by surface scattering. In essence, scattering from the surface provides coupling between different phonon branches, and due to asymmetries in the coupling and differences in the dispersion functions of the different branches. Work done by Kühn et al. indicates that for thin $\text{Si}_x\text{N}_y$ membranes, $G \propto T^{1.5}$ [33]. They found that for wide thin membranes, compared with skinny membranes that the thermal conductivity should be significantly different as a result of different scattering mechanisms. They suggest that thinner membranes not restricted to 1D transport will exhibit different $G(T)$ due to edge scattering effects. Simulations of thermal transport in $\text{Si}_x\text{N}_y$ membranes of dimensions similar to ours, and have successfully recreated a number of the observed dependences observed in experiment [59].
3.1.5 Measuring G

To measure the thermal conductance, or thermal conductivity, we need to observe the power needed to sustain a temperature gradient. We do this by dissipating heat on an object and measuring the resulting temperature difference developed. After applying a constant heating power and establishing thermodynamic equilibrium with the heat bath, the temperature difference across the material indicates how effectively the sample conducts heat. The smaller the temperature difference, the better the conductor. By applying a series of increasing heater powers with the bath temperature held constant we can observe the relationship between heating power applied and temperature difference developed. By finding the slope of \( P(\Delta T = 0) \) we determine the thermal conductance of the material at the bath temperature. This can be accomplished by applying a series of small heater powers where the thermal conductance is assumed to be constant, as well as by introducing larger temperature deviations, and taking the derivative at \( \Delta T = 0 \).

The generated temperature differences, \( \Delta T_{\text{max}} \) between bath and membrane are targeted at ten percent the bath temperature for \( 50 \text{ mK} < T_{\text{set}} < 300 \text{ mK} \), and 30 mK for \( T_{\text{set}} > 300 \text{ mK} \). We have chosen these values because we would like to remain within a small \( \Delta T \) limit in measuring the thermal conductance of the bridges, and these \( \Delta T \)'s show a strongly linear \( P(\Delta T) \) relationship. By using only small temperature differences between the central membrane and bath temperature, the potential change in thermal conductance close to zero; resulting in a linear \( P(\Delta T) \). Observing nonlinearities in the relationship between power applied and temperature difference developed indicates a change in the thermal conductance, and should be avoided as this introduces systematic error into the measurement of G.

We can also apply a series of large heating powers \( P(\Delta T) \) for large \( \Delta T \). We can observe the thermal conductance at base temperature modeling the observing the relationship between heating power applied and temperature, and calculate the slope for \( \Delta T = 0 \). All of our measurements are performed in the low signal limit, and as is indicated in fig. 3.2 the
Figure 3.1: Typical results from a thermal conductance experiment. By increasing the heater power as step number increases we can observe an increase in Noise amplitude as a function of heater Power. By also setting $P=0$ to observe the Noise amplitude vs Temperature, we verify that there is little drift in the measurement and generate a quality calibration curve.

observed response is very linear. Fits to these data typically result in $R^2$ values larger than 0.970, indicating a strong correlation between the data and our linear fit.

### 3.2 Measuring $G$ of Microcalorimeters

We have measured the thermal conductance ($G$) of a number of devices with variations in geometry to observe the effect on transport through the medium. Measurements of $G$ for three geometries have indicated changes in transport phenomenon observed at low temperatures as the width of the structure changes from a central membrane supported by continuous membrane (ribbon), one connected by four legs 50 $\mu$m wide, and one supported by 6 $\mu$m wide legs. Several devices with 6 $\mu$m wide legs have been measured, and we discuss the implications of these devices here.
Figure 3.2: Plot of the heater power required to establish temperature difference between bath for many values of bath Temperature. Thermal Conductance (G) is given by the slope of the low ΔT limit of \( P(\Delta T) \).

3.2.1 6 um devices

Several structures were fabricated with nominally identical \( \text{Si}_x\text{N}_y \) legs supporting the thermal isolation platform. We have measured all of these devices to understand the variations of thermal conductance within nominally identical structures. These devices have 6 \( \mu \text{m} \) wide x 250 \( \mu \text{m} \) long x 200 nm. Beneath the supporting legs on all of these devices is a layer of SiO\(_2\) layer 100 nm thick. This layer is necessary during the fabrication process to stop a Deep Reactive Ion Etch (DRI etch) before this process destroys the membranes the etch releases from the Si wafer. These devices have the most restrictive geometry of all devices we have measured, and are most likely to exhibit effects caused by changes in phonon dimensionality, or be affected by the presence of any stray heating power. These devices have nominally identical shape to the device pictured in fig. 3.3. The device in fig. 3.3 was covered with a Cu film that was used to measure the heat capacity of Cu, all other
Figure 3.3: SEM micrograph of the calorimeter supported by four 4µm x 250µm long legs. All devices are fabricated from 500 nm thick Si$_x$N$_y$ membrane with a 100 nm SiO$_2$ under layer. This SiO$_2$ layer provides an etch stop for a Deep Reactive Ion Etch (DRIetch) that frees the suspended membrane from the Si wafer.

We see from fig. 3.4 that all devices agree at higher temperatures with nearly equal thermal conductance values, but at lower temperatures, there is deviation in the thermal conductance between devices. At the lowest temperatures, the various devices vary at low temperatures by nearly 20 percent. This can be attributed to changes in the surface condition of the different membranes or roughness of the lateral edges. As has been observed by a number of measurements, the thermal conductance of a film at low temperature can be strongly influenced by addition of surface scattering defects [22][55][51][61].

We observe thermal conductance values for the device with Cu covering the entire central membrane comparable to those observed without Cu film in fig. 3.4. The small change in G(T) tells us that our assumption that the temperature gradient is developed only across the legs is valid because the presence of Cu (with a much higher thermal conductivity than Si$_x$N$_y$) marginally alters the observed G. If there were a significant temperature gradient developed across the central membrane, covering it with a high $\kappa$ material would substantially
Figure 3.4: Measured thermal conductance (G) for all devices with 6 µm wide legs, highlighting the measured G(T) for the device with Cu film covering the central membrane.

3.2.2 Comparing G(T) for different Geometries

If we compare the thermal conductances of devices with different supporting leg geometries, we expect to see that devices with more substantial links to bath temperature have a larger thermal conductance. The three geometries we compare have 4 legs that are 6 µm, 50 µm, and 2 legs that are the width of the entire structure (300 µm). All devices have nominally identical Si$_x$N$_y$ film thicknesses and are fabricated from the same wafer with identical processes.

Calculating the thermal conductivities for these devices is a way to compare whether the scattering between different geometries is comparable between these devices. While thermal conductivity is the same for identical composition under bulk conditions, differences in thermal conductivity for identical compositions is a strong indication that the scatter-
Figure 3.5: Measured thermal conductance (G) for all devices with $6 \mu m \times 250 \mu m \times 200$ nm Si$_x$N$_y$ leg geometry and SiO$_2$ layer under.

...ing/transport mechanism is altered by geometry. Such a large difference between cross sectional areas for thermal transport between the 6um and 50um leg widths, we may begin to see effects of reduced dimensional transport as a result of the reduced width in the 6um geometry.

3.2.3 Comparing Thermal Conductance’s of Several Structures

We measured the thermal conductance of several membranes with varying geometry connecting the central membrane to the heat bath. To understand the nature of the thermal transport as it connects the central membrane to the thermal bath. As expected G increases with increased cross sectional area connecting the central membrane with bath. The higher conductance samples with wider leg dimensions have values that strongly overlay between samples, and measured the thermal conductance of the same devices several weeks later after cycling the temperature and exposing to atmospheric conditions as well as within the same cool down, to observe the same measured thermal conductance. The measurements are highly repeatable within the same sample, as well as between identical geometries.

We have observed the effect stray heating has on calibration curves, and causes higher
Figure 3.6: SEM micrograph of three geometries measured in this thesis. a) central membrane (300 $\mu$m x 300 $\mu$m) supported by 4 6 $\mu$m x 250 $\mu$m x 500nm Si$_x$N$_y$ legs. b) 100 $\mu$m x 300 $\mu$m central membrane supported by 4 50 $\mu$m x 250 $\mu$m legs. c) 100 $\mu$m x 300 $\mu$m “central membrane” supported by 2 300 $\mu$m x 250 $\mu$m legs forming a continuous ribbon above the etch pit.

Figure 3.7: The observed T dependence of $G$ for three different geometries. The wider leg geometries seem to have $G \propto T^{1.5}$ dependence throughout the temperature range, while the skinnier legs have a more complicated T dependence. This seems nearly linear at low temperatures and quadratic at higher temperatures.
than expected thermal conductance measurements at the lowest temperatures for devices with thicker legs. If we measure the thermal conductance of the devices with weak thermal link to bath, it is realistic that these devices are most sensitive to systematic errors due to stray heating power. Stray heating effects manifest in the data as a low temperature deviation from linearity in the calibration. When the stray heating power causes the temperature of the membrane to be higher than the bath temperature $T_{\text{membrane}} > T_{\text{bath}}$. This temperature difference should be related to the stray heating power, and the thermal conductance of the device. The existence of such a temperature difference at low temperature causes an inaccurate calibration curve. This inaccuracy in calibration curve causes the calculated temperature difference developed during the measurement of $G$ to be slightly lower than the actual temperature difference as heating power is applied. This will cause measurements of $G$ to be elevated above the actual thermal conductance value at the lowest temperatures.

As the bath temperature is increased the thermal conductance between the central membrane and the heat bath will increase, so for constant stray heating amplitude, the temperature difference between the thermometer and the heat bath will decrease. This means that the measurements at higher bath temperature will be less susceptible to stray heating effects which will give us a clear picture of the thermal transport properties in a moderate temperature range. This effect may also explain the change in low temperature $K(T)$ properties for the skinny legs, while the wider legs do not show this effect.

It is clear from the data in fig. 3.7 that the 6 $\mu$m leg devices have a different dependence with temperature throughout the measured temperature range as $K(T)$ for the thicker samples goes as $T^{1.5}$ for $T_{\text{bath}} < 1K$, while the skinnier legs indicate a $\sim T^2$ dependence. This is an interesting result, as it seems that the physical transport mechanism is changing as the lateral dimension of the thermal connection is changed. This holds true for measurements performed using high heating powers aimed at measuring the exponent of $K(T)$. For measurements performed on wider lateral dimension show a $T^{1.5}$.

Comparing our measurements with those performed by other groups is a useful, but
potentially difficult thing to do because comparing thermal conductivities is not as straightforward as it is at room temperature. This is a result of geometric differences affecting the transport at a fundamental level. Nonetheless it is an informative comparison. Measurements performed by Holmes et al. were performed on $1 \mu m$ thick $\text{Si}_x\text{N}_y$ in a radial geometry, and observed suppression in thermal conductance as Ag surface asperities were introduced, and measurements of 600 nm thick $\text{Si}_x\text{N}_y$ structures with perforated membrane devices very similar to our devices were performed by Goldie et al. \cite{50}[22]. Fig. 3.7 indicates that the observed thermal conductivity between these three groups with different thicknesses and geometries result in different values.

![Figure 3.8: Comparing the reported thermal conductivities for similar Si$_x$N$_y$ membranes. Work done by Holmes et al.[22] and Goldie et al.[50].](image)

The predicted temperature dependence of $G$ for a bulk amorphous material as has been previously studied and reported by many groups is $G(T) \approx T^2$ \cite{20}[47][62][60]. While we observe deviations from this trend, taking descriptions of transport within a bulk material, and comparing calculated thermal conductivities with thin film devices, is somewhat dubi-
ous and misleading. At some point we expect to observe deviation from $T^2$ dependence.

Critical to the analysis of these data is coming up with an estimation of what temperature transport begins to deviate from bulk measurements. This is an indication that the physical mechanism restricting thermal conductance is beginning to change. Some effort has been made to estimate the “cross-over” temperatures for several specific geometries. The temperature refers to the temperature at which thermal transport begins to changes as a result of geometrical constraints. The calculated 2D-1D “cross-over” temperature has been observed and predicted around 200 mK for highly structured geometries [3]. If we try to calculate the cross over temperatures for our devices, we see, that the 2D-1D “cross-over” temperature is very low for devices with 50 µm wide legs, and continuous ribbon geometries [23].

\[ K = k(T^n - T^m_b) \]  \hfill (3.1)

\[ G = nkT^{(n-1)} \]  \hfill (3.2)

### 3.2.4 Dimensionality Phonon Gas

As the dimensions of the transport channel begin influencing the physics within the medium, we expect to see a change in the thermal transport. When the phonon gas conducting heat through the medium changes from a bulk 3D to a 2D gas, $G(T)$ should change. For a 3D phonon gas the description of bulk transport holds, in this case an insulating material will have $G \propto T^3$. For a phonon gas restricted to 2D, $G \propto T^2$ [3].

Since the thermal conductivity is related to the specific heat through eq. 3.1, predicting changes in the specific heat of the phonon gas amounts to a corresponding change in thermal conductance. Work done by Pekola et al. have worked to understand the transport properties of a 2D phonon gas [3]. This nomenclature is adopted to describe dimensionally restricted transport, and refers to a phonon gas in which one or more dimension has been
restricted/eliminated. They investigated 200 nm thick insulating film, and predict a 3D-2D transition at \( T \approx 500 \text{ mK} \). The criteria used to determine this temperature is given by eq. 3.3.

\[
x_0 = \frac{\pi \hbar}{k_B T} \frac{v_s}{d}
\]  

(3.3)

For a thin film, the assumption of continuous density of states in the constricting geometry is no longer an appropriate approximation, and the corrected formulation for specific heat of the phonon gas is given by eqs. 3.4 and 3.3.

\[
c = \frac{3k_b S}{2\pi} \left( \frac{k_b T}{v_s \hbar} \right)^2 \left[ \int_0^\infty \frac{x^3 e^x}{(e^x - 1)^2} dx + \sum_{n=1}^\infty \int_{nx_0}^\infty \frac{x^3 e^x}{(e^x - 1)^2} dx \right]
\]  

(3.4)

For contained geometries the number of allowable phonon modes becomes constrained, and this is why the introduction of the summation across allowed modes. As the temperature is decreased, the summed term converges to zero. This indicates that a 2D phonon gas has a thermal conductance that goes as \( G \propto T^2 \), while 3D phonon gases have \( G \propto T^2 \).

These predictions indicated sharp changes in the exponent of thermal transport as the phonon population condenses in one dimension. These sharp changes can be altered to more broad transitions through surface scattering. The effect of surface scattering on the transition from 3D-2D transport is to prolong the population of certain 3D transport modes, although they are limited in population. The more diffusive the surface scattering events, the more broadened the transition from \( G \propto T^3 \) to \( T^2 \).

### 3.2.5 Comparing Observed G with Quantum of Thermal Conductance (\( g_0 \))

Interesting experiments have been performed to measure quantum effects in thermal conductance within insulating [53] and metal materials [12]. These observations can only occur by highly restricting transport phenomenon at low temperatures. Restricting transport amounts to engineering the thermal link from an elevated temperature to bath temperature, by reducing the cross-sectional area. This engineering specifically targets reducing the di-
dimensionality of the transport mechanism to a 1-D regime. Once the transport is restricted to a 1-D regime, measurements of $g_0$ require prohibiting specific phonon wave numbers. An interesting question to answer at low temperatures with long, skinny transport geometries, is it possible to sustain 1-D transport properties, while having a significant number of phonon modes available. The theoretical work described in [48] describe different phonon waveguides that allow tuning of the phonon permissible frequencies. It seems that 1-D transport can be sustained in different leg geometries without specifically shaping the leg for such “extreme” measurements.

The quantum of thermal conductance is defined by eq. 3.5 and represents the quantum limitation on thermal conductance that is predicted from limits on boson transport. This concept was illustrated by Drummond and Caves in 1994 by calculating the quantum limitations on information transfer by bosons [11].

$$g_0 = \frac{\pi^2 k_B^2 T}{3h} \approx T(9.456 \times 10^{-13} W K^{-2}) \quad (3.5)$$

If we compare measured thermal conductance of our devices with the quantum of thermal conductance, as in fig. 3.9, we see that the observed thermal conductance of our system is much larger than measurements of quantum limited thermal conductance performed by Schwab et al. [53][51]. The extremely small thermal conductance between the suspended membrane and bath is within the realm of exhibiting mode limited thermal transport. Measured thermal conductance of the most restricted geometry correspond to ~100’s of $g_0$. In light of this comparison, it seems likely that our observations are constrained in dimensionality of transport, but is a stretch to say that we are mode limited.

### 3.2.6 Effect of HF etch removing SiO$_2$ Layer

If we observe the change in thermal conductance as a result of removing the SiO$_2$ we can begin to understand how the thermal oxide layer can alter thermal conductance in these
Figure 3.9: Plotting the measured thermal conductance of a calorimeter in terms of $g_0$ (quantum of thermal conductance). The extremely small thermal conductance to bath of these devices is illustrated by the relatively small number (100's) of $g_0$

devices at low temperatures. The thin SiO$_2$ layer may have a number of potential effects in altering the thermal conductance of our sample. The oxide layer may serve to alter the specular scattering coefficient of the underside of the membrane or it may behave as a separate transport channel. We have observed the change in thermal conductance at low temperature having removed the oxide layer from two geometries. We have compared the resulting change in thermal conductance as a result of removing the oxide layer.

We are able to selectively remove the oxide layer by immersing the entire device in an aqueous HF acid etch. Hydrofluoric acid has a very high rate of etching SiO$_2$, while remaining inert to the metals used in fabricating our devices. By dipping these devices into a solution of 10:1 Water to HF for 9 min, we can ensure that the entire oxide layer has been removed. The other aspects of these devices should remain unaltered by the HF etching process.

We observe a decrease in G having removed the oxide layer, in both geometries. This
difference is not uniform through the measured temperature range, and the difference is most at low temperatures for both geometries. As temperature is increased, the differences between thermal transport through the medium approaches zero. This suggests that in the 3D limit the existence of a thin SiO$_2$ layer plays a minimal part in scattering phonons, as we would expect. Also at higher temperatures, the oxide layer is not behaving as a substantial transport channel.

The fact that the oxide layer plays a more substantial part in dictating transport through the most restrictive geometry indicates that limited geometries are more sensitive to the surface condition of the medium. This change in transport can significantly alter the transport efficiency within these thin membranes which can have dramatic influence on the performance of low temperature detectors.
Chapter 4

Heat Capacity Measurements

4.1 Introduction

Recent advances in micro-fabrication techniques have enabled development of calorimeters suitable for performing accurate measurements of very small samples. Early measurements of heat capacity at low temperature were limited to relatively large samples because thermometers, and isolation techniques introduced substantial errors to measuring small heat capacities. It is a challenge to sufficiently isolate samples from bath, and the large addenda heat capacities introduced significant uncertainty limited resolution of bulk calorimeters. Classically the measurement of the heat capacity of an object required the use of macroscopic thermometers, with associated uncertainties in heat capacity, to a sample whose thermal properties were under investigation. This meant that determining the heat capacity of small samples required extremely precise measurements that were prohibitively difficult to perform. In order to determine the heat capacity of a small sample, one was forced to measure the heat capacity of a much larger sample, calculate the specific heat, and scale down to find the heat capacity of a smaller sample. Measuring the heat capacity of a larger sample and scaling down to represent heat capacities of much thinner films is reasonable as long as the material still behaves as a bulk material, but does not re-
Figure 4.1: Diagram of bare calorimeter with associate thermal conductances $G_{legs}$ and $C_{total}$ determine the thermal time constant of the device

flect the presence of any other small heat capacities that might be introduced at interfaces, and should not scale with the dimension of the material.

By reducing the size of the calorimeter, and limiting the thermal link to bath we are able to make precision calorimeters sensitive enough to measure thermal properties of materials and structures into the micro scale. This allows measurements in to the interactions within micro machined structures, interfaces, and highly engineered films(patterned, or multi-layered).

4.2  Calorimetric Measurement Method Summary

4.2.1  Semi-adiabatic Calorimetry

Early measurements of heat capacity were performed by attempting to completely isolate the sample from bath, and observing the temperature difference developed from the application of a heat pulse. This is suitable for objects whose masses were relatively large (g-kg scale), where the presence of small heat leak was insignificant compared to the total heat applied. However, trying to measure very small heat capacities requires that much
smaller heat pulses should be applied in order to prevent very large temperature differences being developed. Where the heat capacities of the films are small enough that even very small variations in the incident heat, and the potential existence of parasitic heating can cause significant systematic errors. The creation of weak thermal links to bath can be realized in thin films by geometrically limiting the thermal link. Even these thermal conductances can be large enough to prohibit adiabatic measurement technique of small samples at low temperatures.

\[ C = \frac{\Delta Q}{\Delta T} \]  

(4.1)

Semi-adiabatic calorimetric measurements are performed by establishing thermal equilibrium between the sample to be measured and heat bath. The application of a known heat pulse causes a temperature rise in the sample. This relationship between the heat dissipated and temperature difference dictates the heat capacity as shown in eq. 4.1. This technique has the advantage that the heat capacity is directly probed, and with complete thermal isolation does can very accurately determine the heat capacity of the sample and addenda. Due to the extremely long time constant associated extremely weak link to thermal bath, prolonged periods of time must pass in order to repeat a measurement. This relationship between the heat dissipated on the sample and the rise in temperature begins to degrade with stronger thermal coupling to bath and requires an alternative measurement technique. For small samples it is difficult or impossible to reduce the heat leak from sample to bath to a level that is much smaller than the heat used to raise the temperature.

When the thermal isolation of the sample to be measured is incomplete, we are able to determine the heat capacity of the sample by making a complementary measurement of the thermal conductance between sample and environment. This thermal conductance and heat capacity introduces a time constant between the sample and environment. Making this time constant as long as possible we are able to determine the heat capacity of the sample by using the simple relationship between the heat applied to the sample and the resulting rise in temperature. Adiabatic calorimetry is useful for certain applications, but is unrealistic to
apply to very small heat capacity measurements.

4.2.2 AC Calorimetry

If thermal isolation of the sample from bath is incomplete, we are able to observe the heat capacity of our small sample by first quantifying the thermal conductance between sample and bath. By measuring the heat transport we are able to account for heat lost from the sample to bath during the measurement period. Observing the temperature of the sample as a function of heating power indicates the heat capacity of the sample. We can observe this behavior by applying an AC heating power, DC heating power, or Sweeping the heater power in time. Any of these patterns have expected temperature responses that depend on the heat capacity and thermal conductance of the calorimeter.

As the need to measure smaller and smaller samples at colder temperatures became necessary alternative techniques determine the heat capacity of a sample were established. Sullivan and Seidel worked to develop a complementary measurement technique to measure the heat capacity of small samples at cold temperature by measuring the temperature oscillations developed as a result of an AC heating signal [54]. By driving a heater signal at frequency $f_{\text{heater}} = 2\pi w$ the power dissipated by the heater into the sample is modulating at $2f_{\text{applied}}$. Therefore we expect to observe temperature oscillations at twice the frequency of the heating current.

AC calorimetric measurements probe the frequency response of the thermal system to determine the thermal time constant of the sample to be measured. This works by developing a quasi-equilibrium between calorimeter and bath that is constantly modulating. By understanding the frequency response in amplitude and phase, we are able to identify the time constant of the system. This technique requires measurement of the thermal conductance of the sample, as well as the thermal time constant ($\tau$). These two measurements constitute measurement of the heat capacity of the system. This technique is susceptible to complications arising from $\tau_2$ effects, strongly non-linear response, potentially long settling
times, and has the advantage of continuous readout.

By accurately measuring the amplitude of the temperature oscillations as a function of frequency and fitting to an equation of the form eq. 4.2. We are able to determine the thermal time constant of the system. This information along with a determination of the thermal conductance between the sample and bath, and we are able to use the relation \( C = G \tau \) to identify the heat capacity of the sample [34].

\[
\delta T \propto \left[ 1 + \left( \frac{f}{f_c} \right)^2 \right]^{-1/2}
\]  
\[ \text{eq. 4.2} \]

\[
f_c = \left( \frac{2\pi C}{G} \right)^{-1}
\]  
\[ \text{eq. 4.3} \]

While \( \tau_2 \) effects are of a significant concern, they are not unique to AC calorimetric measurements, these effects can distort the measured time constant, and change the calculated heat capacity for the sample. \( \tau_2 \) effects can distort the actual time constant in a couple of ways, first if \( \tau_2 >> \tau_1 \), than the measured time constant of the sample will not be indicative of the sample to be measured, and hence will not physically represent the heat capacity of the sample to be measured. it is also possible, that if \( \tau_2 \) is on the order of \( \tau_1 \), the measured time constant \( \tau_{meas} \) will be neither \( \tau_1 \), nor \( \tau_2 \), and the exponential decay of \( T(t) \) will be a convolved function that is best described through the two time constants.

### 4.2.3 Relaxation Calorimetry

Relaxation calorimetry is another non-adiabatic method requiring separate measurement of the thermal conductance to bath. Relaxation calorimetry provides a measurement technique that allows a little more latitude for detecting the presence of \( \tau_2 \) effects than AC calorimetric techniques because you are able to directly observe the temperature of the membrane as a function of time, where the lock in technique, only monitors the amplitude of the temperature oscillations as a function of frequency. In this way a person is able to better distinguish whether the temperature decay is exhibiting some of the hallmarks of \( \tau_2 \)
4.3 Measurements of the Time Constant

Many of the previously discussed calorimetric techniques require the prior determination of the thermal conductance to bath as well as a measurement of the thermal time constant in order to constitute a measurement of the heat capacity of the sample. For these
measurements, potential complications arise with increased complexity in measurement setup, and in the associated error of the calorimetric measurement. Using Johnson Noise Thermometry to determine the time constant of the micrcalorimeter requires an accurate determination of noise amplitudes as a function of time immediately after an application of heat. By applying a constant power long enough to establish thermal equilibrium between the calorimeter, sample, and bath establishes a temperature difference across the supporting legs. We have measured the time constant of one sample using both AC and relaxation calorimetry.
4.3.1 AC

Steady state AC calorimetry measures the time constant $dt$ of the system by probing the temperature oscillations resulting from an AC current at frequency $f$ passing through the heater for a number of different frequencies. The Joule power dissipated by this current is proportionate to the square of the current, and the power dissipated by the heater modulates at $2f_{\text{appl}}$. The temperature response of the heater and calorimeter will vary as the heating power dissipated by the resistor, thus the temperature of the membrane should oscillate at twice the applied frequency. The amplitude of the temperature oscillation is observed as an change in the amplitude of noise generated by the thermometer at a frequency twice that of the heating signal. We determine the amplitude of temperature oscillation by measuring the amplitude of the $2f_{\text{appl}}$ signal within the PSD, or by using a lock-in technique. Thus, applying a heater frequency at $f$ induces a temperature oscillation at $2f_{\text{appl}}$. As we sweep through different heater frequencies we should observe the following three regions.

First for frequencies much lower than the critical frequency, a maximum of temperature oscillation is realized, and the amplitude of temperature fluctuation is independent of the frequency. This indicates that the central membrane is in equilibrium as the heater current oscillates between $0 < I < I_{\text{max}}$. The $2f_{\text{appl}}$ response in this region should be at a maximum, indicating the largest fluctuation in temperature as heater power is varied. The second region, where the heater current oscillates at a frequency near the critical frequency, the changes in the power dissipated by the heater are fast enough that the central membrane is unable to reach the maximum temperature it achieved at lower frequencies, and is unable to relax to bath temperature. In this region the amplitude of temperature fluctuations begins to decrease as frequency increases. In the third region the frequency oscillations are faster than the central membrane is able to respond to, and hence the amplitude of the temperature oscillations is zero.

The amplitude of the $2f_{\text{appl}}$ response can be monitored by observing the amplitude of the $2f_{\text{appl}}$ signal in a Fourier transform of the noise data. The amplitude can equivalently
be measured using a lock-in technique. By then fitting the amplitude of the temperature oscillation as a function of heater frequency to the equation where $T_{DC}$ is the amplitude of the temperature fluctuation in the low frequency limit, or the temperature difference developed by applying a DC heating current whose magnitude is equal to the maximum current of the AC signal. By identifying the critical frequency of the calorimeter, and using the relation we know the time constant of the calorimeter.

$$\frac{\Delta T}{T_{DC}} = \left( \sqrt{1 + \left( \frac{f_{app}}{2\pi f_C} \right)^2} \right)^{-1}$$  \hspace{1cm} (4.1)

$$\tau_{cal} = \frac{2\pi}{f_C}$$  \hspace{1cm} (4.2)

Early attempts to implement this technique on our devices suffered from long measurement times. This was a result of needing to use long filter time constants on the lock-in amplifier. To safely observe the $2f_{app}$ amplitude using the Lock-in amplifier would require a 30 second filter time constant, as a result a single data point required nearly 40 min to record. This proved too time consuming, because $\tau_1$ for these devices is greater than 100 we were required to use heating frequencies below 10 Hz. A number of reasons prompted us to seek an alternative technique to observe $\tau_1$.

### 4.3.2 Relaxation

We measure the time constant by observing the temperature response of the central membrane after allowing the system to come to thermal equilibrium with in the presence of a heating power applied bath. We have positioned the heaters symmetrically about the central membrane to help establish an isothermal central platform, and ensure that the developed temperature difference is confined to the supporting legs.

With the device at bath temperature, we apply a constant heating power to the central membrane. The noise amplitude generated by the thermometer will increase proportional
Figure 4.4: Plot of $V^2(t)$ for 5 temperature set points. By fitting the exponential decay of voltage variance as a function of time to determine the time constant, we are able to determine the thermal time constant of the sample.

to the temperature rise on the membrane. This rise in temperature will decay to some temperature $T_{\text{mem}} > T_b$. If once the central membrane and bath reach thermal equilibrium, we suddenly remove the current flowing through the heaters, the central membrane is no longer in thermal equilibrium, and the temperature will exponentially decay from the elevated $T_{\text{mem}}$ to $T_b$. By observing the temperature as a function of time, we are able to fit to an exponential decay, and determine the decay time constant. Because the variance of the voltage observed is proportional to the temperature of the membrane, we can determine $\tau_{\text{measured}}$ by observing the voltage decay time constant, and no other conversions are necessary.

In observing the thermal time constant of the system in order to determine the heat capacity of the sample under measurement, we need to be wary of the potential existence of $\tau_2$ effects. This effect is best described by observing that the various heat capacities on the sample are thermally coupled to the temperature of the membrane through electron-phonon interactions. As we observe the noise amplitude of the thermometer, we are probing the
temperature of the electrons within the thermometer. At low temperatures, the coupling between electrons in the thermometer and phonons in the membrane is given by

We see that at sufficiently low temperatures, the effective thermal conductance (coupling) between the thermometer and membrane, as well as the other metallic films on the membrane diminishes. The internal time constant of the system will be dictated by the G’s and C’s of the different bodies on the calorimeter itself. These G’s and C’s establish the internal time constant, and how this compares with the "external” time constant can influence the validity of our measurement. This can be visualized by looking at the heat flow within the sample as the heating power is switched off.

We assume that all bodies on the membrane are heated for a sufficient amount of time to equilibrate the membrane, thermometer, heater, and film with bath temperature. After the heating power is brought to zero, the power flowing down the suspending Si$_x$N$_y$ legs will begin to decrease exponentially. In order to accurately measure the time constant of the entire sample (membrane, thermometer, heater, and film) all of these bodies must not develop substantial temperature differences between them.

The internal time constant, $\tau_2$, must be considerably shorter than the external time constant $\tau_1$ in order to preserve an isothermal relationship between the membrane and metallic films. If there were a substantial temperature difference developed between the metal films and the membrane, we might observe an exponential decay involving two terms. Hence the $\tau_2$ nomenclature. This effect was noticed and discussed in Bachmann’s paper discussing relaxation calorimetry[4]. There condition for 1 percent accuracy in a relaxation style calorimetric measurement is that

$$\tau_2 < 5\tau_1$$  (4.3)
Figure 4.5: Cartoon schematic of the calorimeter indicating the associated thermal conductances (G’s) and heat capacities of the system. The thermal conductance between the heater, thermometer, and metallic film on the membrane is determined by the electron-phonon (EP) coupling. Because of the effective thermal conductance between the various heat capacities on the calorimeter there exists an internal thermal time constant for the system ($\tau_2$).

Figure 4.6: Normalized Temperature vs Time curve for a 6µm leg width device. This data is fit to three equations, to analyze potential $\tau_2$ effects. We see only marginal gains in $R^2$ values for including additional free parameters to the fitting routine. This is an indication that we are not suffering $\tau_2$ effects in our measurements. This device in particular should be most susceptible to these types of effects because of the much lower external thermal time constant.)
4.4 Measurement Details

We have developed a technique to perform relaxation calorimetry using Johnson Noise Thermometry. By first measuring the thermal conductance of the device, then the time constant of the exponential temperature decay we are able to determine the heat capacity of the system. We observe the temperature decay by synchronizing a square wave heating signal with a series of voltage measurements in time. By measuring the observed voltage output as a function of time for several cycles of heating/cooling, we are able to determine the temperature of the thermometer as a function of time.

The voltage generated by the noise thermometer on the membrane is amplified by a two stage SQUID amplification chain, passed through a band pass filter amplified at room temperature, and fed into a Data AcQuisition (DAQ) card for processing within LABVIEW. Each series of voltage measurements is written to disc, and the current mean voltage as a function of time is recorded. During this process, each of the voltage measurements in a given decay $V_j(t_i)$ are added together to facilitate an accurate measurement of the mean voltage at a given time in the decay. After a number of decay processes are observed, each of the recorded voltage series are recalled, and used to calculate the mean variance of the voltage as a function of time. By sampling the voltage at a sufficiently fast rate, a wealth of voltage variance estimates can be observed within a decay process. We sample the voltage output from the measurement change at a rate of 250 kSa/s. The sampling rate of our voltage measurement limits the range of decay processes that can be observed, but is more than sufficient for relatively fast determination of $100ms \lesssim \tau$.

Higher data acquisition rates translate to, not only the ability to observe shorter temperature decay processes, but also allow the same uncertainty in time constant measurement for fewer decay observations if you employ a windowed averaging technique. The signal generated by the temperature decay is inherently noisy. The signal we are attempting to measure is noise, with a subtly decreasing amplitude that is caused by the removal of heating current passing through the calorimeter heater. Typical applications of JNT involve time
averaged noise measurements of the voltage generated by the resistor held at thermal equilibrium. In this instance, we are looking for non-equilibrium measurements of the noise amplitude. Therefore, one must be careful when using a "time based" averaging technique.

This relatively high density of voltage measurements allows further improvement upon the noise amplitude estimate at a given time. If we then also apply a widowed averaging scheme where

$$\bar{\sigma}^2(t_i) = \frac{1}{m} \sum_{j=0}^{m} \sigma^2_{i+j}$$

(4.1)

This processing further decreases the noise on the calculation of the temperature as a function of time.

If we then calculate the mean voltage variance before the decay process and more than 7 time constants beyond the decay process. By using these observations to constrain an exponential fit to the noise amplitude vs time and the mean voltage variance vs time we are able to determine the thermal time constant of the system at a given temperature.

In order to accurately determine $T(t)$ we measure the voltage output of our system for
several hundred decay processes. Each decay process has a series of voltage measurement synchronized in time with the onset of the decay process. Generating an ensemble of decay processes allows an accurate determination of the mean voltage as a function of time. With the mean voltage as a function of time $\bar{V}(t)$ determined it is possible to calculate the variance of the voltage for each of the decays observed. Calculating the mean voltage Variance as a function of time gives an estimate of the temperature of the membrane as a function of time, which is given by the eq. 4.4. We can further reduce the error on $T(t)$ by using a “boxcar” or “windowed” averaging scheme. With careful selection of $w$, and its relationship to the time between measurements this kind of averaging acts as a low pass filter on the estimation of temperature. While there are gains in reducing overall noise by employing such an averaging technique, it is possible to alter the observed time constant by including too many neighbors (too large $w$). The critical frequency of such a low pass filter is given by the sampling rate and windowing width shown in eq. 4.2. As long as the averaging time-scale is much shorter than the decay process, we are not altering the observed time constant by including this type of averaging. The windowed averaging routine is described by equation 4.5.

$$f_c = \frac{2\pi}{Sw}$$  \hspace{1cm} (4.2)

$$f_c >> \frac{2\pi}{\tau}$$  \hspace{1cm} (4.3)

$$\sigma_V(t_j) = \frac{1}{N-1} \sum_{i=0}^{N} (\bar{V}(t_j) - V_i(t_j))^2$$  \hspace{1cm} (4.4)

$$\bar{\sigma}_V(t_j) = \frac{1}{2w} \sum_{k=(j-w)}^{j+w} \sigma_V(t_k)$$  \hspace{1cm} (4.5)

Calculating the variance of the voltage measurement at a given time in the decay process
tells us the amplitude of the noise generated by the thermometer on the central membrane. Observing several hundred decay processes reduces the uncertainty in the noise amplitude to improve the quality of the data. The error associated with noise amplitude as the number of observations increases should go as $\sqrt{N}$. As more measurements of the noise amplitude vs time are acquired, the relative uncertainty in $T(t)$ decreases. Since the voltage variance is proportional to temperature for small temperature deviations throughout the temperature range, exponential decay in the mean variance of the output voltage is proportional to exponential decay of the temperature. If we calculate the time constant of the exponential decay in mean variance signal we are also calculating the time constant of the temperature decay, and is given by equation 4.6.

$$T(t) = (T_1 - T_0) e^{-t\tau^{-1}} \quad (4.6)$$

In order to measure $\tau$ of the calorimeter accurately, we need to approach the small heating limit. By doing this, we minimize potential differences in $G$ and $C$ of the sample and structure. To ensure sufficient signal to noise ratio needed to accurately observe $\tau$ we would like to maximize $\delta T$, without exceeding the small signal limit. To better understand the relationship between $\delta T$ and $\tau$, a series of relaxation measurements were performed at several base temperatures to observe the relationship. For $\delta T$ approaching zero, we expect to see significant uncertainty in $\tau_{measured}$ due to limitations in signal to noise ratio. We can improve $\tau$ estimates for low $\delta T$, but this becomes prohibitively long in terms of the number of decay processes required to minimize uncertainty. As $\delta T$ is increased we increase the signal to noise ratio, and by using values of $\delta T$ that correspond with a linear $P(dT)$, we can improve experimental uncertainties associated with $\tau$. Figure 4.8 illustrates the difficulties associated with minimizing the uncertainty in $\tau$ associated with signal to noise ratio while performing relaxation measurements.
Figure 4.8: Calculated $\tau$ vs $\delta T$ used to measure. Significant uncertainties prohibit accurate measurements of $\tau$ for $\delta T$ approaching 0. Targeted heater amplitudes for measuring $\tau$ in this manner are 10 percent base temperature, for $T_{\text{base}} < 300$ mK, 30 mK for $300$ mK $< T_{\text{base}} < 700$ mK, and 50 mK above 70 mK.

### 4.5 Heat Capacity of Calorimeter

Several devices were fabricated on suspended Si$_x$N$_y$ membranes with only heaters, thermometers, and superconducting leads patterned onto the central membrane. These "bare" calorimeters have been measured, and used to determine the heat capacity of a sample subsequently deposited onto the central membrane. The relative error and magnitude of the heat capacity of the central membrane $C_{\text{cal}}$ identifies the lowest heat capacity that we can distinguish from the calorimeter itself. We have measured the heat capacity of these devices, and compared the observed heat capacity of the central membrane to see how well they compare. We see that all unaltered heat capacity measurements for these devices agree quite well, with little deviation as seen in fig. 4.9. The data marked as SiO$_2$ removed was measured after etching the SiO$_2$ layer, and indicates a significant change to the heat capacity.

Measurements indicate a $T^3$ dependence for $T > 1 K$ and a linear relationship at tem-
Figure 4.9: Measured C(T) for all “bare” calorimeters. This represents the heat capacity of the membrane, thermometers, and leads on the film. The heat capacity is strongly altered after removing SiO$_3$ under layer.

temperatures below 1 K. We expect that the potential dimensionality shift in phonon population at low temperature alters the phonon heat capacities, but the linear dependence on heat capacity for amorphous SiO$_2$ and Si$_x$N$_y$ is also expected from TLS systems in the amorphous membranes. Many observations of linear heat capacity in bulk amorphous materials at low temperatures have been reported, and this is expected to dominate at temperatures where the phonon gas is not restricted by geometry. For temperatures at which the phonon dimensionality are restricted, the phonon heat capacity will change, but the heat capacity of TLS’s in the different materials should remain unaffected. [27][45][8].

As both SiO$_2$ and Si$_x$N$_y$ are amorphous materials, both materials should exhibit linear heat capacities in temperature as TLS begin to dominate. An interesting question is how the population of TLS’s within the different materials scale compared to each other. If we compare the expected contribution to heat capacity of the SiO$_2$ layer with measured heat capacity before and after removing this material, we see that the change in observed heat capacity is not explained by a simple subtraction, as shown in fig. 4.10. The significant
shift in heat capacity after removing SiO₂ film indicates that calorimeters such as these are sensitive to potential shifts in fabrication technique, and the heat capacity of such a thin (100 nm) membrane of SiO₂ is significant in comparison to the heat capacity of SiₓNᵧ at these temperatures. While the anticipated heat capacity of the oxide layer is too small to account for the shift observed, removing the oxide layer also alters the interface with SiₓNᵧ membrane. It is possible that unaccounted for heat capacity exists at the interface between the two insulating materials, and by removing the oxide layer, we have also altered the nature of the interracial heat capacity.

![Figure 4.10: Comparison of the measured heat capacity of the micrcalorimeter before and after SiO₂ removal with estimates for heater and thermometer, and SiO₂ heat capacities.](image)

Excess heat capacity in systems similar to ours have been observed and attributed to surface conditions in the processing of the calorimeters. Observations by Kenyon et al. at JPL have observed significant excess heat capacity in thin SiₓNᵧ membranes and have been show to be highly dependent on fabrication technique. They attributed the excess heat capacity to additional surface states on exposed faces and edges of SiₓNᵧ membranes that are process dependent [31] [27].
4.6 Heat Capacity of Cu films

We have observed the heat capacity of a number of calorimeters, two of which have a relative excess of Cu deposited on the central membrane. These two observations of the heat capacity have an anomalously high heat capacity. The difference between observed and expected heat capacity is nearly 100x, with only samples with substantial Cu film coverage exhibiting the much larger heat capacity. This indicates that the deposited Cu films are the source of the anomalous heat capacity.

Figure 4.11: SEM micrographs of Samples CU1 and Cu2. Geometry I is identical to that of Cu1, SiN1,2,3 while Cu2 is the only sample with geometry II. The suspended calorimetry platform with central membrane supported by 4 Si$_x$N$_y$ legs with lithographically patterned heaters, thermometers (Cu), and leads (Nb) A) Geometry I with 6µm x 250µm legs and 300 x 300 µm central membrane B) Geometry II with 50µm x 250µm legs and 150 x 300 µm central membrane

The thermal conductance measurements indicate good agreement among all of the devices with similar geometry, and a much higher G for the device with 50 µm leg width. The measurements of the time constant of the different devices indicates that the devices with excess Cu deposited have a much longer time constant than was expected. Comparing $\tau_{\text{measured}}$ for Cu1 and BN; having identical geometries, indicate a difference of nearly 100x. This translates to a measured heat capacity difference of the same value. Comparing
Figure 4.12: Measured heat capacity of 2 devices with excess Cu content deposited on the central membrane plotted against devices without excess Cu content, and the expected heat capacity of Cu film. There is nearly 100x difference in heat capacity of bare calorimeters ($\text{Si}_x\text{N}_y$) to the calorimeter of identical geometry with a patterned Cu film covering the central membrane. This 100x factor is also roughly the difference between expected and apparent $C_{Cu}$

the expected heat capacity of the different measured samples compared with the expected contribution due to the excess Cu only indicates the existence of an unpredicted heat capacity.

By comparing the measured specific heat (J mol$^{-1}$ K$^{-1}$) of Cu1 and Cu 2 we see that the two devices have heat capacities that scale with the volume of Cu deposited on the surface of the central membrane. The specific heat predicted from theory is given by $4.5^*$, with measured values of $a$ and $B$ of 0.695 mJ mol$^{-1}$ K$^{-2}$ and 0.0480 mJ mol$^{-1}$ K$^{-4}$.[30][38]. In this equation $a$ represents the electron contribution to the specific heat, with $B$ representing the lattice contribution. At sufficiently low T, the heat capacity of Cu should be dominated by the electron contribution. Our data indicate a linear dependence of C with T, as expected for a metal at low temperature.

The magnitude of this contribution is much larger than theory predicts. At these low temperatures we expect the electrons to dominate the heat capacity and should observe
Figure 4.13: a) Measured Thermal conductivity of all devices. Samples Cu1, Si$_x$N$_y$1,2,3 have nominally identical geometry, while sample Cu2 has much wider legs connecting the central membrane to bath. b) Measured $\tau$ as a function of temperature for all devices. Nearly 100x increase in $\tau$ of a geometrically identical sample is observed in CU1. This dramatic increase in heat capacity is due to the excess Cu deposited on the central membrane.

$a \propto T$ dependence, which is observed in both Cu1 and Cu2. The magnitude of the observed heat capacity is nearly 2 orders of magnitude larger than the value predicted for Cu. This anomalous heat capacity is very large, and clearly dominates the heat capacity of the calorimeter.

\[ c = \alpha T + BT^3 \]  

(4.1)

The heat capacity predicted from normal estimations described by equation 4.1, with measured values of $\alpha$ and B of 0.695 m$J$mol$^{-1}$ K$^{-2}$ and 0.0480 m$J$mol$^{-1}$ K$^{-4}$. In this equation $\alpha$ represents the electron contribution to the specific heat, with B representing the lattice contribution. At sufficiently low T, the heat capacity of Cu should be dominated by the electron contribution. Our data indicate a linear dependence of C with T, as expected for a metal at low temperature. The magnitude of this contribution is much larger than theory predicts. Our observations indicate an $\alpha = 22.7$ mJ mol$^{-1}$K$^{-2}$ whereas measurements performed by other groups observe the much lower 0.695 mJ mol$^{-1}$K$^{-2}$ [38] [35] [29] [56].

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Figure 4.14: Measured specific heat capacity of Cu1, and Cu2. These measurements indicate that the excess heat capacity observed in our samples scales with the Cu content deposited on the calorimeter.

Previous measurements of the heat capacity of doped Cu samples (0.1 at. percent Mn) has been extensively studied as a spin glass system [58] [39] [16][45]. Mn atoms with spins randomly included within the Cu matrix that exhibit RKKY interactions. This behavior is described as a Two Level System (TLS) very similar to the description used to explain variations in thermal properties within amorphous materials. These TLS have an energy distribution of within bounds \(-D < e < D\). The heat capacity predicted by a network of TLSs with a distribution of energies within \(-D < e < D\) is described by the relation and in the low temperature limit of \(k_B T << e\) leads to a heat capacity that is linear in \(T\) [39].

\[
C(T) = \int_0^\infty n_0 \left( \frac{E^2}{4k_B T^2} \right) \text{sech}^2\left(\frac{E}{2k_B T}\right) dE
\]

\[
C(T) = \left( \frac{\pi^2}{6} \right)n_0 k_B^2 T
\] (4.2) (4.3)

The description of disordered spins within a normal metal accurately reproduces heat capacity measurements for a number normal metal systems doped by a small amount of...
Figure 4.15: By scaling the heat capacity observed by Wenger and Keesom in $Cu_{1-x}Mn_x$ spin glasses and calculating the energy per magnetic defect, we estimate that $2E11$ spin impurities in our Cu film can describe the observed excess heat capacity.

Magnetic impurities. As the amount of dopant is increased beyond dilute quantities describing magnetic heat capacity as “glass-like” is no longer valid. More concentrated magnetic impurities would exhibit a more traditional heat capacity of a paramagnet as the impurities couple more directly. The “glass-like” description assumes a trace amount of magnetic impurities. By calculating the heat capacity per spin of data previously taken by Wenger and Keesom on dilute Cu Mn systems and using their data to provide a rough estimate of the number of spins necessary to explain the abundance of heat capacity at low temperatures, we estimate that the number of spins to be $\sim 10^{13}$ [58]. While this is a crude estimation of the number of spins, the number predicted by this estimate is $\sim 100$’s of ppm.

We have performed relaxation calorimetric measurements on a number of micromachined low stress silicon nitride membranes, with differences in Cu film coverage. We have observed an unusually high specific heat of the Cu film. This high $c_{Cu}$ scales with the geometry of Cu coverage, indicating that the excess heat capacity is indeed a result of the Cu film. This observation has been made despite significant differences between the geometry
Figure 4.16: Measurement of impurity concentrations using SIMS analysis indicate magnetic impurities from Mo and Fe in a “sister” film grown in similar fashion from a more sterile deposition chamber.

connecting the sample to bath, and is nearly 100x the expected heat capacity of Cu. We have analyzed the excess heat capacity of these films to understand possible sources of this dramatic enhancement of the low temperature heat capacity of these films. Both the magnitude and temperature dependence suggest that the heat capacity of the Cu films we have measured is due to a magnetic heat capacity between disperse TLS’s within the Cu film itself. We think that these TLS’s are the result of unintended magnetic impurities within the Cu lattice, and give estimates of the magnetic impurity population nearing 100 ppm. This excess heat capacity can be explained for such impurity populations by modeling the TLS interaction as it has been done in spin-glass measurements of Cu Mn and Au Fe systems.

Material analysis conducted on Cu films deposited under similar conditions, in a similar deposition system using Secondary Ion Mass Spectroscopy (SIMS) indicate indicate 10’s of ppm impurity concentration for several magnetic materials as seen in fig. 4.16. The dominant magnetic impurities present this Cu film are Mo (~50 ppm) Fe (~20 ppm) and Cr (10 ppm). If these dilute magnetic impurities interact through RKKY coupling, we can expect contributions to the heat capacity of the system not accounted for in the predicted heat capacity of Cu. These interactions at low temperature can be described by TLS tunneling model, and would account for a linear dependence with temperature, as we have observed.
Magnetic TLS interactions are a kind of Schottky anomaly, where the heat capacity is linear in temperature up to a maximum, and strongly decay as temperature is further increased. Observed maxima for CuMn and AuFe systems depend on the energy separation between TLS’s and fall near 8–12 K [37]. This description of magnetic heat capacity has been used to successfully describe heat capacity of Spin-Glass systems. More measurements to determine whether our Cu film is exhibiting other typical spin-glass characteristics would need to be performed to establish whether our Cu films are actually spin-glasses, but the qualitative description of such and excess heat capacity seems to be the only reasonable understanding that can account for such a dramatic difference in observed heat capacity.

Other explanations for observed excesses in heat capacity of noble metals includes increases of “a few percent” for Cu with significant O$_2$ and H$_2$ inclusions (100’s ppm) [41] [36]. This source of heat capacity does not account for the dramatic increase in heat capacity we have observed. Attempting to model the excess heat capacity as paramagnet interacting with a weak magnetic field does not account for the linear dependence with temperature.
Bibliography


Appendix A

Measurement VI’s

A.1 VI Library

The structure of the VI’s associated with measurements performed on AnDRe follows a library style. All VI’s currently and previously used to measure properties of the Low Temperature Devices are included in libraries within the AndreLVLBB library. This is done to facilitate communication between the different VI’s running to make measurements, perform analysis, and data produced by STAR Cryo software.

The sub libraries are organized by the measurement equipment and the goal of the measurement. For instance all measurements using the Daq card acquisition technique are organized within “Daq card librar.lvlib” and are organized within sub libraries like “Daq card static voltage variance measurements.lvlib” for measurements that are intended to measure the voltage variance without synchronizing with heating pulses. These Vi’s include measurements like Voltage Variance vs time, performing thermal conductance measurements, measuring the noise amplitude against changing temperatures, and heater powers.
A.1.1 ADR SharedVariables.lvlib

This Sub library contains all of the information provided by the STAR Cryo ADR software, and includes shared variables that allow access from the STAR software to all of the VI’s developed to interface with this control software. These shared variables include the temperature set-point of the STAR software, the 50mK stage temperature observed by the RuOx thermometer, the temperature of the magnet, 77K bath, 4K bath, magnet current, PID output Voltage etc. These variables are useful and necessary to access in order to understand the state of the magnet and ADR.

Vi’s in the “Daq relaxation measurements.lvlib” are organized to include all VI’s associated with measuring the thermal relaxation time constant of the device under measurement.

A.1.2 DaQ card library.lvlib

In order to perform nearly all measurements using AnDRe, this library will be accessed. This library contains nearly all of the VI’s used to perform measurements of the noise
Figure A.2: VI’s associated with ADR shared Variables. Each variable is labeled according to the SRS mainframe equipment responsible for measuring the quantity. Each of these variables represents the SRS output data for the equipment monitoring the value. For instance, \texttt{Mgnt}_1 represents the Current flowing through the magnet as observed from the STAR cryo panel, and the variable \texttt{921} stores the most recent measurement of the SRS 921 AC resistance bridge corresponding to the temperature of the 50mK stage.

This library is broken into two portions corresponding to measurements that are intended to observe the noise amplitude under ”static” conditions, and those used to perform relaxation measurements. Useful VI’s listed under the ”Static Voltage Variance Measurements.lvlib” heading are listed below in fig

\texttt{Kwith cal Daq version baselinemsmtcontrol.vi}

This VI is used to perform a measurement of the thermal conductance of a sample. This VI will establish temperature, apply a series of powers and measure the noise amplitude using the DaQ card. The way that these quantities are determined can be controlled from the front panel of this VI, and will remain stored for the current instance of the running VI. No ”live” updating of parameters is possible.

The method for selecting temperatures to measure $G$ of the sample is selected by either ”Autopopulate T set msmts” which will determine temperatures to measure $G$ by a populating an array with the lowest value given by
Figure A.3: Caption for daqcardlibrarylvlib.

Figure A.4: Screen capture of "Kwith cal Daq version baseline msmt control.vi". The
Appendix B

Noise Spectrum vs Temperature

Measuring the noise generated by the thermometer as a function of temperature is critical to the success of any determination of G and C, and is therefore important to establish the relationship early in the measurement period. A number of things must be done in order to rely on the validity of the measurement, and this appendix will cover some of the issues that can be presented during the measurement process.

It is critical to first establish the SQUID tune parameters that will be used at low temperature. A typically Regen (or demagnetization) will result in the base temperature of the ADR 50mK stage between 44 and 50mK. From this base temperature the power supply will allow regulation of the 50mK stage up to nearly 1K. This makes it possible to capture the noise amplitude as a function of temperature in the range of $T_{\text{base}} - 1K$. This information is very useful in determining the quality of measurement that is available on the current run.

The noise amplitude determined by measuring the variance of the voltage ($V^2$) should be extremely linear with temperature. The point at which the noise vs T curve begins to deviate from linearity at low temperature will indicate the presence of stray heating power. It is important to mitigate these effects and preserve as linear a relation as possible. Plotted below are two noise vs T curves for the same experimental period that indicate the presence of a substantial stray heating power, that was subsequently limited by diminishing the stray
EM radiation penetrating the heating cable.

Another strong indication of stray heating is made by observing the spectral composition of the temperature response. This is done by connecting the measurement cable to the SRS 770 and observing the spectral composition of the thermometer response. Any spikes in the thermometer’s response indicates that there is a signal penetrating the heater or thermometer cabling, and effort should be taken to mitigate this effect.

Steps that can be taken to reduce the effect that a stray heating signal poses include:

1) Improving the cable shielding. This can be done by tightening the Cu-Ni braiding enclosing both the heater and thermometer cabling.

2) Moving the cryostat within the room. Previous runs of data were corrupted by the presence of a large (1mV/rtHz) signal caused by the room’s proximity to large electric motors/vibrations in the room.

3) Observing the amplitude of potential stray heating signal fluctuations.
Appendix C

Niobium Bonding Wire Treatment

Procedure

Recent developments in implementing our thermometry have facilitated the use of Nb bonding wire to make electrical connections. This wire allows a fully superconducting thermometer loop for temperatures up to \( \sim 2.8 \) K. At this temperature, the Pb/Sn solder covering the PCB, or providing interconnects at the PCB will go normal. This appendix serves as a recipe for annealing, preparing, and bonding Nb wire to form superconducting interconnects for the SQUID, and Experimental setup.

We have purchased 0.001” Nb wire from ESPI. A five meter spool was purchased, and used largely to experiment with vacuum annealing recipe, and should last several cool downs. It is important to prevent any oils, or foreign substances from coming into contact with the wire, as Nb is highly reactive to a number of elements. For a single bonding, I used 20 cm of Nb wire, first cleaning the wire in a UHV cleaning process. First ultrasonically cleaning the wire in a mild deionized water and detergent mixture, then only deionized water, then Acetone, then IPA.

Once the wire is sufficiently clean, I connect the wire to the vacuum feed-through, using normal Pb/Sn solder. I make sure to connect the wire after threading one end of the wire
through a small washer (#2–3). This weight prevents unwanted twisting, and coiling of the wire. When I have soldered both ends of the Nb wire to the vacuum feed-through, I then begin evacuating the annealing chamber. It should be noted that once the wire is clean, care should be made to get the wire into a controlled vacuum environment shortly thereafter.

Be sure that the heating circuit is closed, and the resistance of the line should be ∼10’s of Ohms. Evacuating the annealing chamber to a pressure below 3 E-6 Torr is sufficient to anneal the wire. I then begin annealing the wire, by increasing the current passing through the wire in 5 mA intervals every 15 s. Increasing the current too quickly will cause the wire to break, and the process will need to be repeated. This process of increasing current is repeated until the current is slightly less than 200 mA. The wire consistently breaks for currents higher than 220 mA, and this should be avoided. When the maximum current is reached, it should be maintained for 300 s. It should be clear that heating is occurring when the Nb filament is glowing red and white hot as current is increased. First signs of heating should be observed at 40 mA.

After annealing the wire, ramp the current down, at 5 mA at 10 s intervals until no current flows. Then allow the wire to cool over the next hour or two. It is important that the wire cool completely before exposing to atmospheric pressure. At elevated temperatures, Nb is extremely reactive with atmosphere, and early exposure will render the wire impossible to bond.

Once the wire is successfully annealed, and the sample mount is ready to wire-bond, the force setting for the wire bond must be adjusted to accommodate the difference in material hardness. By increasing the bonding force, the wire bonder will be able to make connections. Opening the cover of the wire-bonder, increasing the bonding force, by rotating the force adjustment thumbscrew 5.5 turns will provide sufficient force to form ultrasonic wire-bonds.

I have had more success laying the wire to be bonded across the bond pad and making connections this way, than using the feeding mechanism of the wire bonder. With one end
properly positioned, attempting a wire-bond should flatten the Nb wire on the bond pad. For each attempt at wire-bonding, you should use a “dummy” chip to verify that the force and power settings are adequate for forming bonds, particularly on the SQUID die. Harder wire, such as Nb requires stronger bonding force and possibly more ultrasonic power. With higher settings, it is possible to damage the bond pads on the chip. This should be avoided, as it can irreparably damage the SQUID, or the device you are trying to connect.

With one free end of the Nb wire positioned, make two bonds. the first bond should allow sufficient “tail” to make another bond, and break the tail off. This can be accomplished in one step by forming a second bond, and holding the bonding tool in contact with the wire. With the second bond made, you can then attempt to gently scrape off the excess tail. These two bonds on each end also provide additional contact to the bond pad in case slight damage to the bond pad.

Performing the same process on the other end of the electrical connection, completes the bonding process. If it is difficult to form a reliable bond, slightly increasing the power, or force can aid in forming connections. This should be held to a minimum, as excess power and force can damage the substrate. Instead, starting from a different segment of wire can often be helpful.