Magnetic Properties of an Isolated Quantum Dot

by

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Abstract

Magnetic nanoparticles are a new class of materials with promising potential application in high density media storage. Understanding their fundamental physical properties is essential for future applications as well as being an interesting subject for the theoretical study of single domain magnets. This thesis provides the initial framework for the design and test of a superconducting magnetometer for use in measuring the magnetic fields from an isolated nanomagnet. A method for imaging and manipulating the nanoparticles using an AFM is developed. The design of a Josephson Junction magnetometer is provided along with testing of its basic IV characteristics. Based on these results, a new design has been submitted and is ready for testing.

Thesis Supervisor: Rajeev J. Ram
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There are several people whose guidance and support have made this work possible. Most importantly, my advisor Professor Rajeev Ram. As a student with little previous research experience he gave me the opportunity and the confidence to contribute to an exciting and challenging project. His passion for learning is contagious. He truly cares about his students both inside and outside of the lab environment. Weekend gatherings at his house were some of the most memorable times and I am proud to have been a member of his research team.

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Chapter 1

Introduction

1.1 Overview

In recent years, the ability to engineer systems with smaller and smaller dimensions has led to a new class of materials called nanoparticles. These particles exhibit many quantum mechanical properties not found in bulk form. Use of these particles in nanosystems is a growing field of study with a broad range of applications from thin film transistors to high density media storage and other systems where small scale devices are needed. As such, the study of the fundamental physics behind these nanoparticles is of great interest.

In order to use and study nanoparticles one must first be able to “see” them using appropriate imaging tools such as AFM, TEM, and SEM. However, imaging is not enough. One must be able to measure their material properties and understand the fundamental nature of how they interact with other particles and other systems.

Measuring these fundamental properties has proved to be a challenging task. Smaller devices usually bring smaller signal strength. The need to develop measurement devices that can measure such small scale signals is very important to the
growth of nanoparticle systems.

This thesis presents the study of the magnetic properties of a single isolated magnetic nanoparticle using a Josephson Junction magnetometer. A Josephson Junction is a superconducting tunnel junction whose tunneling current is sensitive to magnetic fields. In order to isolate the particles, an Atomic Force Microscope is used to do both imaging and manipulation on a nanometer scale.

This chapter will give an introduction to each component of the thesis. It begins with a brief discussion on magnetic materials and theory to give the reader the right terminology when discussing magnetic particles. This is followed by a description of the nanoparticle synthesis process along with current results and theories on their magnetic properties. Next, a description of the measurement device used in this thesis is provided to illustrate how it can measure single particle magnetic fields. At the end of the chapter a section on previous measurements of similar systems provides context for the work.

1.2 Magnetic Materials

When certain materials are placed in an external magnetic field, the magnetic dipoles inside tend to align parallel (or antiparallel) with the external field. These dipoles produce internal magnetic fields that add (or subtract) from the external magnetic field. The extent to which they add is the characteristic used to classify different types of magnets.

When one measures the total flux created from the total field, $\Phi_{\text{observed}}$ and compares it to the flux created by the external field only, $\Phi_0$, the following characteriza-
Although all materials have some magnetic properties, diamagnetic and paramagnetic materials show only slight change from the applied flux, on the order of 0.2% [3], and thus are said to be non-magnetic.

### 1.2.1 Magnetic Dipoles

A magnetic pole is the most basic building block for magnetic fields. A single pole of strength $p$ generates a magnetic field of strength,

$$H = \frac{p}{d^2}. \quad (1.1)$$

Single magnetic poles do not exist in nature. Only pairs of poles, or dipoles are found. A magnetic dipole is defined as

$$m = p \times l \quad (1.2)$$

where $l$ is the length between the two poles. As $l \to 0$ and $p \to \infty$ the dipole strength reaches a finite limit. The field from a dipole of strength $m$ is given by

$$H = \frac{m}{d^3}. \quad (1.3)$$
1.2.2 Magnetization

The total magnetic field from inside a magnetic material is often thought of as the sum of a collection of dipoles. The total dipole strength \( m \) is a function of volume and usually given in terms of its magnetization \( M \) where

\[
M = \frac{m_{\text{total}}}{V}.
\]  

(1.4)

Different materials have different magnetizations and the magnetization \( M \) is an indication of the strength of the magnetic material.

When a material is unmagnetized, all the individual dipoles add in such a way as to cancel each other completely. In this case, \( M=0 \). However, when an external field, \( H \), is applied the dipoles tend to align and \( M \) increases.

The susceptibility, \( \kappa \), is defined as the ratio of the net magnetization, \( M \), to the applied field, \( H \).

\[
\kappa = \frac{M}{H}
\]  

(1.5)

Dia-, para-, and ferromagnetic materials all show different susceptibility curves. Dia- and paramagnetic curves are both linear in \( H \). However, the slope is negative for diamagnetic substances and positive for paramagnetic.

Ferromagnetic materials have non-linear susceptibility curves. Figure 1-1 shows a typical ferromagnetic curve. Two main features are present. First, the magnetization reaches a limit where any further increase in \( H \) doesn’t produce any increase in \( M \). This is known as magnetization saturation and denoted \( M_s \).

In addition, the curve shows hysteresis. When the applied field is reduced, the magnetization does not fall to zero. Instead, it stays at some finite value. This is why ferromagnets are often thought of as permanent magnets.

In order to reduce \( M \) all the way to zero, an additional \( H \) field in the opposite
1.2. MAGNETIC MATERIALS

direction must be applied. The field necessary to achieve zero magnetization after having reached $M_s$ is known as the coercive field, $H_c$. Another key parameter is known as the switching field. This is the $H$ field necessary to take $M$ from its positive remnant state to its negative remnant state, essentially flipping the sign of the magnetization direction. Table 1.1 gives $M_s$ values for a few standard materials at room temperature.

<table>
<thead>
<tr>
<th>Element</th>
<th>$M$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>1714</td>
</tr>
<tr>
<td>Co</td>
<td>1422</td>
</tr>
<tr>
<td>Ni</td>
<td>484.1</td>
</tr>
</tbody>
</table>

Table 1.1: Magnetization (emu/cm³) at 20°

A few other properties of ferromagnets should be noted. Above a certain temperature, known as the blocking temperature, $T_B$, ferromagnets become what is know as superparamagnetic. This happens when the thermal energy ($kT_B$) is enough to flip or rotate the magnetic dipoles inside the ferromagnet. A superparamagnet cannot
CHAPTER 1. INTRODUCTION

sustain any permanent magnetization.

In addition to the blocking temperature, particle size can also lead to superparamagnetism. When the material size falls below a critical size scale, $D_p$, a ferromagnet also becomes superparamagnetic. Moreover, $D_p$ and $T_B$ are related quantities and together form a phase space relation defining regions between ferromagnetic and superparamagnetic regimes.

1.3 Quantum Dots

Quantum dots are one subclass of nanoparticles. Magnetic quantum dots are interesting because they often act as single domain particles. This may allow them to be used as single bits in high density media storage.

The magnetic quantum dots used in this thesis are provided by Moungi Bawendi and his group in the Department of Chemistry at MIT. Samples were provided by Dirk Weiss and Joe Tracy. The magnetic dots are made of Co and have length scales on the order of $3-10$ nm. The Co dots have been shown to have very small size distribution on the order of $5-7\%$. At these length scales, the dots are thought to act as single domain magnets. Such small size variation coupled with their single domain nature make these dots ideal for high density media storage.

1.3.1 Particle Synthesis

A brief description of the synthesis process is given in this section. Understanding how the particles are made and handled gives the reader a better appreciation of the challenges and limitations of these particles. For instance, when pure cobalt oxidizes, its total magnetization is reduced. The nanoparticles used in this thesis suffer from the same problem, however organic capping groups can be used to limit the oxidation.
1.3. QUANTUM DOTS

process.

The synthesis process for the Co nanoparticles used in this thesis was developed in 2001 by Dmitry Dinega. A full chemical description of the process can be found in his Ph.D thesis [1]. The basic process involves the nucleation of Co atoms into spheres. The spheres are then capped with organic surfactant to prevent oxidation and agglomeration.

Individual cobalt atoms are created through the reduction of dicobaltoctacarbonyl (DCOC) in the presence of dioctyle ether (DOE). The DCOC powder is dissolved in DOE and injected into a solution of DOE and trioctylphosphine oxide (TOPO). The solution is then heated. Around 190 to 200° C the presence of carbon monoxide indicates the reduction of DCOC and the formation of pure Co. At this stage, the Co atoms begin to nucleate and form particles. Sodium sterate is then added to serve as a capping group and to slow the nucleation process. The mixture is then allowed to cool.

The nanoparticles are separated by precipitating the mixture in ethanol, centrifuging, and discarding the excess liquid. The particles are then rediscover in hexane and excess surfactant. This process yields particles in the 3-10 nm range with size distributions of 5-7%.

1.3.2 Magnetic Properties

Room temperature measurements done in [1] show that the nanoparticles are superparamagnetic at room temperature up to at least 12 nm. Low temperature measurements revealed hysterisis below $T_B$ indicating ferromagnetic behavior. Table 1.2 gives experimental data for blocking temperatures at different size scales. The magnetic measurements were done using a SQUID, and the particles were imbedded in parafin wax.
Table 1.2: Blocking temperature vs. particle size. Data taken from [1]

<table>
<thead>
<tr>
<th>Average Size (nm)</th>
<th>$T_B$ (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.9</td>
<td>44</td>
</tr>
<tr>
<td>4.8</td>
<td>88</td>
</tr>
<tr>
<td>5.3</td>
<td>110</td>
</tr>
<tr>
<td>6.9</td>
<td>220</td>
</tr>
</tbody>
</table>

1.4 Josephson Junctions

The measurement device used in this thesis is a Josephson junction. A Josephson junction is a superconducting tunnel junction. The Josephson junction was first discovered by Brian Josephson in the early 1960's. In 1973, Josephson was awarded the Nobel prize for the discovery.

A Josephson junction is formed by a thin insulating region placed between two superconducting contacts. The Josephson effect is a macroscopic quantum effect where pairs of electrons (Cooper pairs) tunnel across the insulating barrier and form a macroscopic current. A Josephson junction can support this current, or supercurrent, without any voltage drop across the device.

The basic equations governing a Josephson junction are,

$$i = I_c \sin(\varphi)$$  \hspace{1cm} (1.6)

$$v = \frac{\Phi_0}{2\pi} \frac{d\varphi}{dt}$$ \hspace{1cm} (1.7)

$I_c$ is known as the critical current and represents the maximum current the junction can support at zero voltage. $\Phi_0$ is the magnetic flux quantum unit and $\varphi$ is the quantum mechanical phase difference across the junction. When the phase varies in time a voltage is developed across the junction. The above relations are derived in
1.4. JOSEPHSON JUNCTIONS

[4] and discussed further in Chapter 3.

It turns out that the maximum critical current is a function of magnetic flux. This effect will be discussed in Chapter 4. More precisely, \( I_c \) is sensitive to the magnetic flux threading through the insulating region. This flux dependence is given by,

\[
I_{c\text{max}}(\Phi_J) = I_c \left| \frac{\sin \frac{\pi \Phi_J}{\Phi_0}}{\frac{\pi \Phi_J}{\Phi_0}} \right| \tag{1.8}
\]

where \( \Phi_J \) is the flux through the junction and given by,

\[
\Phi_J = \mu_0 H \times A. \tag{1.9}
\]

Here, \( A \) refers to the junction area and \( H \) the magnetic field threading the junction. A plot of this function is shown in Figure 1-2. The response is analogous to that of a Fraunhofer pattern produced in optics. By measuring the change in critical current one can determine how much flux is threading the junction. In addition, the magnetic field can be determined indirectly if the area is known.

Looking at Figure 1-2 one can see that in order to obtain the highest sensitivity, the Josephson Junction should be fluxed biased to \( \Phi_J/\Phi_0 = 0.66 \). This point defines the highest slope in the \( I_C(\Phi_J) \) plot and should be used as the DC operating point for the Josephson Junction when doing sensitive magnetic measurements.

1.4.1 Calibration and Flux Bias Design

Calibration is done by designing another superconducting wire to run on top of the junction as shown in Figure 1-3. A known current is forced through the biasing wire which creates a magnetic field that threads the Josephson junction. The calibration wire can also be used to flux bias the junction to a DC operating point on the Fraunhofer pattern.
1.4.2 Chip Design

The Josephson junctions along with the control wire are designed using the KIC software package and submitted to the HYPRES foundry for processing. The junctions are designed in accordance to HYPRES' design rules [2]. The smallest junction possible with the rules is a $3 \times 3 \mu m^2$. Other junctions were obtained from Karl Berggren at MIT's Lincoln Lab.

1.4.3 Theoretical Prediction

The magnetic field a distance $r$ from a magnetic dipole is given by

$$H \approx \frac{m}{r^3} \quad (1.10)$$
The saturation magnetization of cobalt is

\[ M_s = 1422 \frac{emu}{cm^3}. \]  \hspace{1cm} (1.11)

Assuming a spherical shape and diameter of 5nm gives a dipole strength of

\[ m = MV = 7.45 \times 10^{-16} \text{ emu}. \]  \hspace{1cm} (1.12)

Using the HYPRES design, the Co dot would be roughly 4000Å from the insulating layer in the junction which gives a field of

\[ H = 11.64 \times 10^{-3} \text{ Oe}. \]  \hspace{1cm} (1.13)
The cross sectional area of the insulating region is $100\text{nm} \times 2\mu\text{m}$, or $2 \times 10^{-13} \text{m}^2$. The total flux through the junction is then

$$\Phi_J = 2.3 \times 10^{-15} \text{Oe} - \text{m}^2$$

$$\frac{\Phi_J}{\Phi_0} = 1.2 \times 10^{-4}. \quad (1.15)$$

This only leads to a very small change in the maximum supercurrent. For a maximum $I_C$ of $500\mu\text{A}$, one obtains a $\Delta i_{\text{max}}$ of $50\text{nA}$. Although this is a very small current, it can measured with highly sensitive electronics.

### 1.4.4 Measurement Technique

The Co dot will be placed on top of the biasing wire shown in Figure 1-3. The field coming from the dot will thread the junction in the same way that the flux biasing element will. Using the technique found in [5], the Josephson junction will be resistively shunted and biased at a level just above $I_{c,\text{max}}$. The theoretical sensitivity of a $1\mu\text{m}$ junction at 4.2K is $S_\Phi \approx 10^{-9}\Phi/Hz^{1/2}$ [5].

### 1.5 Previous Measurements

Magnetic measurements on individual quantum dots have proven difficult due to their small size and resulting small magnetic fields. Many measurements have been done on arrays of magnetic dots, but not much work on one isolated nanoparticle. The first main work was done by Wernsdorfer et al. using a micro-bridge-DC-SQUID [6]. Their work focused on measuring magnetic anisotropy, remnant magnetic field, and spatial switching field distribution. The Wernsdorfer group used 3nm Co-spheres embedded into the SQUID matrix in their measurements.
Another technique was demonstrated by [7] using the local Hall effect (LHE). Here, the measurement was done on individual thin-film NiFe nanomagnets. The films ranged in width from 100 to 500nm. The work focused mainly on taking hysteresis loops on the NiFe magnets and measuring the coercivity, \( H_c \).

The technique used in this thesis is similar to the Wernsdorfer approach. There, the nanoparticles are defined by ion beam etching or lift-off techniques. This thesis uses particles synthesized through a wet chemistry process. In addition, the SQUID in [6] was grown on top of the substrate already containing nanoparticles. This thesis works in the reverse manner with the particles deposited on top of the Josephson magnetometer.

### 1.6 Thesis Outline

This thesis presents the initial work in the design and testing of the Josephson magnetometer. A lot of work and effort has gone into obtaining stable Josephson IV curves. In addition, initial magnetic calibration of the Josephson magnetometer is provided using an external magnet. A method for manipulating the particles on a nanometer length scale is developed in order to be able to position the particles near the junction to provide maximum magnetic sensitivity.

Understanding the hysteresis loops from isolated magnetic dots is an important step in understanding how to engineer nanomagnets for use in high density media storage and other magnetic devices. In addition, fundamental studies of isolated magnetic nanoparticles are necessary to validate theoretical models for single domain particles.

Chapter 2 introduces the Atomic Force Microscope (AFM) as both an imaging and manipulation tool. Single particle images are presented along with a method for manipulating the nanoparticles on a mica surface.
CHAPTER 1. INTRODUCTION

The Josephson Junction is presented in Chapter 3. A brief discussion of superconductivity is given along with a more detailed explanation of Josephson dynamics. The design process is discussed along with the HYPRES growth process. In addition, experimental results of Josephson Junction measurements. Based on these results, a new design is given.

The magnetic measurements are given in Chapter 4. The theory behind a Josephson magnetometer is presented. Experimental results include the magnetic calibration of a Josephson Junction with an external magnet.

A summary of the thesis is given in Chapter 4.5. This chapter provides a discussion on the challenges and accomplishments of this work along with suggestions for future work.
Chapter 2

Atomic Force Microscopy

The Atomic Force Microscope (AFM) was initially developed as an imaging tool to provide resolution on the atomic scale. It has been used in a multitude of disciplines from magnetic media to DNA mechanics [8] [9]. Standard AFM measures surface topography by sensing the deflection of a sharp tip attached to the end of a cantilever as the tip is scanned above the sample. Recently, the AFM has been used as both an imaging and manipulation tool [10], [11], [12], and [13].

In this Chapter the AFM is used for both imaging and manipulation of the Co nanoparticles. The ability to image and manipulate nanoparticles is important because it allows one to successfully isolate particles from each other as well as position the particles close to magnetic sensing devices.

2.1 AFM Imaging

2.1.1 Theory

An AFM works by shining a laser off of the end of a cantilever beam and sensing the deflection of the beam as it interacts with the surface. For topographical imaging,
CHAPTER 2. ATOMIC FORCE MICROSCOPY

Figure 2-1: AFM setup. The laser beam is reflected from the cantilever tip to a photodetector the deflections are caused by short-range Van der Walls forces between the surface and a sharp tip attached to the end of the cantilever. Figure 2-1 shows a picture of the basic setup. Other AFM modes include Magnetic Force Microscopy (MFM), Electrical Force Microscopy (EFM), and Phase Contrast Imaging using tapping mode.

2.1.2 Tapping Mode AFM

Topographic AFM images are taken in two main modes, contact and non-contact. In contact mode the tip literally makes contact with the surface and is deflected as it runs across the surface. In non-contact mode, the tip is driven near its resonance frequency (100-300 kHz) and scanned a few nm above the surface. As the tip oscillates, it taps on the surface. When the tip is close to the surface, van Der Walls forces change the natural resonance of the cantilever. A feedback loop raises or lowers the cantilever
to provide a constant force above the surface. A detailed description of cantilever dynamics can be found in [14]. All the topographic images taken in this thesis are performed in tapping mode.

2.1.3 AFM Tip

In order to provide high resolution a sharp tip must be used. Normal tapping mode tips are made from etched silicon and have tip radii on the order of 5-10 nm. The tip is cone shaped and extends from the cantilever roughly 15-20 μm.

2.2 Sample Preparation

2.2.1 Washing

The cobalt nanoparticles are initially in a solution of hexane and excess surfactant. The hexane serves as a solvent and the excess surfactant helps prevent particle agglomeration and oxidation. In order to image single particles with an AFM, the excess surfactant must be removed and the solution diluted such that spin coating on a surface produces only a few particles per square micron.

To remove the excess surfactant, roughly 1 mL of solution is mixed with 9 mL of methanol. Since the particle capping groups are non-polar and methanol is a polar solvent, the particles come out of solution. This is known as crashing out the particles. The methanol/particle mixture is sonicated for 5-10 minutes and then centrifuged at 3900 RPM for 3 minutes. After centrifugation the heavy particles collect at the bottom of the vial and the excess liquid can be poured out. The discarded liquid is a mixture of methanol and excess surfactant. This process is usually repeated 2-3 times to provide adequate washing. After the last sequence the particles are redissolved in hexane.
2.2.2 Substrate

Since the particles are only 5-10 nm in size a suitable substrate must be chosen with surface roughness less than 1 nm. Most of the AFM imaging was done on a mica substrate. Figure 2-2 shows an AFM image of mica and Figure 2-3 shows a cross section. Mica provides surface roughness on the order of 0.05 nm over a 250 nm region.

In order to do nanomanipulation, it's also important to use a substrate that has the right sample to substrate interaction. AFM nanomanipulation is performed by literally pushing the particles across the substrate surface with the AFM tip. If the adhesion between sample and substrate is too high, the AFM tip will not be able to provide enough force to move the particles. However, if the sample to substrate adhesion is too light, the particles can be picked up by the AFM tip.

2.2.3 Deposition

After washing the particles were diluted in a 95/5 Hexane/Octane solution and spun coat on the mica substrate. The Hexane/Octane mixture provides the right solvent evaporation rate during deposition to allow the particles to evenly spread across the substrate. A single drop of solution was used during the spin coating.

2.2.4 Concentration Calibration

In order to calibrate solution concentration to deposited particle density, optical density (OD) measurements were taken on the diluted solutions. The measurement was done with a Spectronic Genesys 20 spectrophotometer at a wavelength of 325 nm with pure hexane solvent used as a baseline.
2.3 Data

2.3.1 Substrate

An AFM image of the mica substrate is shown in Figure 2-2. The images shows a 250x250 nm scan. A cross section is shown in Figure 2-3 and shows a surface roughness less than 1 nm. This makes the mica ideal to use as a substrate for single particle imaging. The RMS roughness is only 0.04 nm.

2.3.2 Concentration Calibration

Figure 2-4 shows an AFM image of an uncalibrated deposition of nanoparticles on a glass slide. The particles are stacked on top of each other such that individual particles are not isolated. In order to image isolated single particles, the need to
reduce the sample concentration is clear.

Figures 2-5-2-8 shows a series of images at different solution concentrations. A clear change in particle density can be seen. The number of particles in a 1x1 μm² scan was counted using the particle analysis tool within Digital Instruments' AFM software. The particle analysis algorithm works by setting a baseline threshold and looking for grain boundaries above the threshold.

An OD measurement was performed on each sample and compared with the particle count. The plot is shown in Figure 2-9. The number of counts at each OD value is the average of a series of four AFM images. Each image was separated by roughly 5-10 μm to reduce the effect of local particle density fluctuation. In order to have less than ten particles/μm² the OD needs to be just above the hexane baseline.

2.3.3 Single Particle Imaging

By reducing the concentration of particles on a deposited surface single particle resolution was achieved. The imaged nanoparticles range in size from 3-10 nm. A clean image is shown in Figure 2-10. With all AFM images, a smearing of particle size in
Figure 2-4: Uncalibrated deposition of nanoparticles on glass.
Figure 2-5: High concentration deposition taken in phase contrast mode.
<table>
<thead>
<tr>
<th>Concentration</th>
<th>Deposition Depth (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.25</td>
<td>0.75</td>
</tr>
<tr>
<td>0.50</td>
<td>12.5</td>
</tr>
<tr>
<td>0.75</td>
<td>25.0</td>
</tr>
</tbody>
</table>

Figure 2-6: Medium concentration deposition. 1μm scan.
the lateral dimension is seen.

2.4 Nanomanipulation

The ability to dilute and image single nanoparticles is the first step in being able to isolate particles for magnetic measurements. The imaging in the previous section has a practical limit of 5-10 particles/μm². However, the smallest Josephson junctions used in this thesis are 9 μm². As was mentioned in [10], [11], [12], and [13], the AFM has recently been shown as a suitable tool for nanomanipulation.

In order to manipulate particles on nanometer length scales, precise Positioning of the cantilever tip is required. A Digital Instruments 3100 AFM with the Nanoscope IV controller was used in this thesis. This configuration utilizes a closed loop XY position feedback. The allows for an RMS XY noise less than 2 nm [15].
Figure 2-8: Second low concentration deposition.
Figure 2-9: Particle count normalized to a 2x2 μm region. The trend shows the correlation between OD measurement and number of particles in a 2x2 μm scan. Error bars represent the statistical counting error of √N.
Figure 2-10: Single particle image. (A) 2x2μm scan with 9 particles (B) Cross section over single particle of 4nm.
CHAPTER 2. ATOMIC FORCE MICROSCOPY

In addition to the closed loop feedback for precise XY positioning, the Digital Instruments AFM employs a new software package for nanomanipulation. The combined software and hardware package is titled NanoMan [15]. NanoMan provides a graphical interface to control the tip position, obtain AFM images, and manipulate particles. Figure 2-11 shows the user interface. The arrow indicate the direction of the cantilever tip.

The tip position is controlled in a point and click fashion. Two main types of moves are allowed. First, an XY move allows the cantilever tip to be positioned accurately above the surface. A Z move raises or lowers the cantilever tip a prescribed distance. Both types of moves have variable speed controls. General operating speeds for XY moves are 500nm/sec and 10nm/sec for Z directed moves. The XY distance is controlled via the point and click method and the Z distance is controlled by a panel in the software.

After an initial scan, the tip is moved to the center of the image where it is raised off the surface and the vertical feedback is turned off. The tip position is indicated by a red crosshair in the center of the screen. To manipulate a particle the tip is first moved so that it is close to a particle, but offset to one side. The tip is then lowered to the surface using a Z move. Next, another XY move literally pushes the particle along the surface. After the second XY move the tip automatically lifts off the surface and returns to the center position. The tip motion during a move is very much like a bulldozer. This is more accurate when moving particles with sizes less than the tip. This is true in this case where the tip is about 50 nm and the particles only 5 nm in size.

Figure 2-12 shows an initial 2x2 μm² scan along with a cross section of one of the particles. The particles in the image range from 3-5 nm. Figure 2-13 shows the result on the initial particle move. The arrow indicates the direction of the cantilever
motion. The particle is moved about 300 nm. Moves up to 1 µm have been performed in both the horizontal and vertical direction.

Figure 2-14 shows the result of a series of moves. The particles have been arranged in a line. The absence of neighboring particles around the line in the right images is evidence that the particles previously in that region have been manipulated into the fabricated line.

2.5 Discussion

When doing either imaging or nanomanipulation with the AFM, sample preparation is the most important factor for obtaining good results. Poor sample preparation can prevent the user from being able to identify particles from excess surfactant as well as failure to separate individual particles in an image. The following section outlines a few common problems caused by poor sample preparation along with AFM images that highlight these events.

One main problem occurs when the sample has not been washed thoroughly.
Figure 2-12: Initial image prior to nanomanipulation. 9 particles are present in a $2 \times 2 \mu m^2$ region.
Figure 2-13: First move. (A) Arrow indicates the direction of the cantilever tip. (B) Result of nanomanipulation. The particle is moved roughly 300 nm.
enough. In this case, excess surfactant gets deposited on the sample substrate. When this happens, small speckle-like dots show up on the substrate. An example of this is shown in figure 2-15. In this particular image, the magnetic nanoparticles are still easily identified. In addition to the difference in lateral size, the excess surfactant can be identified by looking at the cross section. The surfactant shows vertical heights of only 1nm which is inconsistent with even the smallest magnetic particles [1].

Another problem that can arise is particle agglomeration. The excess surfactant helps prevent particle agglomeration. However, after the washing process, the excess surfactant is removed and the particles will agglomerate over time. The general guideline is to perform the substrate deposition within a week after the washing step. Figure 2-16 shows an image where the particles are agglomerated together. This can also occur if the particle density prior to deposition is too high.

When performing nanomanipulation the biggest issue is sample to surface and
sample to tip interaction. The AFM works as a nanomanipulation tool by literally pushing particles on a surface. However, if the surface adhesion to the sample is too large, the AFM tip will not be able to exert a large enough force and is subject to breaking or damage. In addition, if the sample to tip interaction is too large, the sample can become stuck to the tip and effectively removed from the surface.

Figure 2-17 shows an example of this interaction. The first image shows the initial image with two labeled moves that were performed. The after image shows one moved particle and one which has been eliminated from the image. The particle was probably picked up by the AFM tip. Another possibility is that the particle was pushed outside the image region. This could happen if the particle-substrate interaction was so light that the force from the AFM tip literally jettisons the particle.
Figure 2-16: AFM image showing particle agglomeration taken in phase contrast mode.

across the surface. However, this is not likely since most moves do not result in the particle being removed.

Another issue when performing nanomanipulation is that the particles can be dragged along the surface. This is a result of the raster scan during the AFM image capture. Figure 2-18 shows an example of this effect. The first image shows the initial image and a desired vertical move. The second image is the after image taken with the raster scan in the down direction. You can see that the moved particle is nudged downward as the AFM tip moves. At some point, the particle-surface interaction is strong enough such that the particle position becomes fixed. The third image shows the final result.

The remaining question is whether or not this technique can be applied to a
Josephson Junction surface. This will be necessary in order to position the nanoparticle close to the Josephson magnetometer in order to have the highest possible sensitivity. Specifically, can individual nanoparticles be imaged and manipulated on a different surface. The main obstacle is surface roughness. The mica substrate was chosen because it provides a very smooth surface. Initial surface roughness measurements on both the HYPRES and Lincoln Lab Josephson Junction chips indicate that they have surface roughness values on the order of $5-10\,\text{nm}$. Figure 2-19 shows cross sectional images for both the HYPRES and Lincoln Lab chips. The $1\mu\text{m}$ HYPRES image is taken over the top Niobium layer. The Lincoln Lab fabrication process ends by coating the entire chip with an additional insulating layer so the above image is taken over a $1\mu\text{m}$ region of SiO$_2$. Other surface issues such as surface adhesion may also play a role when attempting nanomanipulation on the Josephson samples.

2.6 Summary

In this chapter the AFM is introduced as both an imaging and manipulation tool. In this thesis the AFM is used to perform both of those tasks on cobalt nanoparticles. The ultimate goal is to be able to isolate the particles from each other and manipulate one to a region close to a magnetic sensing device. The results are summarized below.

A method for diluting the particles and doing single particle imaging has been developed. The solution density is measured with a spectrophotometer and calibrated with particle counts from a series of AFM images. This method allows the user to reliably deposit nanoparticles onto a surface to give around $5-10\,\text{particles/}\mu\text{m}^2$. Single particle AFM imaging has been performed on a Mica substrate.

The AFM has also been used to perform nanomanipulation using the new Digital Instruments NanoMan software and hardware package. Utilizing a closed loop feedback control for precise lateral control on the nanometer range, NanoMan has been
used to manipulate particles as small as 3 nm. Movements ranging from 100 nm to 1 \( \mu \text{m} \) have been performed.
Figure 2-17: (A) Initial scan with two labeled moves. (B) Result of moves. The top particle is no longer present. There is a slight drift from image A to B in the northeasterly direction.
Figure 2-18: (A) Initial scan with desired vertical move. (B) After scan. The moved particle becomes dislodged and is dragged by the raster motion of the AFM tip. (C) Final result after particle position remains fixed.
Figure 2-19: Surface roughness for Josephson samples. (A) HYPRES chip over top Nb layer. (B) Lincoln Lab chip coated with insulating SiO2
Chapter 3

Josephson Junctions

In the early 1960’s, Brian Josephson discovered what is now known as the Josephson effect. He found that two superconducting layers separated by a thin insulating region could support currents without any voltage drop across the junction. Further work showed that these currents, known as supercurrents, varied dramatically with the magnetic field through the junction. This effect allows Josephson Junctions to be used as highly sensitive magnetometers. The magnetic properties of Josephson Junctions will be discussed further in Chapter 4.

This chapter presents a basic introduction to superconductivity along with a derivation of the Josephson effect. This is followed by experimental results on Josephson Junctions with comparison to simulated results.

3.1 Superconductivity

When superconductors are cooled below a critical Temperature, $T_c$, they exhibit a series of properties which separate them from normal metals and semiconductors. Two main properties include:
CHAPTER 3. JOSEPHSON JUNCTIONS

- Zero DC electrical resistance
- Perfect Diamagnetism

When the temperature falls below $T_c$ the DC resistance in a superconductor suddenly drops to zero. The effect of zero DC resistance is that currents can exist in a superconducting wire without any dissipation. However, a superconducting wire can only support a finite amount of current. In addition, high magnetic fields can also serve to eliminate the superconducting effect. Together, the maximum current density, $J_c$, maximum magnetic field, $H_c$, and critical temperature, $T_c$, form a phase space relationship which defines the region of superconductivity. In general we will be always be working below $H_c$ and $J_c$, and I will often refer to the transition only in terms of $T_c$. The second effect, perfect diamagnetism, says that when cooled below $T_c$, a superconductor will expel all of the magnetic flux inside it. This effect will be important when discussing how an applied magnetic field is distorted near a Josephson Junction.

Another important aspect of superconductors is that current is carried by pairs of electrons known as superelectrons or Cooper pairs. A Cooper pair consists of a pair of electrons bound together below $T_c$. As such, the relevant charge and mass quantities are equal to twice that of a normal electron. Following the convention in [4], where it might be confusing all superconducting quantities are denoted with a '\(\ast\)'. The binding energy of a Cooper pair is $2\Delta$, or one $\Delta$ for each electron. This is known as the gap voltage. For Nb, $\Delta$ is 1.5mV and the gap is 3mV.

3.1.1 London Equations

In 1935 Hans and Fritz London proposed a series of relations to describe superconductivity. These relations, known as the London Equations are the starting point for
3.1. **SUPERCONDUCTIVITY**

describing electric and magnetic fields inside superconductors. The equations were first presented as a phenomenological solution to observed behavior and later derived using the more comprehensive Bardeen, Cooper, Schrieffer (BCS) theory [16] [17].

The first London equation is given by:

\[ \mathbf{E} = \frac{\partial}{\partial t} (\Lambda \mathbf{J}_s) \]  

(3.1)

where

\[ \Lambda = \frac{m^*}{n^* (q^*)^2}. \]  

(3.2)

Here, \( J_s \) is the superconducting current density. The first London equation describes the effect of zero DC electrical resistance. Note however, that an AC current can still produce a voltage. Only in DC is a superconducting wire truly lossless.

The first London equation can also be used to show that the time derivative of a magnetic field in a superconductor must decay over a characteristic length scale. This length scale is given by

\[ \lambda = \sqrt{\Lambda/\mu_0}. \]  

(3.3)

For a typical superconductor such as Nb, \( \lambda \) is on the order of 85 nm. However, the first London equation only predicts a decay for the time derivative of the magnetic field. A superconductor shows flux expulsion even for static magnetic fields. The second London equation can be used to describe the full superconducting properties and is given by

\[ \nabla \times (\Lambda \mathbf{J}_s) = -\mathbf{B}. \]  

(3.4)
This is often referred to as the Meissner effect.

3.1.2 Supercurrent

Although the above London equations accurately describe a superconductor, the phenomenon of superconductivity is really a quantum mechanical manifestation. In normal metals, scattering events tend to randomize the phase of the electron wavefunction. However, in superconductors, the phase remains a coherent quantity.

To begin, we need to introduce the concept of a probability current and the Hamiltonian for a charged quantum mechanical particle. The probability current, or flow of probability of a wave function for a free particle is defined as

\[ J = \frac{\hbar}{2im} (\psi^* \nabla \psi - \psi \nabla \psi^*) . \]  

(3.5)

For a charged particle the Hamiltonian is given by

\[ H = \frac{1}{2m} \left( \frac{\hbar}{i} \nabla - qA \right)^2 + q\phi \]

(3.6)

where \( A \) is the magnetic vector potential and \( \phi \) the electric scalar potential. This leads to the probability current for a charged particle which is given by

\[ J = Re \left\{ \psi^* \left( \frac{\hbar}{im} \nabla - \frac{q}{m} A \right) \psi \right\} . \]

(3.7)

Given the equations for the Hamiltonian and probability current it’s necessary to introduce the concept of the wave function for superelectrons. The wave function is given not for a single superelectron, but for a macroscopic ensemble of superelectrons.
3.1. **SUPERCONDUCTIVITY**

The ensemble wave function is given by

\[ \Psi = \sqrt{n^*} e^{i\theta} \]  

(3.8)

where \( n^* \) is the density of superelectrons and \( \theta \) is the phase. Note that since superelectrons are made up of two bound electrons

\[ n^* = \frac{n}{2}. \]  

(3.9)

Plugging this into the probability current equation we can define a superconducting current density.

\[ J_s = q^* n^* \left( \frac{\hbar}{m^*} \nabla \theta - \frac{q^*}{m^*} A \right) \]  

(3.10)

Here all quantities are written in terms of their *’d quantities.

It should be noted that these relations can be used to derive both the first and second London equations. Another important result which will be discussed in Chapter 4 is the concept of flux quantization. This results from the cyclic nature of the phase and allows us to define a quantity known as \( \Phi_0 \).

\[ \Phi_0 = \frac{2\pi \hbar}{|q^*|} \]  

(3.11)

\[ = 2.06 \times 10^{-15} \text{ T} - \text{m}^2 \]  

(3.12)

Another equation that will be important is the time evolution of the phase given by

\[ \frac{\partial}{\partial t} \theta = -\frac{1}{\hbar} \left[ \frac{\Lambda J_s^2}{2n^*} + q^* \phi \right]. \]  

(3.13)
3.2 Josephson Effect

3.2.1 Josephson Equations

Equations 3.7 and 3.13 are the starting point for defining the Josephson relations. Written in terms of the quantity $\Phi_0$, they are repeated below.

\[
J_s = -\frac{1}{\Lambda} \left[ A + \frac{\Phi_0}{2\pi} \nabla \theta \right]
\]  

(3.14)

and

\[
\frac{\partial}{\partial t} \theta = -\frac{1}{\hbar} \left[ \frac{\Lambda J_s^2}{2n^*} + q^* \phi \right]
\]  

(3.15)

Deriving the Josephson equations is really an exercise in solving Schrodinger's equation across a potential barrier. A model for the barrier is given in Figure 3-1. In the absence of electric or magnetic fields, zero potential is defined in the superconducting regions where the superelectrons are free particles.

Assuming no electric or magnetic fields and that the current density is the same on both sides of the junction we can write

\[
J_s(\pm a, t) = -\frac{\Phi_0}{2\pi\Lambda} \nabla \theta(\pm a, t)
\]  

\[= J_0\]  

(3.16)

(3.17)

and

\[
\frac{\partial}{\partial t} \theta(\pm a, t) = -\frac{1}{\hbar} \left( \frac{\Lambda J_0^2}{2n^*} \right).
\]  

(3.18)

The wavefunctions in the superconducting regions of the Josephson Junction are
3.2. JOSEPHSON EFFECT

Figure 3-1: $V(x)$ across the insulating barrier in a Josephson Junction

defined just as for a superconducting wire.

$$
\Psi(-a) = \sqrt{n_1^*} e^{i\theta_1} \quad (3.19)
$$

$$
\Psi(+a) = \sqrt{n_2^*} e^{i\theta_2} \quad (3.20)
$$

Plugging these into the superconducting current density equations and solving equations 3.17 and 3.18 self consistently we arrive at the defining equation for a Josephson Junction.

$$
J_s = J_c \sin(\theta_1 - \theta_2) \quad (3.21)
$$

$J_c$ is known as the critical current density and sets an upper limit on the amount of current a Josephson Junction can support. It should be noted that this is not the
same critical current as discussed for a general superconducting wire. Rather, the Josephson critical current is a function of the height and width of the tunnel barrier.

\[ J_c = \frac{e^* h}{m^* \zeta} \frac{\sqrt{n_1 n_2}}{2 \sinh(a/\zeta) \cosh(a/\zeta)} \]  (3.22)

Here, \( 2a \) is the barrier width and \( \zeta \) defines the barrier strength.

\[ \zeta = \sqrt{\frac{\hbar^2}{2m^* V_0}} \]  (3.23)

A more detailed derivation of the Josephson equation can be found in [4] and many other superconducting textbooks.

Removing the restriction on electric and magnetic fields one obtains a more detailed equation.

\[ \mathbf{J}_s = \mathbf{J}_c \sin(\varphi) \]  (3.24)

where

\[ \varphi = \theta_1 - \theta_2 - \frac{2\pi}{\Phi_0} \int_1^2 \mathbf{A} \cdot d\mathbf{l} \]  (3.25)

and

\[ \frac{\partial \varphi}{\partial t} = \frac{2\pi}{\Phi_0} \int_1^2 \mathbf{E} \cdot d\mathbf{l}. \]  (3.26)

It’s not surprising that a change in phase drives a current. We saw this in the probability current equation and a Josephson Junction gives a similar result. In addition, Equation 3.26 is known as the voltage-phase relation. It says that a change in phase will induce a voltage across the junction. Similarly, if a voltage is applied, the
3.2. JOSEPHSON EFFECT

phase will change and the current will oscillate. This is known as the AC Josephson effect. The above equations are usually written in term of \( I_c \) and \( v \) instead of \( J \) and \( E \).

\[
I_c = J_c \times \text{Area} \quad (3.27)
\]

\[
i = I_c \sin(\varphi) \quad (3.28)
\]

\[
v = \frac{\Phi_0}{2\pi} \frac{d\varphi}{dt} \quad (3.29)
\]

For a rectangular junction, the magnetic dependence on \( I_c \) is given by

\[
I_c(\Phi_J) = I_{c_{\text{max}}} \left| \frac{\sin(\pi \frac{\Phi_J}{\Phi_0})}{\pi \frac{\Phi_J}{\Phi_0}} \right|. \quad (3.30)
\]

This relation will be derived in Chapter 4. It is presented here for completeness and the reader’s awareness. Here, \( \Phi_J \) represents the magnetic flux threading the junctions insulating region. A plot of this relation is given in Figure 3-2.

Equations 3.28 and 3.29 are the defining equations for the Josephson Junction. Taking the time derivative of 3.28 we obtain

\[
\frac{di}{dt} = \left[ I_c \cos(\varphi) \frac{2\pi}{\Phi_0} \right] v. \quad (3.31)
\]

We see that a Josephson Junction can be thought of as a non-linear inductor.

In the next section we will introduce the concept of junction capacitance and resistance and model the behavior of current-biased junctions.

3.2.2 Generalized Junctions

At zero \( T \), all the electrons are bound together in Cooper pairs. However, at finite temperatures some Cooper pairs are broken. In a Josephson Junction, this provides
Figure 3-2: Theoretical magnetic dependence of $I_c$ for a rectangular Josephson Junction
a parallel path for current to flow. In addition, a capacitance is formed between the
two superconducting layers and the dielectric barrier. When put together, these three
parallel paths define a generalized Josephson Junction that is often referred to as the
Resistively Shunted Josephson (RSJ) model. A model of the circuit is given in Figure
3-3.

The junctions used in this thesis were obtained through the HYPRES foundry as
well as Karl Berggren at MIT Lincoln Labs. HYPRES quotes a junction capacitance
given by

\[ C_s = \frac{10.0}{(0.20 - 0.043 \log_{10}(J_c))} \]  \hspace{1cm} (3.32)

where \( C_s \) is given in \( \mu F/\mu m^2 \) and \( J_c \) in \( kA/cm^2 \) \[2\].

The resistive channel is voltage dependent. When the voltage is less than \( 2\Delta \), there
is not enough energy present to break a Cooper pair. However, as was mentioned
above, at finite temperatures there are normal electrons present. These electrons can
also tunnel across the barrier. For now we’ll call the resistance they provide \( R_{sg} \).
Above $v = 2\Delta$, there is sufficient energy to break Cooper pairs and almost all the current is due to normal electrons.

When a Josephson Junction is biased just above $I_c$ a voltage equal to $\pi\Delta/2e$ develops [4]. This defines the so called normal resistance.

$$I_cR_n = \frac{\pi\Delta}{2e} \text{ [Volts]}$$

(3.33)

This is known as the constant $I_cR_n$ product. It provides an inverse relationship between the critical current, $I_c$, and the normal resistance, $R_n$. In practice, the experimentally observed relation

$$I_cR_n = 0.68 \times 2\Delta \text{ [Volts]}$$

(3.34)

is used [18]. For voltages above $2\Delta$, $R_n$ is used in the RSJ model.

We are now in a position to more fully define $R_{sg}$. The two-fluid model as discussed in [4] defines the number of normal electrons present at finite temperature and zero applied voltage. The fact that there are normal electrons present at finite temperatures comes from that fact that a small fraction of superelectrons have energies above the gap energy. The relevant formula is

$$n(T) = n_{tot} \left( \frac{T}{T_c} \right)^4 \text{ [cm}^{-3}]$$

(3.35)

where $n_{tot}$ is the total density of charged particles. The assumption that’s made is that $R_{sg}$ is given by the same fraction of $R_n$.

$$R_{sg} = R_n \ast \frac{n_{tot}}{n(T)}$$

(3.36)

Typically, $R_n$ is on the order of a few Ohm’s whereas $R_{sg}$ can range from hundreds of
3.3. Junction Design

Ohm's to several kilo-Ohms at 2 K. We can write the resistive channel as a function of voltage as

\[ R(v) = \begin{cases} R_{sg} & \text{if } |v| < 2\Delta \\ R_n & \text{otherwise} \end{cases} \]  

(3.37)

3.3 Junction Design

Two sets of junction designs were used in this thesis. First, a mask design was submitted to the HYPRES foundry. Other junctions were obtained through Karl Berggren at MIT's Lincoln Laboratory. Junction sizes from both sources range from 1 \( \mu \text{m}^2 \) to 100 \( \mu \text{m}^2 \). All junctions in this thesis are square junctions, so I will often refer to their sizes in terms of only one side. For example a 10 \( \mu \text{m} \) junction is really a 10 \( \times \) 10 \( \mu \text{m}^2 \) junction.

Each junction source uses a Nb based process. The tunnel barrier is made from Aluminum Oxide to form a Niobium/Aluminum Oxide/Niobium trilayer. The gap voltage for Nb based Josephson Junctions is 1.5mV or 3.0 mV to break one Cooper pair. The critical temperature for Nb is 9.2 K.

The following section will describe the HYPRES chip design. The layout was done using the KIC software package. The HYPRES design rules can be found in [2] and a more detailed description of the fabrication process in [19]. Table 3.1 gives a brief description of each layer and their function. The HYPRES foundry can produce current densities ranging from 100 A/cm\(^2\) to 1000 A/cm\(^2\).

Figure 3-4 shows the completed design. The full chip is 5mm\( \times \)5mm. The large squares near the outside of the chip serve as contact pads for making electrical connections. The five rectangular boxes in the middle of the chip are M1 layers which serve as the bottom contact for each junction. Two to three junctions are placed on each rectangle. The rectangle in the lower right corner was unintentionally not
Table 3.1: Layers from HYPRES design process taken from [2].

connected to the outside ring of bond pads and as a result was unusable.

Figure 3-5 shows a close-up view of a single 10µm junction. The junction is located between the M2 and M1 layers. The M3 layers runs on top of the junction and functions as a flux bias control wire as discussed below.

In order to measure magnetic fields, it’s important to flux bias the Josephson Junction to the region in Figure 1-2 with the highest slope. In order to accomplish this, a control wire was designed above the junctions. The control wire is implemented using the M3 layer in the HYPRES process. Figure 3-6 shows a picture of this design. When a current is run through the control wire, its resulting magnetic field will thread the Josephson Junction. The control wire can also be used to calibrate the Josephson Junction’s magnetic dependence.

It should be noted from Figure 3-5 that there is significant overlap between the M2 and M1 layers. This creates a waveguide structure between the two layers. As derived in [20], there is an inductance per unit length whenever two superconducting layers overlap. When the wire width is greater than the its thickness the formula
Figure 3-4: Screen shot of KIC used in HYPRES submission.
Figure 3-5: Screen shot of single junction area. The junction is located between the M2 and M1 layers. The M3 layer is used as the calibration control wire.
3.3. JUNCTION DESIGN

Figure 3-6: Control wire design. Arrows indicate direction of current flow. The junction is defined between the M1 and M2 layers. The magnetic field from the control wire (M3) can thread the junction and be used for calibration and flux bias.
CHAPTER 3. JOSEPHSON JUNCTIONS

reduces to

\[ L = \frac{\mu}{W} (h + 2\lambda) \text{ [H/m]} \] (3.38)

where \( W \) is the width of the overlap, \( h \) the distance between layers, and \( \lambda \) the penetration depth. It’s possible that this inductance results in a suppressed supercurrent via an extra flux present at the junction \( \Phi \approx L \cdot I_c \) [21]. This effect will be discussed further in the discussion section.

In order to eliminate the effect of the superconducting inductance, a new HYPRES design was submitted. The full design is shown in Figure 3-7 with a close-up of a single junction in Figure 3-8. In the new design the M1 layer ends at the junction and the M2 layer begins at the junction. This causes the minimum overlap between layers. The M3 layer still runs over the junction and serves as a control wire for calibration and flux bias. To date, the new design has not been tested.

3.4 Simulation

Now that we have the full RSJ model we are in a position to simulate Josephson Junction dynamics and evaluate their performance. The total current from all three parallel channels is given by

\[ i = I_c \sin(\phi) + \frac{v}{R(v)} + C \frac{\partial v}{\partial t} \] (3.39)

Together with the voltage-phase relation

\[ \frac{\partial \phi}{\partial t} = \frac{2\pi v}{\Phi_0} \] (3.40)
Figure 3-7: Updated HYPRES design with limited overlap between M1 and M2 layers.
Figure 3-8: Close-up view of single 10 µm junction from new HYPRES submission. The M1 layer starts on the left and ends right at the junction. The M2 layer begins at the junction and continues to the right. The M3 layer is the upside down U and serves as the control wire.
these equations describe the full time evolution of the Josephson system. Written in matrix form the time derivatives are

$$
\begin{bmatrix}
\frac{\partial \varphi}{\partial t} \\
\frac{\partial n}{\partial t}
\end{bmatrix}
= \begin{bmatrix}
\frac{2\pi v}{\Phi_0} \\
\frac{1}{C} \left( i - I_c \sin \varphi - \frac{v}{R(v)} \right)
\end{bmatrix}.
$$

(3.41)

Before presenting the simulation, it’s important to have a few time constants in mind. From the model given in Figure 3-3 there’s an RC time constant that will certainly play a roll in the junction dynamics. In addition, there’s a junction constant that sets an important time scale for the system.

Suppose a constant voltage is applied to the junction. This will cause the phase to evolve linearly in time and the current through the junction will oscillate in time. The junction current will be given by

$$
i = I_c \sin \left( \frac{2\pi vt}{\Phi_0} + \varphi_0 \right).
$$

(3.42)

It’s clear that there’s a fundamental frequency, $f_J$ for the junction. Using the gap voltage for Nb based junctions of 3mV, we obtain a Josephson frequency of $f_J = 1.45 \times 10^{12}$ or 1.45 THz.

For the simulation, I’ve used the following parameters. A critical current density of 700 A/cm$^2$ and a junction size of 10 $\mu$m. These numbers are chosen to simulate the size and current densities I received from HYPRES. This gives a junction capacitance of $C=4.8$ pF, a normal resistance of 2.9$\Omega$, and a subgap of 1.3$\Omega$ at a temperature of 2K. Using the normal resistance value we can define an RC time constant equal to $\tau_{RC} = 14 \times 10^{-12}$ seconds or 71 GHz. Another important parameter is known as the Stewart-McCumber parameter, $\beta$.

$$
\beta = \frac{\tau_{RC}}{\tau_J}
$$

(3.43)
This parameter defines the relative scale between the RC and junction time constants. As we will see, when $\beta > 1$ the circuit will be hysteretic. For this simulation $\beta = 129$. Figure 3-9 shows the result of the simulation.

The reason for the hysteresis is as follows. When the current is increased from zero, the Josephson Junction time constant is much faster than $\tau_{RC}$ and the junction can remain in the zero voltage state. Once $i > I_c$ a voltage develops as the current passes through normal channels only. However, as the current is reduced below the critical current, the time constant for the capacitor to discharge is much slower than the junction time constant and a voltage remains.

Figure 3-9 shows a circuit diagram for the simulation. A 2 Volt AC sin wave was used as the source. This voltage is converted to a current through a 1 kΩ resistor. This is the same setup used in the real experiment.

Figure 3-10 shows the result of the simulation. Note that for Josephson Junction IV curves current is on the y-axis and voltage on the x-axis. Starting from the origin,
3.4. SIMULATION

As the current is increased, the voltage remains at zero. When the current exceeds $I_c$, a voltage develops. This causes the phase to oscillate as well as the current through the junction. The remaining current must travel through the RC channel. The time-average voltage through a parallel RC circuit is just given by $v = IR$. The full time average voltage is derived in [4] and given by

$$< v(t) > = iR_n \sqrt{1 - \left( \frac{I_c}{i} \right)^2} \text{ for } i > I_c. \quad (3.44)$$

As the current is now decreased, the voltage follows the hysteretic path and continues with an average voltage of $v = iR_n$.  

Figure 3-10: Josephson Junction Simulation with $\beta > 1$. 

![Figure 3-10: Josephson Junction Simulation with $\beta > 1$.](image)
3.5 Experimental Setup

3.5.1 Cryostat

The junctions are tested using a Desert Cryogenics probe station cryostat which can achieve temperatures near 1.4 Kelvin. Most superconducting measurements use immersion based cryostats. Immersion based systems offer the advantage of complete thermal contact with the sample and more flexibility in magnetic shielding. However, a probe station system allows for optical access and the potential for different types of experiments.

In the Desert system, the sample is mounted on an all metal chip carrier and screwed via a copper plate on top of the copper cold finger. The chamber is first pumped to a pressure near $1 \times 10^{-4}$ Torr. This process takes around 1 hour. Liquid helium is then forced through a transfer tube using over pressure on a helium dewar. This lowers the chamber pressure to $1 \times 10^{-6}$ Torr. In a period of 30 minutes the cold finger temperature can be lowered to 4 K. The temperature is lowered further by pumping on the helium exhaust.

3.5.2 Thermal Contact

In a probe station cryostat, much more attention to thermal contact must be made. Poor thermal contact can result in the cold finger reaching the desired 1.4 K and the sample sitting anywhere from 10 - 20K. For Nb based junctions, $T_c = 9.2$K so it’s desirable to achieve sample temperatures less than 5K. Several attempts at making the correct contact were used including GE Varnish and Apiezon Thermal Grease. These all resulted in sample temperatures ranging from 10-20 K. In the end, Indium solder was used between the sample and gold plated chip carrier. In addition, the same Indium solder was used between the chip carrier and the copper plate. The copper
3.5. EXPERIMENTAL SETUP

plate was clamped on top of the cold finger using four screws. This allowed the sample to reach temperatures roughly 0.5 K above the cold finger temperature. This can be verified by warming the cold finger to 8.5 K and watching the transition between superconducting and normal states. This effect is shown in the results section.

Another thermal issue comes from the electrical wiring. The wires begin at room temperature and travel all the way to the sample. This thermal load is dealt with by wrapping the wires several times around the cold finger before eventually reaching the sample. In addition, alloy wires are used that have good electrical conductivity, but poorer thermal conductivity compared to normal copper wire.

3.5.3 Test Circuit

In order to eliminate the effects of parasitic resistance, the Josephson Junctions are tested using a 4-point measurement technique. A 4-point measurement uses separate leads for biasing and sensing so that the voltage measured is only across the device. This type of probing is also referred to as a Kelvin connection.

Two different setups were used to test devices. First, an HP 4156C parameter analyzer was used. The 4156C is capable of accurate current biasing and voltage sensing. The current is changed in small increments from a starting to ending point. Typical current sweeps were from -2mA to 2mA in steps of 4 μA. A double sweep was usually performed meaning the current was swept from the start to stop, and then back again.

The second setup is a Function Generator/Oscilloscope setup. Figure 3-11 shows a block diagram of the setup. As in the simulations, the circuit is biased using a function generator. The voltage is swept using a 4 Vpp triangle wave at 1 kHz. As far as the junction time constants are concerned, 1 kHz is essentially DC. This allows the user to obtain many full IV curves in a short amount of time. The 1kΩ resistor
CHAPTER 3. JOSEPHSON JUNCTIONS

R Gain

Figure 3-11: Function generator electrical test circuit. The junction is current biased by converting a triangle wave voltage through a 1kΩ resistor. A four measurement is made to remove parasitic effects.

is used to convert the voltage into a current.

The current in the circuit is determined by measuring the voltage across the 30Ω resistor. This measurement is done differentially between points A and B and fed into a Stanford Research Systems (SRS) model SRS560 voltage amplifier. The junction voltage is also read differentially between points C and D and fed into a separate SRS amplifier. Circuit connections are made with coaxial RG-58 cables of length one meter. The amplifier outputs are read with an HP Infinium Oscilloscope. The
equations for the junction current and voltage are given below.

\[ i = \frac{v_{AB}}{R_{gain} \times 30} \]  \hspace{1cm} (3.45)

\[ v = \frac{v_{CD}}{J_{gain}} \]  \hspace{1cm} (3.46)

### 3.5.4 ESD Precautions

It should be noted that Josephson Junctions are extremely ESD sensitive. Careful handling and test procedures must be employed in order to avoid damaging the junctions. The following ESD protocol was used during the testing and handling of all devices.

1. A grounding strap was worn at all times to prevent discharge from the handler to the device and grounded through an outlet ground.

2. A SIMCO CenturION 9e single fan ion blower was used for at least 5 minutes prior to bonding and kept on during bonding.

3. Any soldering is done with an ESD soldering iron and always below 200°C. Temperatures exceeding 200°C can cause Nb migration through the insulating barrier creating an effective short.

4. Junction leads are shorted together externally to prevent any voltage discharge while pumping on cryostat chamber and during cooling process. The leads are reshorted after electrical testing is completed.

5. Wait to do any electrical testing before the radiation shield falls below 30 K.

Many Josephson Junctions were damaged before implementing this protocol. Many thanks to Professor Terry Orlando’s group at MIT and to Karl Berggren at MIT’s
Lincoln Lab for advising on the proper protocol. The above protocol was used roughly 10 times and gave a success rate of around 80%.

3.6 Results

3.6.1 Measurements

The following section gives a series of measurements along with a discussion of their main features and characteristic values. All the measurements were done at a cold finger temperature of 1.4K unless otherwise noted. This indicates a sample temperature of 2K.

A typical IV curve taken with the 4156C is shown in Figure 3-12. The junction was an 8μm junction from the HYPRES design with a current density of 1042 A/cm². This gives an expected $I_c$ of 666.8μA, $R_n$ of 3Ω, $R_{ss}$ of 1.37 kΩ, $C$ of 5.5pF, and $\beta$ of 89. The sweep is a double sweep from -2 mA to 2 mA in 4 μA steps.

Two main features on the above curve are different from the theory. First, the maximum supercurrent I see is less than the expected $I_c$. Every junction tested to date has shown some form of suppressed supercurrent. The best observed results still show a suppressed supercurrent around 70% of $I_c$. Another feature missing is the presence of any hysteresis. The sweep follows the same curve in both directions. This is true even if the sweep starts from 0 current instead of -2mA.

Another HYPRES junction was tested on the Function Generator setup. This is shown in Figure 3-13. This time a 10μm junction with a critical current density of 740 A/cm². This gives junction values of $I_c = 740\mu\Lambda$, $R_n = 2.75\Omega$, $R_{ss} = 1.23k\Omega$, $C = 4.86pF$, and $\beta = 122$. The function generator was set to a triangle wave at 5 VPP and 326 Hz.

Once again, there is a suppressed supercurrent and lack of hysteresis. One thing
Figure 3-12: 8μm junction taken with the 4156C. $I_c = 666.8μA$ and $\beta = 89$. The IV curve shows no hysteresis along with a suppressed supercurrent.
to note is that the function generator measurement is much nosier than the 4156C. In addition, the function generator plot shows several sweeps superimposed on each other. Typically, 5-20 sweeps/data set are given.

The testing of Josephson Junctions obtained from Karl Berggren was an attempt to test whether or not there was something wrong with either of the two setups or the HYPRES chip design. Figure 3-14 shows an IV curve of a 10μm junction taken with the 4156C setup. The expected $I_c$ is 700 μA. The same junction taken with the function generator setup is shown in Figure 3-15. The two plots show the same level of supercurrent suppression along with an absence of hysteresis. This seems to indicate that the two setups can produce the same results.

Figure 3-16 shows a comparison between the simulated IV curve with data from
Figure 3-14: 10µm junction from Karl Berggren taken with the 4156C. $J_c = 700$ A/cm²
Figure 3-15: 10μm junction from Karl Berggren taken with the function generator. 
$J_c = 700 \text{ A/cm}^2$
3.6. RESULTS

Figure 3-16: Comparison between theory and measurement for the 10μm Lincoln Lab junction. The two measurement setups line up roughly on top of each other.

the Lincoln Lab junction using both measurement setups. The two measurements are in fairly good agreement with each other. Aside from the lack of hysteresis and suppressed $I_c$, the two measurement curves show a decrease in $R_n$. This is surprising since one would expect the $I_c$ to be higher than the measured value. An increase in $I_c$ would indicate a lower $R_n$ value from the constant $I_cR_n$ product. The voltage gap lines up nicely with the theory.

The only evidence of potential hysteresis was obtained accidentally. When the B input from Figure 3-11 was connected to the SRS amplifier, but not to the 30 Ω resistor the appearance of hysteresis was seen. This is shown in Figure 3-17. However, making the full connection on the B input removed the hysteresis. An image of real hysteresis can be seen in Appendix B. This plot was taken by Karl Berggren at Lincoln Lab.
Figure 3-17: Appearance of hysteresis taken with the B input on the SRS disconnected. Taken with Lincoln Lab Josephson Junction.
3.6. RESULTS

The above measurements were all taken with a cold finger temperature of 1.4 K. If the temperature is raised above roughly 8.5 K, a change in the IV curve is seen. This indicates that the sample is a little over 0.5 K above the cold finger temperature. At this point, the junction appears simply as a resistor whose value is equal to $R_n$. This effect is seen in Figure 3-18. The 10μm junction is the same as in Figure 3-13. Sharp IV changes above and below the critical temperature are seen in both setups. Recooling to $T < T_c$ brings the junction back into its superconducting state.

3.6.2 Junction Failure

Prior to implementing the proper ESD protocol nearly half of the attempts to take Josephson Junction measurements resulted in junction failure. ESD is an issue be-
cause the oxide layer creating the tunnel junction is extremely thin. Large voltage spikes can cause Nb pinholes to form through the oxide. After failure, the junction is seen as a short below $T_c$. Figure 3-19 shows an example of junction failure.

3.7 Discussion

Unfortunately, to date, the Josephson Junction measurements do not match with the theory. The theoretical predictions based on junction size and critical current density indicate that the junctions should have high $\beta$ values and show hysteresis. In each setup, this effect is not seen.

In order to remove the occurrence of hysteresis, $\beta$ must be lowered to one or lower. A typical $\beta$ predicted from the theory is around 90. Assuming that the junction

Figure 3-19: Example of junction failure caused by ESD.
capacitance is accurate, we would need to shunt the Josephson junction with a very low resistor.

\[
\frac{\beta_{\text{theory}}}{\beta_{\text{observed}}} = \frac{90}{1} \quad (3.47)
\]

or

\[
\frac{R_n}{R_{\text{eff}}} = \frac{90}{1} \quad (3.48)
\]

where \( R_{\text{eff}} \) is the effective resistance caused by the parallel combination of \( R_n \) and \( R_{\text{shunt}} \).

\[
R_{\text{eff}} = \frac{R_{\text{shunt}}R_n}{R_{\text{shunt}} + R_n} = \frac{R_n}{90} \quad (3.49)
\]

Plugging in a value of 3\( \Omega \) for \( R_n \) we get a needed shunt resistor of 0.034\( \Omega \). This seems rather unlikely and there is no other evidence to support such a short. The effect of stray capacitance would only serve to increase the \( \beta \) and is not an explanation that could remove hysteresis.

The appearance is a suppressed supercurrent is easier to comprehend. The effect of magnetic fields on Josephson Junctions will be discussed further in Chapter 4. However, we have already the theoretical dependence of the maximum critical current when magnetic flux threads the junction. If any background magnetic flux or magnetic flux noise is in the system, the maximum supercurrent can become suppressed.

One potential reason for magnetic noise is insufficient magnetic shielding. In a probe station cryostat it is more difficult to shield the junction from ambient magnetic fields due to space issues. Typically, Josephson Junction circuits are tested in liquid
Helium dewars where large Mu-metal magnetic shield can be used. These cans are often lined with a Pb or In which have transition temperatures of 7.2 and 3.4 K respectively. If the can is cooled below those temperatures, the superconducting lining will serve to expel any flux inside the chip region. I would suspect that more attention to magnetic shielding is necessary to reduce the overall noise and limit any flux trapping.

Evidence for this conclusion comes from the day to day observation of the overall stability of the measurement. When the cryostat vacuum pump is running during a measurement, even the highest observed supercurrent is only stable for a few seconds. The highest supercurrent can be reached by warming the system above $T_c$ and recooling. This procedure takes advantage of a superconductor's ability to expel all flux when cooled below $T_c$. Turning off the vacuum pump during the measurement allows the junction to become relatively stable over a time scale of a few minutes. In addition, the ion gauge used to measure the pressure in the cryostat chamber is made from a large magnet. Removing the ion gauge helped increase the maximum observed supercurrent.

As was mentioned earlier, the effect of the inductance between two superconducting wires was not taken into account in the first HYPRES design. The inductance per unit length is again given by

$$ L = \frac{\mu_0}{W} (h + 2\lambda) \quad (3.51) $$

In the HYPRES design, the width, $W$, of the overlap is equal to the junction size. The height between the M1 and M2 layers is 400 nm, and the average overlap length is roughly 300$\mu$m. A 10$\mu$m junction gives an total inductance of 21 pH. At an $I_c$ of 700$\mu$A, this provides a total flux of 7.3$\Phi_0$. This flux is certainly not all threading the junction. However, it's not unreasonable to assume that some portion of this
extra flux is threading the junction and causing an supercurrent suppression for the HYPRES junctions. This was the motivation for the new HYPRES design and has yet to be tested.

Although there remain a handful of issues to overcome, several accomplishments from this work can be identified. First is the development of an ESD protocol for handling, bonding, and testing of Josephson Junctions in cryostat probe station. The protocol has been used many times and significantly reduces the chance of junction failure. In addition, thermal problems have been removed and we reliably cool the system to 1.5 K.

Some positives can also be taken from the Josephson measurements. The gap voltages are consistent with the theoretical values and the normal resistances are in the right range. This indicates that in large part, the Josephson effect can be observed using this setup. In addition, in order to do magnetic measurements, it's not completely necessary to have any hysterisis. In fact, it would probably be useful to remove the hysteretic effect to eliminate any ambiguity in the magnetic measurement. The Josephson Junctions still showed magnetic dependence as shown in Chapter 4. In order to do more sensitive measurements, it will still be necessary to remove some of the noise from the system.
Chapter 4

Magnetic Measurements

Having successfully observed the Josephson effect we are now in a position to look for magnetic dependence. The magnetic dependence of Josephson junctions has been studied both theoretically and experimentally [22] [23] [24]. In addition, the magnetic dependence of Josephson Junctions are the basis for work in superconducting quantum computing using SQUID based devices [25] [26] [27].

The magnetic dependence of Josephson Junctions is a consequence of the quantization of magnetic flux in a closed path. The result is a dependence on the maximum critical current given by

\[ I_{c_{\text{max}}} = I_0 \left| \frac{\sin\left( \frac{\pi \Phi_J}{\Phi_0} \right)}{\frac{\pi \Phi_J}{\Phi_0}} \right| \]  

(4.1)

where \( \Phi_J \) is the flux threading the junction barrier and \( I_0 \) is the maximum critical current at zero applied flux. Note that the Josephson Junction is sensitive to magnetic flux and not magnetic fields. In this way, Josephson Junctions can be used to measure magnetic flux in fractions of \( \Phi_0 \).

This chapter will provide a derivation of the above relation along with experi-
mental results of the magnetic dependence of a Josephson Junction. To date, the Josephson Junctions used in this thesis are not sensitive enough to measure single magnetic nanoparticles. A discussion on the current sensitivity and limitations of the Josephson magnetometer will be given along with suggestions for improving future performance.

4.1 Theory

4.1.1 Flux Quantization

The quantization of flux in superconductors is an inherently quantum mechanical phenomenon which results from the cyclic value of the phase in the superconducting wave function. The derivation below roughly follows that in [4].

We begin with the supercurrent equation given by

$$J_s = -\frac{1}{\Lambda} \left[ A - \frac{h}{q^*} \nabla \theta \right]. \quad (4.2)$$

Integrating this equation around a closed path yields

$$\oint_C (\Lambda J_s) \cdot dl = -\oint_C A \cdot dl - \oint_C \frac{h}{q^*} \nabla \theta \cdot dl. \quad (4.3)$$

By taking the integration path well inside a superconductor the left hand side of the equation becomes zero. This is a consequence of the second London equation which shows that magnetic fields decay to zero in a superconductor. The decay length is given by $\lambda$ and is referred to as the penetration depth. For Nb, the penetration depth is only 85nm. As such, any current found in a superconducting wire flows near the surface.

Using Stokes theorem the first part of the right hand side can side can be converted
4.1. **THEORY**

To a surface integral.

\[
\oint_C A \cdot dl = \int_S (\nabla \times A) \cdot dA = \int_S B \cdot dA = \Phi \tag{4.4}
\]

This is now recognizable as the magnetic flux crossing the surface boundary normal to the closed loop. The last part of equation 4.3 is given by

\[
\oint_C \theta \cdot dl = \lim_{a \to b} \theta_b - \theta_a = 2\pi n \tag{4.7}
\]

The phase is measured modulo 2\pi which leads to the above result. Putting it all together we obtain

\[
\int_S B \cdot dA = \frac{2\pi \hbar n}{q^*} \tag{4.9}
\]

The result, is that any magnetic flux in a superconductor must be quantized. This also leads to the definition of \( \Phi_0 \) which is known as the quantum flux unit.

\[
\Phi_0 = \frac{\pi \hbar}{e} = 2.06 \times 10^{-15} \text{Tm} \tag{4.10}
\]

4.1.2 **Josephson Dependence**

The derivation of the magnetic flux dependence of a Josephson Junction follows a similar procedure used for a single superconducting wire. This time, we are concerned
with the gauge invariant phase, $\varphi$, given in equation 3.25. In Chapter 3, we assumed that the current density and phase difference across a junction were constant over the junction area. In the presence of a magnetic field, this is not true. The full phase dependence is given in equation 3.25 and repeated below.

\[
\varphi = \theta_2 - \theta_1 - \frac{2\pi}{\Phi_0} \int_1^2 \mathbf{A} \cdot d\mathbf{l}
\]  \hspace{1cm} (4.12)

We are interested in the change in $\varphi$ across the length of the junction. To begin, we'll look at the difference between two points separated by a distance $\Delta y$. We actually use four points to make a closed path as shown in Figure 4-1. A relation from SQUID analysis in [4] yields

\[
\varphi(P) - \varphi(Q) = 2\pi n + \frac{2\pi \Phi}{\Phi_0} + \frac{2\pi}{\Phi_0} \oint \Lambda J \cdot d\mathbf{l}.
\]  \hspace{1cm} (4.13)

The last part of equation 4.13 vanishes since the total current in the closed path is zero. The rest of the equation shows that $\varphi$ is a function of the magnetic flux within the closed loop.
4.1. THEORY

There cannot be a magnetic field in the z direction due to the second London equation. However, the insulating region can support magnetic fields and we are free to have magnetic fields there in either the x or y directions. Assuming a uniform field, we are free to choose our axis such that $B = B_z$ such that

$$\Phi = B_x(2\lambda + d)\Delta y$$

(4.14)

where $d$ is the insulating barrier thickness and $\lambda$ the superconducting penetration depth. Remember that magnetic fields are present within a superconducting layer up to a length given by $\lambda$. Taking the limit as $\Delta y$ goes to zero we obtain

$$\frac{\partial \phi}{\partial y} = \frac{2\pi B_x \ast h_{eff}}{\Phi_0}$$

(4.15)

Here, $h_{eff}$ represents the effective “magnetic thickness” of the Josephson Junction where magnetic fields are present. Integrating this equation along the y direction yields

$$\varphi(y) = \frac{2\pi}{\Phi_0} B_x h_{eff} + \varphi(0)$$

(4.16)

Plugging this into the Josephson equations we obtain the full magnetic flux dependence.

$$J = J_c \sin \varphi(y).$$

(4.17)

The total current is found by integrating over the area of the junction. For a square junction or width $w$

$$i = \int_{w/2}^{w/2} J_c dx \int_{w/2}^{w/2} \sin \varphi(y) dy.$$
Direct integration and a few trigonometric identities yields

\[ i = I_c \frac{\sin\left(\frac{\pi \phi_j}{\Phi_0}\right)}{\frac{\pi \phi_j}{\Phi_0}} \sin(\varphi(0)). \]  

(4.19)

Since we are only interested in the maximum supercurrent allowed we can set \( \varphi(0) = \pi/2 \) and take the absolute.

\[ i = I_c \left| \frac{\sin\left(\frac{\pi \phi_j}{\Phi_0}\right)}{\frac{\pi \phi_j}{\Phi_0}} \right| \]  

(4.20)

This the same as the Fraunhofer diffraction pattern found in optics. In fact, another way to obtain the magnetic dependence of a Josephson Junction is to take the Fourier transform of its geometry. A square junction yields the above \( \sin(\pi x)/\pi x \) pattern.

We have shown that the maximum supercurrent found in a Josephson Junction is a function of the magnetic flux through the insulating region. This is the basis of the Josephson magnetometer. The Josephson magnetometer can measure magnetic flux in fractions of the quantum flux unit \( \Phi_0 \). Since \( \Phi_J = BA \), larger junctions will provide greater magnetic sensitivity.

### 4.2 Experimental Setup

The external magnetic field was applied via a strong disk magnet of diameter 53 mm and thickness 6 mm. The magnet was suspended above the cryostat directly above the chip location. The vertical distance from the magnet to Josephson sample was varied and the resulting maximum critical current was measured. Figure 4-2 shows a picture of the setup.

The magnetic field strength was first measured using a Lakeshore 421 Gaussmeter. This Gaussmeter uses a Hall probe to measure magnetic field strength. Measurements
4.3. RESULTS

Figure 4-2: Setup for magnetic field measurement. The magnet is suspended above the sample and the vertical distance varied.

were taken along the same distance scales as used when positioning the magnet above the Josephson sample.

Before each measurement, the cold finger temperature was heated above 20 K and recooled to 1.4 K. This was done in order to remove any trapped flux in the system. The magnet was then placed above the sample and lowered to the appropriate height. The maximum critical current for a particular run was determined by the maximum measured current with corresponding voltage less than 50μV.

4.3 Results

A plot of the magnetic calibration curve is shown in Figure 4-3. The distance was varied in 0.5 cm increments. The background magnetic field was 0.30 Guass. This data was used to estimate the magnetic field seen by the Josephson Junction when
Figure 4-3: Magnetic calibration curve. Data points taken in 0.5 cm increments. Hall probe oriented perpendicular to the magnetic.

the magnet was positioned at an equivalent distance above the sample.

Figure 4-4 shows the result of the measured maximum critical current versus magnet distance for the 10µm Lincoln Lab junction. The $J_c$ for this sample is $700\,\text{A/cm}^2$ which gives a theoretical zero field critical current of $700\,\mu\text{m}$. Two trials are shown which show the same general trend. The maximum difference between the two trials in current is $72.8\,\mu\text{A}$. The minimum difference is $6.6\,\mu\text{A}$ with a mean difference of $31.6\,\mu\text{A}$. There are 22 data points in each trial with distances ranging from 12 to 22.5 cm in 0.5cm steps.

The distance values were converted to magnetic field using the calibration data. The resulting plot is plotted in Figure 4-5 against the theoretical Fraunhofer pattern. A zero field data point is added to the data from Figure 4-5. The zero field maximum critical current is only $470.2\,\mu\text{A}$. The theoretical magnetic flux dependence is converted
Figure 4-4: Three data sets of maximum critical current versus magnet distance for a 10μm junction with $I_c = 700μA$. 
Magnetic Field vs. Critical Current

Figure 4-5: Three data sets of maximum critical current versus magnetic field for a 10μm junction with $I_c = 700\mu$A.

to magnetic a magnetic field dependence by using the known area of the junction. The 10μm junction has an effective area of

$$A_{eff} = 10\mu m \times (2\lambda + a)$$  \hspace{1cm} (4.21)

where $\lambda$ is the penetration depth of 85 nm and $a$ is the insulating thickness of 3 nm.

### 4.4 Discussion

The magnetic dependence of a Josephson Junction does not depend on the hysteretic nature of the junction. However, the noise issues from the discussion on basic junction dynamics can effect the magnetic measurement. At the present time, the variability
and noise in measuring the maximum supercurrent is too high to measure single nanoparticles.

Suppose a 10\(\mu\)m junction was flux biased to the point of highest slope in the Fraunhofer pattern. This is a bias point of \(\Phi_J/\Phi_0 = 0.66\) which corresponds to a magnetic field of 7.85 Guass. Using the mean difference in maximum supercurrent between data sets of 31.6\(\mu\)A we can obtain a rough estimate for the sensitivity of the Josephson magnetometer. A 30\(\mu\)A spread around the bias point gives a 0.37 Guass spread in magnetic field. The expected magnetic field for a single nanomagnet located 400 nm above the junction is only 1.46E-4 Guass.

One thing to note is that the magnetic field calibration data was taken outside the cryostat and at room temperature. This is important because the true magnetic field threading the junction is most likely different than that used above. One reason for this difference is that magnetic field lines are warped by the superconducting layers via the Meissner effect.

It should be clear that external magnetic fields can couple into the system and reduce the suppercurent seen in the Josephson Junction. Even when the magnet is completely removed from the vicinity of the cryostat, the zero field supercurrent is only 470\(\mu\)A. If there were no other noise sources present, this would indicate a magnetic field of 5.6 Guass threading the junction. This seems rather high considering the earth’s magnetic field is only 0.5 Guass. However, better shielding must be implemented to reduce any magnetic coupling to the external world.

Even though the noise, magnetic or electrical, is too high to measure magnetic fields on the magnitude of the nanoparticles, the initial magnetic dependence of a Josephson Junction has been shown. The next step will be reducing the variability in the supercurrent measurement and getting more accurate results on the magnetic field the junction sees. In addition, other designs such as SQUID technology may be
used to achieve higher coupling between sample and the magnetometer. A SQUID has the advantage of increasing the effective area for flux coupling.

### 4.5 Summary

In this chapter the theory behind the magnetic flux dependence of Josephson Junctions has been shown. In addition, initial data on the magnetic dependence for of a 10μm, 700μA junction is given. The magnetic dependence shows the right general form, although the overall noise limits the sensitivity of the Josephson magnetometer.
4.6 Summary

The goal of this thesis is to present an initial framework for the design and testing of a superconducting magnetometer for measuring isolated magnetic quantum dots. A method for imaging and manipulating nanoparticles on a mica surface is presented along with testing of a Josephson Junction magnetometer using a probe station cryostat.

Chapter 2 presents the AFM as both an imaging and nanomanipulation tool. A method for preparing the 5 nm Co nanoparticles for clean imaging is given. An optical density measurement is used to calibrate particle density before deposition on a mica substrate. This technique allows the user to image single nanoparticles. A series of nanomanipulations on a mica substrate are performed with moves ranging from 30 nm to 1 μm. The mica substrate was chosen because it has surface roughness less than 1 nm. Initial testing on the superconducting chips shows surface roughness in the 5-10 nm range.

The Josephson Junction is introduced in Chapter 3. The reader is provided a basic introduction to superconductivity and the Josephson relations are derived. Simulation of a 10μm junction is given with an explanation of the expected IV characteristics. Experiments are performed on two sets on junctions. First, self-designed junctions fabricated at the HYPRES foundry. These junctions show a suppressed level of supercurrent. This is possibly caused by a self-inductance due to the overlap between the two superconducting layers that form the Josephson Junction. A new HYPRES process has been submitted and received that minimizes this overlap, but has not yet been tested. Other junctions were obtained from Karl Berggren at MIT Lincoln Lab. These junctions show a higher level of supercurrent although still show some
suppression caused by electrical and magnetic noise. Both junction types failed to show the expected hysteresis.

Chapter 4 introduces the Josephson Junction as a magnetometer. The magnetic flux dependence on the maximum supercurrent is derived along with experimental data from the Lincoln Lab chip. The external magnetic field is provided via a strong disk magnet suspended above the cryostat. The average trial to trial difference is 31.6 μA or 0.37 Gauss when flux biased to the maximum slope on the theoretical Fraunhofer pattern.

4.7 Future Work

The following section provides suggestions for future work related to this thesis. A series of challenging steps remain in order to successfully measure the magnetic properties on an isolated quantum dot. I hope that this thesis has provided the proper framework for taking those steps and the following suggestions will prove helpful in accomplishing the overall goal.

4.7.1 AFM

Surface Roughness and Nanomanipulation

The ability to image and manipulate nanoparticles has been demonstrated on a mica substrate. However, these techniques may not translate to other surfaces. Surface roughness is the key issue. The initial testing on the superconducting chips show that they are too rough to image single nanoparticles. However, conversations with Karl Berggren indicate that the Lincoln Lab process can fabricate chips with a surface roughness less than 1 nm.

Confirmation of these roughness numbers along with deposition of nanoparticles...
onto the surface is a necessary next step. If the raw surface still proves challenging to work with, other surface treatments may be necessary to provide a smooth surface and the correct level of sample to surface adhesion. The ability to deposit and manipulate particles on the superconducting surface will allow the user to position the magnetic nanoparticles for maximum magnetic sensitivity.

4.7.2 Josephson Junctions

Experimental Setup

Further understanding of the experimental setup and how it affects the dynamics of the Josephson Junctions is necessary. To date, although general Josephson behavior is clearly present, the lack of hysterisis is troubling because it indicates that there is an unknown effect taking place.

The first place to look is the electrical setup. Questions that remain unresolved include whether or not any parasitic resistance, capacitance, or even inductance is pushing the Josephson Junctions into a non-hysteretic regime. In addition, the noise levels on the function generator setup produce switching events with a width of around 20 $\mu$A. A more accurate measurement of the switching event is needed to perform sensitive magnetic measurements.

Magnetic Shielding

Another source of noise is magnetic. The same effect that is the basis for the sensitive magnetic measurement can serve to suppress supercurrent if stray magnetic fields are present. Typical shielding mechanisms include Mu-metal shielding and enclosing the device in a superconducting shield [26]. These techniques should be investigated taking into account the spatial limitation of a probe station cryostat.
Second HYPRES Sample

The second HYPRES submission has yet to be tested and should be the next step for anyone continuing this work. The new design minimizes the self-inductance caused by overlapping layers and should provide a good framework from which to improve testing and design techniques. The HYPRES design contains a control wire for more direct magnetic calibration and flux biasing. If the HYPRES design works, but the surface roughness issues are still unresolved, it may be possible to incorporate the design into the Lincoln Lab process which may provide a smoother surface.

4.7.3 Magnetic Measurements

Magnetic Modeling

A better understanding of how external magnetic fields are warped in the presence of a superconductor is needed. Specifically, what is the local field threading the junction given an external magnetic source. This is needed to confirm the measured magnetic dependence and put a better bound on the limits of the Josephson Junction magnetometer.

4.8 Final Words

The ability to measure the magnetic properties of an isolated quantum dot remains a challenging and worthwhile goal. A superconducting magnetometer can provide the necessary sensitivity although its implementation has proven more difficult than previously expected. The final Josephson Junction magnetometer used in this thesis is sensitive to magnetic field on the order of 0.5 Gauss.

The most important accomplishment in this work is the ability to image and manipulate nanoparticles on nanometer length scales. This provides a framework for
positioning nanoparticles close to a magnetic sensing device. Experimental work is still needed to extend this technique to other surfaces more likely used for magnetic sensing.
Appendix A

Matlab Code

A.1 Josephson Simulation

jjsimul.m

% Josephson Junction
% Simulation Code

global Rn
global Phi0
global C
global T
global t
global Ic
global Time
global Y
global scale
global R1
global R2
global Delta
global Rsg
global Rshunt

close all

%Junction Size
% Current Density
\[ J_c = 700 \text{ A/cm}^2 \]

% Some Constants
\[ \Phi_0 = 2.06 \times 10^{-15} \text{ T-m}^2 \]
\[ \varepsilon_0 = 8.85 \times 10^{-12} \text{ F/m} \]
\[ \Delta = 1.5 \times 10^{-3} \] % Normal gap voltage
\[ \mu_0 = 4\pi \times 10^{-7} \text{ H/m} \]
\[ \lambda_0 = 33 \times 10^{-9} \text{ nm} \]
\[ m_e = 9.1 \times 10^{-31} \text{ kg} \]
\[ q = 1.6 \times 10^{-19} \text{ C} \]
\[ T = 2 \text{ Kelvin} \]

% Resistors
\[ R_1 = 1 \times 10^3 \]
\[ R_2 = 30.2 \]

% Critical Current
\[ \text{Area} = (jsize \times 10^{-6})^2 \]
\[ I_c = J_c \times \text{Area} \times (100)^2 \]

% Taken from constant \( I_cR_n \) product
\[ R_n = 0.68 \times 2 \Delta / I_c \]

% Calculation of subgap resistance
\[ m_{star} = 2 \times m_e \]
\[ q_{star} = 2 \times q \]
\[ n_{star} = m_{star} / (\lambda_0 \times 2 \times q_{star} \times 2 \times \mu_0) \]
\[ n_{tot} = 2 \times n_{star} \]
\[ n = n_{tot} \times (T / 9.3)^4 \]
\[ R_{sg} = R_n \times n_{tot} / n \]

% Shunt resistor for changing beta value
\[ R_{shunt} = 1 \times 10^6 \]
A.1. JOSEPHSON SIMULATION

% Barrier Thickness - In the nm range
\( a = 3 \times 10^{-9} \); \( \text{nm} \)

% Junction Capacitance
\[
C = \frac{10}{(0.20 - 0.043 \times \log_{10}(Jc/1\times10^3)) \times 1 \times 10^{-15} \times \text{Area}/1 \times 10^{-12}}
\]

% Time Constants
\[
\text{Tau}_{J,Rn} = \frac{\Phi_0}{2\pi I_c R_n};
\]
\[
\text{Tau}_{RC} = R_n C;
\]

\( \beta = \frac{\text{Tau}_{RC}}{\text{Tau}_{J,Rn}} \);

% Create time vector
\[
T = \text{Tau}_{J,Rn};
\]
\[
scale = 1 \times 10^5;
\]
\[
\text{points} = 500;
\]
\[
t = \text{linspace}(0, 0.5 \times \text{scale} \times T, \text{points});
\]

% Run ODE solver
\[
\text{options} = \text{odeset}('\text{RelTol}',1 \times 10^{-3},'\text{AbsTol}',1 \times 10^{-6});
\]
\[
[\text{Time}, Y] = \text{ode23s}('\text{testder2}', t, [0 0], \text{options});
\]

% Calculate current
\[
\text{Itot} = (\text{voltage}(t)' - Y(:,1))/(R_1 + R_2);
\]

figure
plot(Y(:,1), Itot);

\text{title}(['\beta = ', \text{num2str}(\beta), ', R_n = ', \text{num2str}(R_n)]);
xlabel('Voltage');
ylabel('Current');
grid on
axis([-1 \times 10^{-3} 4 \times 10^{-3} -0.2 \times 10^{-3} 1.2 \times 10^{-3}])

\text{testder2.m}
% Returns the derivative for the voltage and phase
% relations in a Josephson Junction

function dydt = testder2(t,x)
global Rn
global Phi0
global C
global Ic
global T
global Ic
global scale
global R1
global R2
global Rsg
global Delta
global Rshunt

v = x(1);
phi = x(2);  

if abs(v) < 2*Delta
    R = Rsg;
else
    R = Rn;
end

R = R*Rshunt/(R+Rshunt);

dvdt = (voltage(t)/(R1+R2) - v*(1/(R1+R2) + 1/R) - Ic*sin(phi))/C;

dphidt = 2*pi*v./Phi0;

dydt = [dvdt; dphidt];

voltage.m
% Function that take in a time element and returns
% the corresponding voltage in the Josephson Junction
% setup.

function V = voltage(t)
global T
A.2. Normal Resistance

normal_r.m

% Takes in junction size and temperatures
% and returns the normal resistance, subgap resistance,
% beta value, junction capacitance, and time constants.

function normal_r(T, jsize);
close all

%Inputs:
%Temperature
%Junction Size (in microns)

% Some Constants
Phi0 = 2.06E−15; % T−m^2
epsilon0 = 8.85E−12; %F/m
Delta = 1.5E−3;
Jc = 700; %A/cm^2
mu0 = 4*pi*1E−7; %H/m
lambda0 = 33E−9; %nm
me = 9.1E−31; %kg
q = 1.6E−19; %C

% Critical Current
Area = (jsize*1E−6)^2;
Ic = Jc*Area*(100)^2

%Taken from constant IcRn product
Rn = 0.68*2*Delta/Ic
%% Calculation of subgap resistance
mstar = 2*me;
qstar = 2*q;
nstar = mstar/(lambda0^2*qstar^2*mu0);
ntot = 2*nstar;
n = ntot*(T/9.2)^4;
Rsg = Rn*ntot/n

C = 10/(0.20 - 0.043*log10(Jc/1E3))*1E-15*(Area/1E-12)

%% Time Constants
Tau_J_Rn = Phi0/(2*pi*2*Delta);
Tau_J_Rsg = Phi0/(2*pi*Ic*Rsg)

Tau_RC = Rn*C

beta_Rn = Tau_RC/Tau_J_Rn
beta_Rsg = Tau_RC/Tau_J_Rsg
Appendix B

Lincoln Lab Image

Figure B-1: Josephson IV curve showing clear hysteresis. Image from Karl Berggren at MIT Lincoln Lab.
Bibliography


