RESISTANCE OSCILLATIONS IN SUPERCONDUCTING ALUMINUM NANO ARRAYS AND LOOPS

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Nothing is impossible. Not if you can imagine it. That’s what being a scientist is all about.
— Professor Hubert J. Farnsworth, *Futurama*

Dedicated to my family, without whom none of this would be possible.
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INTRODUCTION

This year marks the 105th anniversary of Heike Kameringh Onnes’ discovery of superconductivity. This surprising discovery was the result of Onnes’ work culminating in the liquefaction of helium, which gave him access to temperatures low enough to allow materials to become superconducting and earned him a Nobel Prize shortly after the discovery. There was of course much interest in this new field, yet a complete theoretical understanding of this phenomenon remained elusive for many decades. It wasn’t until the 1950’s and 1960’s that a, “complete and satisfactory theoretical picture of classical superconductors” took shape with the introduction of the phenomenological Ginzburg-Landau theory of superconductivity in 1950 and the microscopic BCS theory in 1957 [1]. Landau earned a Nobel Prize for his theory of phase transitions, which was applied to many fields, including superconductivity. The BCS theory of superconductivity also earned its authors a Nobel Prize for their discovery.

It was not until almost three decades later that the field of superconductivity was upended again by the discovery of high temperature superconductors by Bednorz and Müller. These superconductors were made from ceramics, instead of metals like the classical superconductors. They exhibited the same phenomenological behavior as their classical brethren, yet a microscopic understanding of the mechanism making them possible has to this day not been found. Despite this lack of theoretical understanding, much progress has been made in the fabrication of superconductors with ever higher transition temperatures, with one important goal being the realization of superconductors at room temperature. The highest temperature achieved to date is 203 K in a metallic compound H_2S, thus belonging to the BCS-type classical superconductors. This compound, however, only becomes metallic at 90 GPa, which is \(1 \times 10^4\) times greater than air pressure under ambient conditions [2]. More interesting then, is the record for highest transition temperature under ambient conditions, which is held by a ceramic compound and thus an unconventional superconductor, HgBa_2CuO_4+\(\delta\), at 133 K [3].

Despite not being well understood and not yet having achieved room temperature superconductors, superconductivity has already led to amazing new technologies, without which progress in other areas would not be possible. Important technologies include powerful magnets used in medical applications like magnetic resonance imaging (MRI) and functional magnetic resonance imaging (fMRI), which has revolutionized the field of brain imaging and as a result cognitive science. More examples of powerful superconducting magnets include those used in particle accelerators, like those at CERN, which helped with the discovery of the Higgs boson. The list of applications for powerful magnets made of superconductors goes on and on, as does the list of other important contributions superconductors make to technologies, like particle detectors and SQUID magnetometers.

One fascinating field that remains to be mentioned, in which superconductors will play a key role, is that of quantum computing. Superconductors offer many advantages as the building blocks of a supercomputer, one of them being the fact that the electron state in a superconductor can be described by a macroscopic wave function. Add to this the fact that superconducting rings can trap persistent currents that are so stable that the current would die out after \(10^{390}\) years and the possibilities abound [11]. As such, interest has grown in investigating the flux dynamics of small superconducting rings [4], especially into the quantum coherence effects in...
superconducting rings as they may be promising systems for realizing quantum computers [5, 7].

It is in this context that we set out on the research project described in this dissertation to study so-called double networks of superconducting aluminum. The motivation from this work came from our Israeli collaborators, who discovered magnetoresistance oscillations in double networks of high temperature superconductors with much larger amplitudes than could be explained by the Little-Parks effect. They proposed a theory based on vortex/anti-vortex dynamics to explain these enhanced amplitudes. They were motivated in part by the theoretical predictions of oscillations with a different period than that found in the Little-Parks effect.

This dissertation is structured as follows:

1. Chapter 2 explores some of the basic theoretical background behind superconductivity as is relevant to the systems we studied.
2. Chapter 3 presents research on mesoscopic superconductors conducted over the last 50 years, starting with the Little-Parks effect.
3. In chapter 4 the sample fabrication techniques, experimental measurement setup, and experimental measurement techniques are described.
4. Chapter 5 describes the results from measurements we conducted while we were still optimizing the sample design.
5. Chapter 6 presents the data from multiple measurement series performed on two especially good samples.
6. Chapter 7 summarizes the central aspects of this work.
2.1 Introduction

Superconductivity is the well-known phenomenon in which a conducting material with finite resistance suddenly looses all resistance when cooled below its critical temperature, $T_c$. Another equally important but less well-known electromagnetic characteristic of a superconductor is the expulsion of any externally applied magnetic field from its interior as it is cooled below $T_c$.

The following sections will give a short introduction into superconductivity and some specific topics in superconductivity that are relevant to the research presented later. Much of this discussion can be found in text books like [11,8,9].

2.2 The London Equations

Superconductivity was first discovered experimentally by Heike Kamerlingh Onnes in 1911 when he cooled mercury down to liquid helium temperatures, 4.2 K. In doing so he discovered that the resistance of the mercury vanished when cooled to low temperatures. It was not until the 1930s that Walther Meissner and Robert Ochsenfeld discovered a second important electromagnetic characteristic of superconductors, namely the expulsion of magnetic fields from their interior. Shortly thereafter the first theory explaining the electromagnetic behavior of superconductors was proposed by the London brothers. They proposed two equations to describe the behavior of the microscopic electric and magnetic fields in a superconductor:

\[
\vec{E} = \frac{\partial}{\partial t} \left( \Lambda \vec{j}_s \right) \quad (2.1)
\]
\[
\vec{B} = -\nabla \times \left( \Lambda \vec{j}_s \right). \quad (2.2)
\]

With the use of Maxwell’s equations one can derive a single equation for the current density inside the superconductor, $\vec{j}_s$, that relates it to the magnetic vector potential, $\vec{A}$:

\[
\vec{j}_s = -\frac{n_s e^2}{m} \vec{A} = -\frac{\vec{A}}{\Lambda}, \quad \text{where} \quad (2.3)
\]
\[
\Lambda = \frac{m}{n_s e^2}, \quad (2.4)
\]

and $n_s$ is the density of superconducting electrons, $e$ is the charge of an electron, $m$ is the electron mass, and $\Lambda$ is a phenomenological parameter. Note that equation (2.3) is not gauge invariant. By using Ampère’s law ($\nabla \times \vec{B} = \mu_0 \vec{J}$) on the second London equation, equation (2.2), we obtain

\[
\nabla^2 \vec{B} = \frac{1}{\Lambda^2} \vec{B},
\]
where \( \lambda_L \) is the London penetration depth, the penetration depth of the external magnet field into the superconductor. The London penetration depth is defined as

\[
\lambda_L = \left( \frac{m}{\mu_0 n_s e^2} \right)^{1/2}.
\]

(2.5)

The idea of the coherence length, \( \xi_{\text{Pip}} \), was later introduced by Pippard to non-locally generalize the London equation equation 2.3 \[1\]. This characteristic value is similar to the elastic mean free path \( l_e \) in the non-local electrodynamics of normal metals and represents the smallest spatial spread of a superconducting charge carrier wave packet. The coherence length can be calculated via

\[
\xi_{\text{Pip}} = a \frac{\hbar v_F}{k_B T_c},
\]

(2.6)

where \( a = 0.15 \) is a numerical constant, \( \hbar \) is the reduced Plank constant, \( v_F \) is the Fermi velocity and \( k_B \) is the Boltzmann constant. By selecting \( a = 0.18 \) for the numerical constant, this equivalent to the BCS estimate of the Cooper pair’s size, \( \xi_{\text{BCS}} \) in equation 2.58, also called the coherence length.

### 2.3 Ginzburg-Landau Theory of Superconductivity

In 1950 the Russian physicists Vitaly Lazarevich Ginzburg and Lev Landau proposed their phenomenological theory of superconductivity. Their theory stems from Landau’s general theory of second-order phase transitions and uses a complex pseudowavefunction \( \psi \) to describe the local density of the superconducting electrons

\[
n_s = |\psi(x)|^2.
\]

(2.7)

As it is a thermodynamic theory, the Ginzburg-Landau theory starts off by considering the free energy of the superconducting and normal states. Assuming that \( \psi \) is small and varies slowly in space, the free energy can be expanded in a Taylor series, of which only the first few terms are of significance near \( T_c \). For the situation where no external fields or gradients are present we have

\[
f_s - f_n = \alpha(T) |\psi|^2 + \frac{1}{2} \beta(T) |\psi|^4,
\]

(2.8)

where \( f_s \) and \( f_n \) are the free energy densities of the superconducting and normal states respectively. The parameter \( \beta \) must be positive for the theory to be useful. Thus, when \( \alpha > 0 \) the free energy has a single minimum at \( |\psi|^2 = 0 \), corresponding to the normal state. If \( \alpha < 0 \) the free energy has minima wherever \( |\psi|^2 = -\alpha / \beta \). Plugging this result back into equation 2.8 we obtain

\[
f_s - f_n = \frac{-B_c^2}{4\mu_0^2} = \frac{-\alpha^2}{2\beta},
\]

(2.9)

defining the thermodynamic critical field,

\[
B_c = \frac{\alpha \mu_0}{\sqrt{\beta}}.
\]

(2.10)
If we take fields and gradients into account and write the order parameter as \( \psi = |\psi| e^{i\phi} \) we get an additional term in equation (2.8):

\[
\frac{1}{2m^*} \left[ \hbar^2 (\nabla |\psi|)^2 + \left( \hbar \nabla \phi - e^* \vec{A} \right)^2 |\psi|^2 \right].
\] (2.11)

The first term represents the extra energy due to gradients in the magnitude of the order parameter and the second term is the gauge-invariant form of the kinetic energy of the charge carriers in a superconductor, with mass \( m^* \) and charge \( e^* \). This also gives us an effective penetration depth when we equate the kinetic energy term above with that of the superconductor associated with the London supercurrent density equation (2.3),

\[
\lambda_{eff}^2 = \frac{m^*}{4\mu_0 |\psi|^2 e^{*2}}.
\] (2.12)

Using equation (2.8) with the addition for fields and gradients, equation (2.11), we get a nonlinear Schrödinger equation for the superconducting state, the first of the Ginzburg-Landau equations:

\[
\alpha \psi + \beta |\psi|^2 \psi + \frac{1}{2m^*} \left( -i\hbar \nabla - e^* \vec{A} \right)^2 \psi = 0
\] (2.13)

and the second Ginzburg-Landau equation, which expresses the superconducting current density is

\[
\vec{j}_s = \frac{e^*}{m^*} |\psi|^2 \left( \hbar \nabla \phi - e^* \vec{A} \right) = e^* |\psi|^2 \vec{v}_s.
\] (2.14)

### 2.3.1 Characteristic Lengths

The physical meaning of equation (2.13) and equation (2.14) become clear when we examine the behavior of a superconductor at its surfaces or interfaces. In a one-dimensional system with a normal metal-superconductor (NS) phase boundary at \( x = 0 \) and the boundary condition \( \psi(0) = 0 \) the solution to equation (2.13) is

\[
\psi(x) = \psi_0 \tanh \left( \frac{x}{\sqrt{2\xi_{GL}(T)}} \right),
\] (2.15)

where \( \psi_0 \) is the bulk value of the order parameter at a large distance from the surface and \( \xi_{GL}(T) \) is a characteristic length defined as

\[
\xi_{GL}^2(T) = \frac{\hbar^2}{2m^* |\alpha(T)|} = \frac{\xi_{GL}^2(0)}{1 - t},
\] (2.16)

where \( t = T/T_c \) is the reduced temperature. This characteristic length is the Ginzburg-Landau coherence length and in our one-dimensional example is the distance from the surface over which the order parameter returns to its bulk value, \( \psi_0 \). This is different than the coherence length given in equation (2.6), however at temperatures well below \( T_c \), \( \xi_{GL}(T) \approx \xi_{Pip} \). From equation (2.16) it is clear that \( \xi_{GL}(T) \) diverges near \( T_c \).
We can get an expression for the ratio between the Ginzburg-Landau coherence length \( \xi_{GL}(T) \) and the BCS coherence length \( \xi_{BCS} \), which we will discuss in section 2.4 and we get the simplification that near \( T_c \)

\[
\xi_{GL}(T) = 0.74 \frac{\xi_{BCS}}{(1-t)^{1/2}} \quad \text{clean} \tag{2.17}
\]

\[
\xi_{GL}(T) = 0.85 \left( \frac{\xi_{BCS}}{l} \right)^{1/2} \quad \text{dirty} \tag{2.18}
\]

where the clean limit is when the coherence length \( \xi_{GL}(0) \) is much larger than the penetration depth \( \lambda_L(0) \) such that the ratio between the two, \( \kappa = \xi_{GL}(0) / \lambda_L(0) \ll 1 \), and the dirty limit is where \( \kappa \gg 1 \).

Further important results from the Ginzburg-Landau theory are the expressions for the critical magnetic fields for type-I superconductors, where \( \kappa \leq 1/\sqrt{2} \), and type-II superconductors\(^1\), where \( \kappa > 1/\sqrt{2} \):

\[
B_{c1} = \frac{\Phi_0}{4\pi\lambda_L^2(0)} \ln \left( \frac{\lambda_L(0)}{\xi_{GL}(0)} \right) \tag{2.19}
\]

\[
B_{c2} = \frac{\Phi_0}{2\pi \xi_{GL}^2(T)} \tag{2.20}
\]

where \( \Phi_0 = h/2e \) is the magnetic flux quantum and \( \lambda_L \) is the effective penetration depth of the field. The penetration depth expressed in terms of the GL theory is essentially the London penetration depth, \( \lambda_L = \sqrt{m^*/\mu_0 e^2 \psi_0^2} \).

In the case of a thin slab in a parallel field, i.e. a superconductor with its narrowest surface perpendicular to the magnetic field lines, the critical field is

\[
B_{c\parallel} = 2 \sqrt{\frac{6}{\pi d}} \frac{B c_{cL}}{d} \tag{2.21}
\]

where \( d \) is the thickness of the thin film. Using the above result with

\[
\xi_{GL}(T) = \frac{\Phi_0}{2\sqrt{2} B_{c} (T) \lambda_L (T)} \tag{2.22}
\]

gives us

\[
\xi_{GL}(T) = \frac{\sqrt{3} \Phi_0}{\pi d B_{c\parallel} (T)} \tag{2.23}
\]

\(^1\) Most of the focus will be on type-II superconductors as that best describes the superconductors used in our experiments
Thus we have the following expressions for the coherence length:

\[ \xi_{GL}(T) = 0.855 \left( \xi_{BCS} l \right)^{1/2} \]

**dirty**

(2.24)

\[ \xi_{GL}(T) = \frac{\sqrt{3}\Phi_0}{\pi dB_{c\parallel}(T)} \]

(2.25)

\[ \xi_{GL}(T) = \sqrt{\frac{\Phi_0}{2\pi B_{c2}}}. \]

(2.26)

From equation (2.24) we see that one of the methods for determining the coherence length is to determine the mean free path \( l \). This can be done through the use of empirical formula for Al films found in literature [10]:

\[ \rho l = 4.0 \times 10^{-6} \mu \Omega \text{ cm}^2. \]  

(2.27)

### 2.3.2 Fluxoid Quantization

One of the consequences of the single-valuedness requirement of the complex order parameter \( \psi(\vec{r}) = |\psi|e^{i\phi(\vec{r})} \) in a superconductor is that the phase factor only changes in multiples of \( 2\pi \) along any ring-like path in a superconductor. In his analysis of superconductors, F. London introduced the fluxoid \( \Phi' \) to capture this idea:

\[ \Phi' = \Phi + \Lambda \oint \vec{j} \cdot d\vec{s}, \]

(2.28)

where \( \Phi = \oint \vec{A} \cdot d\vec{s} \) is the ordinary flux and \( \vec{v}_s = \frac{1}{2m} \left( \hbar \nabla \phi - 2e\vec{A} \right) \) is the gauge invariant supercurrent velocity. By using Bohr-Sommerfeld quantization we get

\[ \Phi = \frac{1}{2e} \oint \left( 2m\vec{v}_s + 2e\vec{A} \right) \cdot d\vec{s} = \frac{c}{2e} \oint \vec{p} \cdot d\vec{s} \]

\[ = n \frac{\hbar}{2e} = n\Phi_0 \]

(2.29)

which tells us that the magnetic flux through a hole in a superconductor is quantized. This was demonstrated in thick-walled cylindrical superconducting samples by two teams simultaneously [11][12]. This was taken as proof that the superconducting charge carrier is indeed a Cooper pair, as predicted by BCS theory and described in section 2.4. Further experiments with thin-walled superconducting samples were carried out later by Little and Parks, in which they discovered the effect named after them [13]. This topic will be discussed in more detail in section 3.1

### 2.3.3 Resistance in 1D Superconducting Wires

As opposed to bulk superconductors, the transition in thin superconducting wires is much wider, owing in part to fluctuations that disrupt the phase coherence along the wire. Events during which the phase along the wire is momentarily interrupted are called "phase slips". Phase slips can arise from either thermal or quantum fluctuations along the wire.
The theory describing thermally activated phase slips was first developed by Langer and Ambegoaker [15] and then improved upon by McCumber and Halperin [16]. We can visualize this process by plotting the order parameter

\[ \psi(x) = |\psi(x)| e^{i\phi(x)} \]  

(2.30)

of a one-dimensional wire in a polar plane perpendicular, as shown in figure 2.1a. A voltage drop across the wire causes the phase difference across the wire to increase at the Josephson rate (see section 2.5 for a description of the Josephson effect)

\[ \frac{d\phi}{dt} = \frac{2eV}{h} \]  

(2.31)

which leads to an increasing current that will eventually exceed the critical current of the wire. This can be visualized as a cranking of one end of the order parameter around the polar plot while one end is fixed and the helix is tightened as shown in figure 2.1b. The constraint on this behavior follows from equation (2.30):

\[ I \propto \psi(x)^2 \frac{d\phi}{dx} \]  

(2.32)

If fluctuations cause a local reduction in the order parameter \(|\psi|\), then \(d\phi/dx\) becomes large in this region. As the order parameter approaches zero, \(|\psi| \rightarrow 0\), it becomes easy to add or
2.4 Microscopic Theory of Superconductivity

In 1957, Bardeen, Schrieffer and Cooper published their microscopic theory of superconductivity, which was named BCS theory after them [20]. It built on previous work of, among others, Cooper, who posited the idea that electrons in a superconductor combine into pairs, so-called Cooper pairs, and that these are the fundamental charge carriers in superconductors. Shortly after the publication of their theory a Russian scientist, Lev P. Gor'kov, used an approach based on Green’s function to show that Ginzburg-Landau is a special case of BCS theory applicable close to \( T_c \) [21].

The basic element of BCS theory, the Cooper pair, is possible because, as Cooper showed, that even a weak attractive force can cause an instability in the Fermi sea, which leads to at
least one bound pair. One writes the wave function for two electrons with equal and opposite momenta interacting with one another at $T = 0$ as:

$$\psi_0 (\vec{r}_1, \vec{r}_2) = \sum_{\vec{k}} g_{\vec{k}} e^{i\vec{k} \cdot \vec{r}_1} e^{-i\vec{k} \cdot \vec{r}_2},$$

where $g_{\vec{k}}$ is a weighting term. With the two spins from the electrons, the Cooper pair can form with, be singlet or triplet coupling, but with an attractive interaction the singlet coupling has lower energy. This singlet wave function is

$$\psi_0 (\vec{r}_1 - \vec{r}_2) = \left[ \sum_{\vec{k} \neq \vec{k}'} g_{\vec{k}} \cos \vec{k} \cdot (\vec{r}_1 - \vec{r}_2) \right] (|\uparrow\downarrow\rangle - |\downarrow\uparrow\rangle).$$

We can determine $g_{\vec{k}}$ and the energy eigenvalue $E$ by solving

$$(E - 2\epsilon_{\vec{k}}) g_{\vec{k}} = \sum_{\vec{k} \neq \vec{k}'} V_{\vec{k}\vec{k}'} g_{\vec{k}'}.$$

In this expression $\epsilon_{\vec{k}}$ are the unperturbed plane-wave energies and $V_{\vec{k}\vec{k}'}$ the matrix elements of the interaction potential. The BCS approximation simplifies this problem by taking $V_{\vec{k}\vec{k}'} = -V$ for $|E - E_F| < \hbar \omega_D$, with the Debye frequency $\omega_D$, and $V_{\vec{k}\vec{k}'} = 0$ everywhere else. Through substitution, cancellation, and replacing a sum with an integral in equation (2.41), we obtain

$$\frac{1}{N(0)V} = \int_{E_F}^{E_F + \hbar \omega_D} \frac{de}{2e - E} = \frac{1}{2} \ln \frac{2E_F - E + \hbar \omega_D}{2E_F - E},$$

with the density of states at the Fermi energy $N(0)$. Because for most classical superconductors $N(0)V < 0.3$, we can use the weak-coupling approximation, valid for $N(0)V \ll 1$, and obtain for equation (2.42)

$$E \approx 2E_F - 2\hbar \omega_D e^{-2/N(0)V}.$$
If we fix the number of particles to $N$, we obtain a large uncertainty in the phase factor $\varphi$ and vice versa, giving us the uncertainty relation

$$\Delta N \Delta \varphi \gtrsim 1. \quad (2.46)$$

In order to calculate the superconducting energy gap at zero temperature, one uses the variational method on the pairing Hamiltonian

$$H = \sum_{kk'} \epsilon_k n_{kk'} + \sum_{kk'} V_{kl} c_{kk'}^e c_{kk'}^\ast - \sum_{kk'} V_{kl} c_{kk'}^\ast c_{kk'}^e. \quad (2.47)$$

The mean number of particles $N$ is regulated by including the term $-\mu N_{\text{op}}$ where $\mu$ is the chemical potential and $N_{\text{op}}$ is the particle-number operator. Using this with equation (2.44) on equation (2.47) we get

$$\langle \psi_G | H - \mu N_{\text{op}} | \psi_G \rangle = 2 \sum_k \xi_k v_k^2 + \sum_{kk'} V_{kl} v_k u_l p_{kl}, \quad (2.48)$$

where $\xi_k = \epsilon_k - \mu$ is the single-particle energy relative to the Fermi energy. Imposing the constraint $|u_k|^2 + |v_k|^2 = 1$ by setting $u_k = \sin \theta_k$ and $v_k = \cos \theta_k$ and considering that taking $\partial \partial \theta_k$ of equation (2.48) must be zero, and some trigonometric identities we have

$$\tan 2\theta_k = \frac{\sum_l V_{kl} \sin \theta_l}{2 \xi_k}. \quad (2.49)$$

We can now define two quantities, first

$$\Delta_k = - \sum_l V_{kl} u_{l} c_{kl}^\ast = -\frac{1}{2} \sum_l V_{kl} \sin 2\theta_l, \quad (2.50)$$

which is basically independent of $\vec{k}$, and thus the minimum excitation energy, or the superconducting gap $\Delta$. The second quantity we can define is

$$E_k = \left( \Delta_k^2 + \xi_k^2 \right)^{1/2}, \quad (2.51)$$

which represents the excitation of the quasi-particle with a momentum of $\hbar \vec{k}$. Using equation (2.50), the definition of $\Delta_k$, the BCS approximation again and replacing the summation with an integral we get the expression

$$\frac{1}{N(0)V} = \int_0^{\hbar \omega_D} \frac{d^2 \vec{\xi}}{(\Delta + \xi_k^2)^{1/2}} = \sinh^{-1} \frac{\hbar \omega_D}{\Delta} \quad (2.52)$$

and thus for the energy gap $\Delta$

$$\Delta \approx -2 \hbar \omega_D e^{-1/N(0)V}, \quad (2.53)$$

in the weak coupling limit.

Because the quasi-particles are fermions, they obey Fermi statistics at finite temperatures with

$$f(E_k) = \left( e^{\beta E_k} + 1 \right)^{-1} \quad (2.54)$$
where $\beta = 1/k_B T$. This results in

$$\Delta_k = -\sum_l V_{kl} E_l \tanh \frac{\beta E_l}{2} \tag{2.55}$$

and with the BCS approximation and solving for $\Delta(T) \to 0$, we eventually obtain an expression for $T_c$

$$k_B T_c = 1.14 \hbar \omega_D e^{-1/N(0)V}, \tag{2.56}$$

and comparing with equation (2.53) we get the BCS result for the superconducting energy gap:

$$\Delta(0) = 1.764 k_B T_c. \tag{2.57}$$

From this we can define the BCS coherence length, which is a measure for the spatial extent of the Cooper Pair

$$\xi_{BCS} = \frac{\hbar v_f}{\pi \Delta(0)}. \tag{2.58}$$

### 2.5 The Josephson Effect

The Josephson Effect only plays a small role in the systems studied in this work. For this reason it will only be briefly introduced here.

The Josephson effect arises when a weak link separates two superconductors. A weak link can be an insulator, a normal metal, or even a narrow constriction in the superconductor and is called a Josephson contact. The effect of this Josephson contact is to give rise to a finite current without a driving voltage:

$$I_s = I_c \sin \Delta \varphi, \tag{2.59}$$

where $\Delta \varphi$ is the difference in the phase of the wave function of the two different superconductors and $I_c$ is the critical current of the junction.

Furthermore, if a voltage were maintained across the junction, the phase difference $\Delta \varphi$ would oscillate in time according to

$$\frac{d\Delta \varphi}{dt} = \frac{2e}{\hbar} V, \tag{2.60}$$

leading to a total superconducting current

$$I = I_c \sin (\Delta \varphi + \omega_j t), \tag{2.61}$$

with $\omega_j = \frac{2\pi}{\Phi_0} V$.

For real Josephson junctions a more complete description is required. First, we introduce the gauge-invariant phase difference between the two superconductors in a Josephson junction

$$\gamma = \Delta \varphi - \frac{2\pi}{\Phi_0} \int \vec{A} \cdot d\vec{s} \tag{2.62}$$
that will give us truly unique solutions for different physical situations. Second we introduce
the resistively and capacitively shunted junction model (RCSJ model), in which dissipative processes
are approximated using a resistance $R$ and capacitance $C$ both connected in parallel with the
actual junction. This leads to a total current through the three parallel paths of

$$I = I_{c0} \sin \gamma + V/R + C dV/dt,$$  \hspace{1cm} (2.63)

where $I_{c0}$ is simply a coefficient of $\sin \gamma$ that is related to, but can be less than, the observable
critical current of the junction, $I_c$. We can restate equation (2.63) as a second-order differential
equation by replacing $V$ with the equivalent $\gamma$ and the introduction of a dimensionless time
variable $\tau = \omega_p t$, where, $\omega_p$ is the plasma frequency

$$\omega_p = \left( \frac{2eI_{c0}}{\bar{h}C} \right)^{1/2}$$ \hspace{1cm} (2.64)

and a quality factor $Q = \omega_p RC$. The result is

$$\frac{I}{I_{c0}} = \sin \gamma + Q^{-1} \frac{d^2 \gamma}{d\tau^2} + \frac{d\gamma}{d\tau}.$$ \hspace{1cm} (2.65)

We can interpret equation (2.65) as an equation of motion of a particle of mass $(\hbar/2e)^2 C$
moving along the $\gamma$-axis in an effective potential

$$U(\gamma) = -E_J \cos \gamma - \left( \frac{\hbar I}{2e} \right) \gamma,$$ \hspace{1cm} (2.66)
14 THEORETICAL BACKGROUND

Figure 2.3: The experiment proposed by Aharonov and Bohm to test for their theorized effect [26]. An electron beam is directed towards a cylindrical solenoid producing a magnetic field in its core. The electron beam is split at A and each beam traverses a path on either side of the solenoid. The split beams are brought back together and allowed to interfere on a screen located at F.

where $E_J = (h/2e) I_{c_0}$ is the Josephson coupling energy, and the equivalent of viscous drag would be $(h/2e)^2 (1/R) d\gamma/dt$. This is the so-called “tilted washboard model” and is shown in figure 2.2. An externally applied current determines the slope of the entire washboard. When $I = I_{c_0}$ the minima of the cos become horizontal plateaus. For even higher currents, $I \gtrsim I_{c_0}$, there are no longer any minima and the phase increases continuously and leads to a finite junction voltage.

2.6 THE AHARONOV BOHM EFFECT

We will later be introduced to the Little-Parks effect, one of the possible sources of oscillations one can see in superconducting loops. Another possible source of oscillations is the from the Aharonov-Bohm effect, which was first put forth in 1959 by Yakir Aharonov and David Bohm [26]. The effect describes how charged particles can be affected by electromagnetic potentials in regions of space in which electromagnetic fields vanish. In their paper they even proposed an experiment to show this effect, where an electron beam is split and the two resulting beams follow two different paths around a solenoid and are brought back together on the opposite side. It is here that they are allowed to interfere, as shown in figure 2.3. In the classical picture of electrodynamics, whether a magnetic field in the solenoid is turned on or turned off should have no effect on the electrons. However, as shown by Chambers in an experiment set up almost exactly as Aharonov and Bohm proposed, a phase difference was found between the two different paths when a magnetic field was present inside the solenoid [27]. The phase difference was confirmed to be

$$\Delta \Phi = \frac{e}{\hbar} \oint A ds = \frac{e}{\hbar} \Phi = \frac{\Phi}{\Phi_1}$$

(2.67)

where the flux quantization is $\Phi_1 = \frac{h}{e} = 2\Phi_0$.

The experiment of Chambers was performed with electron beams traveling through air. Many assumed that the situation would be different if the experiment were performed using metal pathways instead due to scattering [28]. However, for electrons propagating through a metal, phase coherence is preserved after elastic scattering events but not inelastic scattering events. At low temperatures the inelastic mean free path in metals is orders of magnitude
larger than the elastic mean free path \( l_{\text{el}} \approx 1 \times 10^{-4} \text{ nm} \), \( l_{\text{in}} \approx 1 \times 10^{1} \text{ nm} \) [28]) and with modern fabrication techniques it is possible to create metallic rings with diameters in the size range between these two lengths. Such small metallic systems were measured by Webb et al. and the expected Aharonov-Bohm interference patterns were indeed detected [29].

In addition to the \( h/e \) oscillations they also detected \( h/2e \) oscillations. The \( h/2e \) oscillations in a normal metal (at low temperatures) come from the Al’tshuler-Aronov-Spivak effect which was theorized in 1981 [30]. Shortly thereafter this effect was observed in Magnesium films at helium temperatures [31]. In this effect the two electronic waves traverse the entire circumference of the loop, instead of just half way, and interfere back at the point where they originally split up. The enclosed flux and thus phase difference between the two waves is twice that seen in the AB effect, \( 2\Phi_1 \), which leads to oscillations with half the period of the AB effect, i.e. \( h/2e \).

As the rings get even smaller, such that the circumference is smaller than the \( \xi_{\text{GL}}(0) \), the picture gets even more complicated. This will be dealt with in chapter 3.
MEASUREMENTS AND PREDICTIONS ON NANOSTRUCTURED SUPERCONDUCTORS

The following section outlines the progression of research on small superconducting structures, starting with the Little-Parks (LP) effect. In the years following the discovery of Little and Parks, much research was done on superconductors with decreasing dimensions, approaching the two-dimensional and one-dimensional limits. This research, both experimental and theoretical, found that the behavior of low dimensional superconductors in general, as well as mesoscopic superconducting loops, exhibited anomalous effects. These effects manifest themselves in three different ways. It was found that mesoscopic superconductors could exhibit anomalous resistances in the form of an enhanced resistance near the NS phase boundary exceeding the normal state resistance $R_N$. Related to this, it was found in mesoscopic loops that the amplitude of the LP-like oscillations exceeded the amplitude of what the LP effect predicted. Finally, the oscillation period of mesoscopic loops was found to differ from the LP oscillation period of integral values of the magnetic flux quanta $n\Phi_0$. Quite a number of theoretical papers predicted that oscillations with twice the period of the LP oscillations, namely proportional to $\Phi_1 = 2\Phi_0 = h/e$, should be evident under certain conditions.

The structure of this section is as follows: first the LP effect is described. Then the research into the anomalous resistances first discovered in experiment is presented. The discussion then turns to the various types of anomalous oscillations observed in various experiments as well as predicted by theory. Finally research into "double" networks of mesoscopic loops is presented.

3.1 THE LITTLE-PARKS EFFECT AND MESOSCOPIC LOOPS

Shortly after the experimental confirmation of flux quantization by Deaver and Fairbank and Doll and Nabauer, Little and Parks investigated the effect of mesoscopic superconducting cylinders placed in axial magnetic fields [13, 32–34]. They placed a thin-walled cylinder of thickness $d$ and radius $r$ in a magnetic field parallel to the axis of the cylinder. They measured the resistance as the magnetic field changed and observed oscillations, as shown in figure 3.1a. From the slope of the $R$-versus-$T$ curve they calculated the variation in the transition temperature using

$$\Delta R \approx \frac{dR}{dT}\Delta T_c,$$

as shown in figure 3.1b. An important criterion for the construction of $B_c$-versus-$T$ phase diagrams from isothermal $R$-versus-$B$ measurements is that the width of the resistive transition in a magnetic field be smaller than the periodicity unit:

$$B(R = R_N, T) - B(R = 0, T) \ll \frac{\Phi_0}{\pi r^2}.$$  

This criteria was not satisfied in the original work from Little and Parks but later measurements by Groff and Parks did meet this requirement. From their data they were able to construct the NS phase boundary for a 1.33 µm-diameter aluminum sample, as shown in figure 3.2.
Figure 3.1: a) The lower trace shows the oscillations in the resistance of a tin cylinder at its superconducting transition temperature $T_c$ as a function of the applied magnetic field, shown in the upper trace [13].
b) A schematic depiction of how small changes in $T_c$ can lead to changes in the resistance $R$ of a sample [13].

In their measurements we clearly see a series of parabolas superimposed on a quadratic background. Tinkham [35] developed the theory describing the variation of the transition temperature with the applied external magnetic field in thin-film superconductors, which resulted in

$$\frac{\Delta T_c}{T_c} = \frac{r^2}{8\lambda_e^2(0) B^2_{cB}(0)} \left[ \left( \frac{B - n\Phi_0}{\pi r^2} \right)^2 + \frac{1}{3} \frac{d^2}{r^2} B^2 \right], \quad (3.3)$$

where $\lambda_e(0)$ and $B_{cB}(0)$ are the penetration depth of the film and the bulk critical field at $T = 0$ K. The first term in equation (3.3) describes the periodic cusp-like oscillations seen in the measurements and the second term describes the monotonic background upon which these oscillations are superimposed. Further equations with higher-order terms were also developed, but the precision of the experiments so far have not been able to detect the influence of these [36].
Figure 3.2: A representative phase diagram of a 1.33 µm-diameter aluminum cylinder, extracted along isothermals of $R$-versus-$B$ measurements. The solid curve is a plot of equation (3.3) and the dashed curve is a fit of the non-periodic part of equation (3.3) through the local temperature maxima.
3.2 ANOMALOUS RESISTANCE

Research into ever smaller superconductors continued over the years, but it was not until the late 1980’s and early 1990’s that fabrication techniques advanced enough that researchers could easily manufacture superconducting samples at the nanometer scale. It was in early 1990’s when Santhanam et al. investigated short superconducting wires and found a surprising increase in the resistance above the normal state resistance \( R_N \) just above the transition temperature \( T_c \) \[37\]. They speculated that the origin of this anomalous peak was a quasi-particle charge imbalance around NS phase boundaries.

Shortly thereafter another group investigated how the distance of the probe leads from the NS phase boundary surface effected the height of the anomalous resistance peak in Al thin films \[38\]. In their samples, the central region of the broad Al strips was etched, changing the \( T_c \) of the Al in this area from the rest of the Al sample by about 45 mK, in effect pinning the location of the NS phase boundary when the sample temperature was between the two different \( T_c \)s of the different regions. They placed pairs of probe leads symmetrically around this etched area at increasing distances, as shown in figure 3.3, and compared measurements taken with different lead pairs. They found that the size of the anomaly decreased with greater distance from the NS phase boundary and that it was also reduced by a magnetic field, in full agreement with the findings of Santhanam et al. They argued that this result agrees with the model that quasi-particle injection from the N region into the S region leads to the superconducting potential exceeding \( IR_N \) near the NS interface in the presence of the quasi-particles.

Furthermore, a loop sample geometry was investigated by Vloeberghs et al. Moshchalkov et al. and again resistance enhancements were also found under various circumstances \[39\], some of which we will return to later. As demonstrated by the data represented by black squares in figure 3.4, the resistance exceeds \( R_N \) when the sample is just above \( T_c \). Although their initial research was originally designed to investigate anomalies in the magnetoresistance (MR) oscillations, they dedicated later work to investigation of the temperature-dependent resistance anomaly \[41\]. Their investigations lead them to the conclusion that the resistance anomaly was caused by intrinsic phase slips, as per the Langer-Amegaokar-McCumber-Halperin (LAMH) model for thermal fluctuations in superconductors \[41\]. They had to modify the original theory so it would apply to mesoscopic samples, for instance they

![Figure 3.3: A schematic diagram of the sample measured by Kwong et al. showing the placement of the leads relative to the etched region](image-url)
assumed that normal and superconducting currents cannot coexist in the confined geometry of mesoscopic samples. They came up with a modified formula to describe the resistance,

$$ R = \frac{R_N (\tau_N / \tau_S)}{(\tau_N / \tau_S) + 1} + R_s (\tau_N / \tau_S) + 1 $$

with the resistance from phase slip events, $R_s$, the normal state resistance $R_N$ and the characteristic switching times between the superconducting $\tau_S$ and normal $\tau_N$ states. Figure 3.5 shows that fits with equation (3.4) agree very well with their data. A response to this study was published by Landau and Rinderer in which they argued against the model proposed by Moshchalkov et al. and conclude that the resistance anomalies occur because of the extra resistance of the NS boundary [42]. Their main concern is that the assumption made by Moshchalkov et al.—namely that the normal and superconducting currents cannot coexist—is in direct contradiction with other theoretical studies on phase-slip centers. To back up their claim that this resistance anomaly is not due to sample geometry, they cite more recent studies of wider aluminum strips that also found a resistance anomaly, namely from Kwong et al. and Park et al. [38, 43].

Strunk et al. did further investigations of the resistance anomaly in similar thin aluminum structures [44]. They found that by placing rf-filters in the measuring lines they were able to eliminate the anomaly where the resistance of the sample exceeds the normal resistance at temperatures just above $T_c$. They were then able to reproduce the resistance anomaly by coupling a well-defined rf-signal back into their shielded system. They speculate that the cause of this resistance anomaly is the generation of local charge imbalance round a phase-slip center (PSC), which is created by the rf signal.

Arutyunov et al. conducted yet a further study on the resistance anomaly in mesoscopic superconductors [45]. They propose a model based on the comments of Landau and Rinderer [42], which, contrary to a previous explanation of phase slippages, starts with the important assumption that mesoscopic lift-off samples can in no way be considered homogeneous. They speculate that the cause of the resistance anomaly is the geometric effect of tilted NS boundaries and has to do with the electric-field generated by non-equilibrium quasi-particle injection into the superconductor from the normal region. A tilted NS boundary can be understood as a phase boundary that is not strictly perpendicular to the wire axis. This can have two different causes. First, the cross section of a "wire" is not perfectly square but instead resembles a Gaus-
Figure 3.5: (upper) $R$-versus-$T$ trace with a bias current of $I = 0.03 \mu A$. The solid line shows the fit of $R$ to equation (3.4) using parameters $\tau_n/\tau_0 = 6500$ and $\delta = 0.0565$. (lower) Same as in (upper) but with a bias current of $I = 0.10 \mu A$.

Gaussian form with a flattened top. This means that, starting at the middle and moving towards the edge of the wire, the local $T_c$ of the sample increases. Second, for many mesoscopic samples fabricated using the lift-off technique as is done here, the probe width is similar to the wire width, meaning that the nodes where these two join can no longer be simply considered small perturbations to the sample’s local electrical landscape. The NS boundary near the nodes is then deformed in unpredictable ways. They conclude that because this high-resistive-state varies from sample to sample, it can be seen as a fingerprint of the imperfections in the specific region of the sample under consideration.
In addition to the anomalous resistance peaks discussed in the previous section, further anomalous behavior was found in the magnetic field behavior of mesoscopic superconductors, and, as theoretical models became more sophisticated, predictions of novel effects of mesoscopic superconductors began to abound.

The first paper to predict oscillations with a different period than the magnetic flux quantum, was published over a decade after the discovery of the LP effect. In it, Bogachek et al. predicted a $h/e$-periodicity, i.e. double that of the LP effect ($= 2\Phi_0$). Bogachek et al. stated that the theory describing the LP oscillations based on Ginzburg-Landau theory and developed by Tinkham ignored quantized single-particle excitations, representing another current carrying state in the superconductor. In small dimensions this quantization is crucial to describing the behavior of the system. By adding quantum corrections back into the calculation, one finds a doubling of the period of the $T_c$ oscillations due to the existence of magnetic surface levels. The amplitude of this oscillation is however much smaller than the $h/2e$ oscillations.

Another decade later and the first experimental indication of oscillations with a period other than $\Phi_0$ were found using a lattice network-type sample. Pannetier et al. studied a regular square network of aluminum and found, in addition to the LP effect, oscillations from fractional numbers of the flux quanta. This fractional quantization arises from frustration in the network rearranging the vortices and causing certain parts of the network to return to the normal state so that it can accommodate the flux continuously. In other words it had a major structure at $B = n\Phi_0/A$, where $A$ is the average area of the network’s squares, and a fine structure at $B = n/m\Phi_0$, where $n, m$ are integers.

Sample fabrication techniques improved and researchers came closer to being able to fulfill the requirement that $\xi_{GL}(T) \approx r$, which was the predicted upper limit for seeing interference effects. Along this line of research, Moshchalkov et al. reported finding LP oscillations in mesoscopic superconducting loops whose amplitudes showed anomalous magnetic field and current dependence for small magnetic field strengths. They investigated a simple loop, as shown in figure 3.4. When looking at the MR traces to see the effect of the temperature and the measurement current on the oscillations, one sees that exotic oscillations appear for values of the reduced flux $\Phi/\Phi_0 \leq 2$ and high currents $I \geq 0.1 \mu A$, which then return to the LP effect at higher fields and elevated temperatures above $T_c$. We get a clear picture of this effect when looking at a phase diagram and compare for different measurement currents. Figure 3.7 shows the measurements of Moshchalkov et al. on the same system that Vloeberghs et al. investigated. It is clear from the data that as the measurement current increases, the oscillations in low fields ($\Phi/\Phi_0 \leq 2$) quickly deviate from the normal LP oscillations and this deviation grows, with a maximum oscillation amplitude at about $I = 1 \mu A$, until the critical current, $I_{cr}$, is reached. This is not the superconducting critical current $I_c$. Beyond $I_{cr}$ all magnetic field-dependent oscillations of the transition temperature are suppressed. Moshchalkov et al. speculate that these are related to the anomalous field enhancement of $T_c$ as well as the anomalous resistance bump above $T_c$, as shown in figure 3.4. Specifically, it is the interaction between the superconducting length scales, $\xi_{GL}(T)$, sample dimensions $2\pi r$ and the de Broglie wavelength, $\lambda_D$, which is determined by the transport current, that give rise to the shift from a regime where the phase boundary $T_c$ exhibits LP oscillations to one where the oscillations are suppressed.
Soon thereafter, Moshchalkov et al. investigated the effect of different sample geometries on the critical fields of superconductors. They measured the $B$-$T$ phase boundary of superconducting aluminum wires, loops and squares to illustrate the effect that sample geometry had on the nucleation of superconductivity in mesoscopic samples. They use a modified version of the Tinkham formula, equation (3.3):

$$
\frac{T_c (B) - T_c (B = 0)}{T_c (B = 0)} = - \left( \frac{\pi \xi_{GL} (0) w B}{\sqrt{3} \Phi_0} \right)^2 - \frac{\xi_{GL} (0)}{R_1 R_2} \left( n - \frac{\pi B R_1 R_2}{\Phi_0} \right) \tag{3.5}
$$

where $R_1$, $R_2$ are the inner and outer radius of the ring, respectively. The first term in equation (3.5) gives the behavior of a thin slab with a thickness $w$ in a parallel field, but can also be used for a narrow line in a perpendicular field as the area exposed to the magnetic field has the same geometry. The second term describes the oscillatory behavior of $T_c (B)$ with the change of the winding number $n$. They found that the behavior of a line agreed very well with the first term of equation (3.5), i.e. it had a strictly quadratic phase boundary. They also found that the behavior of a square loop agreed very well with the entirety of equation (3.5), i.e. it had oscillatory behavior superimposed upon the quadratic background of a line.

Zhilyaev et al. also found anomalous resistance oscillations in superconducting loops similar to those studied by Vloeberghs, Moshchalkov and Strunk. They found inverted LP oscillations at low field strengths, which then returned to the expected LP oscillations at higher fields, before entering the normal conductance regime. This effect was also dependent on the current.
At low current levels the behavior was anomalous, with the LP behavior returning at currents higher than 4 µA. They proposed that the leads and the loops generally find themselves at different stages of the superconducting transition. A NS boundary forms and quasi-particles penetrate from the normal region into the superconducting region. These particles penetrate into the superconductor to a depth of $\lambda_Q$, which is the charge imbalance length, the length needed to convert quasi-particles into Cooper pairs. Finally, to test their hypothesis they replicated the same experiment on samples where the lines leading to the loop are much wider such that the leads are superconducting while the loop is in the normal regime. With this reversal of states, the oscillations they observed in the smaller line samples were not present.

Strunk et al. performed further experiments on superconducting aluminum loops, this time looking to see the effect the loop had on the leads near the loop and vice versa. An AFM image, shown in figure 3.8, shows the arrangement of the four different voltage leads used to measure the voltage across different sections of the sample. Their findings show that the leads and the loop are invariably coupled, as indicated by the comparison of the MR of the loop and a section of lead directly next to it, as shown in figure 3.9. To get a better idea of the nature of this coupling, the $B$-$T$ phase boundary for both a loop and a neighboring lead were measured (see figure 3.10a). To fit for the behavior of only a line in a perpendicular magnetic field they used the thin wire in a perpendicular field contribution to Tinkham’s equation equation (3.3):

$$T_c (B) = T_{c0} \left[ 1 - \frac{\pi^2}{3} \left( \frac{d\xi_{GL}(0) B}{\Phi_0} \right)^2 \right],$$

(3.6)
and to fit for the behavior of the loop they used a modified version the full equation equation (3.3):

\[ T_c(B) = T_{c0} \left\{ 1 - \left( \frac{\xi_{GL}(0)}{R_m} \right)^2 \left[ \frac{\pi R_m^2 B}{\Phi_0} \right]^2 \left( 1 + z^2 \right) - 2n \frac{\pi R_m B}{\Phi_0} + \frac{n^2}{2z} \ln \left( \frac{1 + z}{1 - z} \right) \right\} \]

where \( R_m \) is the average of the inner and outer radii of the loop, \( d = R_{\text{max}} - R_{\text{min}} \) is the line width, which was uniform for both the line and the loop, and \( z = d/2R_m \) is the aspect ratio of the loop. The results of their measurements and the associated fits are shown in figure 3.10a. What they find is in agreement with the non-local effect exhibited by their previous independent measurements of the loop and neighboring leads in figure 3.9, namely that the oscillatory behavior associated with a loop in a magnetic field is also seen in the neighboring lead. In fact, they test for increasing loop-lead distance and find that the oscillations persist even up to a distance of 2.0 \( \mu \)m. They also see low-field oscillation anomalies, as in [49], however they attribute these to residual rf radiation reaching the sample. In summary they see oscillations in the lead where one would expect none and see a dampening of the oscillations in the loop. They conclude that there is indeed coupling between the leads and the loop with different transition temperatures between the leads and the loop, however much smaller than would be expected from the LP effect for an isolated loop.

Building on the work of Moshchalkov, Vloeberghs and Strunk, Bruyndoncx et al. studied fluxoid quantization in multiloop structures [51]. Using similar sample fabrication techniques, they created samples with more than one superconducting loop, as shown in figure 3.11. The
bola, named after a type of throwing weapon, consists of two superconducting loops in series, separated by a short length of superconducting wire. The double-loop is two loops connected in series, sharing a common strand, and the triple-loop is three loops connected in series with two shared common strands. Their investigation of the NS phase boundaries of these loops showed that the bola behaved as a single loop and the two connected structures showed additional features in the phase boundary, similar to large networks.

A summary of their measurements is shown in figure 3.10b. Because the samples were constructed with loops all of the same size, the underlying LP oscillations have the same period for all loops.

They calculated the NS phase boundary for each case using the linearized one-dimensional Ginzburg-Landau equation with the boundary conditions imposed by each sample’s geometry. In the London Limit (LL), i.e. under the assumption that the modulus of the order parameter is spatially constant, a parabolic function for the phase boundary \( T(\Phi) \) is obtained for each unique distribution of fluxoids throughout the loops \( \{n_i\} \). For comparison they also used the de Gennes-Alexander (dGA) approach, which allows \( |\Psi(x)| \) to vary spatially. The results of all calculations are shown in figure 3.12 along with the experimental data. The experimental data are represented by the dotted lines, the dashed lines are the results of the calculation in the LL, the solid line the results using the dGA approach and the dashed-dotted line is the dGA approach with a correction that also takes the leads attached to the loop structures into account. The inclusion of the leads in the dGA approach gives the best agreement with the experiment and the \( \xi_{\text{GL}}(0) \) obtained from that calculation agrees within a few percent with the \( \xi_{\text{GL}}(0) \) found from fitting the monotonic background with equation (3.6).
Figure 3.10: a) Directly measured $B$-$T$ phase boundary for a loop (filled circles) and its neighboring lead (open triangles). The inset shows measurements for samples with increasing loop-lead distances [50]. b) NS phase boundaries for the bola, double-loop and triple-loop. All structures exhibit the typical LP oscillations, with additional features superimposed on this in the case of the double and triple-loops. The curves have been shifted along the x-axis for clarity [51].

The additional minima in the phase boundary for the double and triple-loops occurs at points $\Phi = f\Phi_0$ where they obtained $f \approx 0.36$ for the double loop and $f \approx 0.30$ for the triple-loop. This originates from the transition to different quantum states and is different than the maxima found at rational values $1/2, 1/3$, etc of $f$ by Pannetier et al.

The different behavior of the ladders, i.e. the double loop or triple loop, has to do with the presence of nodes, one-dimensional analogs of Abrikosov vortices, in the shared strand between two loops. The double-loop obtains a node at the crossover point $f$, blocking a supercurrent from flowing through the strand, effectively making the double loop act like single loop. In the triple-loop, while there is considerable modulation of $|\Psi|$, no nodes are created and the minima in the phase boundary arise from reversals of the supercurrents in the individual loops.

Figure 3.11: AFM micrographs showing sample geometries: (a) the bola, (b) the double-loop and (c) the triple-loop [51].
3.3 ANOMALOUS OSCILLATIONS IN MESOSCOPIC LOOPS

Research on increasingly complex geometries was undertaken by Behrooz et al. when they studied superconducting wire networks ranging from periodically patterned networks, through quasi-crystalline and incommensurate configurations to randomly patterned networks [52]. In accordance with the findings of Pannetier et al., they find that a periodic network results in a major periodic structure, corresponding to integral flux, with a fine structure superimposed onto this, corresponding to a rational flux. Furthermore, quasi-periodic networks also exhibit a regular depression of $T_c$, albeit in a quasi-periodic manner. Finally, random networks showed no commensurate signal in the $T_c$-versus-$B$ measurements.

3.3.1 Predictions of $h/e$ Oscillation Periods

The long standing goal of many of these studies was to reduce sample size sufficiently to see interference effects, predicted to be the case when $\xi_{GL}(0)$ was comparable to the size of a superconducting ring. While experimental research continued to work towards this goal, theoretical researchers built on the works of Bogachek et al. and other from the 1960’s and 1970’s to further describe the conditions under which one would expect to see, for instance, $h/e$ oscillations.

Loder et al. showed that superconducting loops of, among others, s-wave superconductors with small energy gaps have $h/e$-periodic supercurrents [53]. Their argument is based on the fact that the $h/2e$ periodicity arises not just from Cooper pairing of the charge carrying electrons, but also from the requirement that the energy be degenerate in two different classes of supercurrent-carrying states [46, 54, 55]. One class has energy minima at odd multiples of $\Phi_0$ and the other at even multiples. This degeneracy is lifted in discrete systems.
In $s$-wave superconductors with an inner diameter of $d \ll \xi_{BCS}$, the $h/2e$ periodicity forces the multiply connected superconductors threaded by flux $nh/2e$ to be degenerate in $n$. However, when $d \leq \xi_{BCS}$ the discrete quantum nature of the electronic states in the ring matters and the energies at half-integer and integer flux quanta are generally different, thus the supercurrent is $h/e$ periodic \cite{57}. They predict that one should see this periodicity for $s$-wave superconductors with rings smaller than $\xi_{BCS}$. Loder et al. also made these predictions for high-$T_c$ superconductors \cite{57}.

Wei and Goldbart studied $s$-wave NS boundary oscillations using BCS theory and Gor’kov’s approach at zero temperature. Ginzburg-Landau theory is not valid at low temperatures as it is a description of the center-of-mass wave function of the Cooper pairs, and the pairs can separate and rejoin, which the center-of-mass approach misses. They also come to the same conclusion as other studies, that $h/e$ periodicity should return for systems with very small radii \cite{58}.

Vakaryuk argues theoretically that $h/e$-periodicity arises in small superconductors due to the Cooper pair’s internal energy dependence on the center-of-mass state and not on the presence of quasi-particles \cite{59}. When the size of the superconductor approaches the BCS coherence length $\xi_{BCS}$ the internal energy of a pair becomes dependent on the overall phase of the wavefunction and differs for even and odd parity states. This leads to an offset in different current-carrying states for the different induced flux states of the superconductor, breaking the $h/2e$ periodicity up to $h/e$ periodicity as shown in figure 3.13.

Loder et al. did further studies in the crossover from $h/e$ behavior to $h/2e$ behavior in small superconducting rings \cite{60}. They developed a description of how the $h/e$ is balanced with $h/2e$ and transitions to $h/2e$. There are two different condensate states that, in the thermodynamic limit, are degenerate for integer and half-integer flux values \cite{61}. The degeneracy is lifted in discrete systems \cite{54,56}, which can be brought to bear in confined geometries \cite{53}.

Schwiete and Oreg theoretically studied how fluctuating Cooper pairs can cause a large persistent current in small superconducting rings $\xi_{GL} (0) > R$, despite being in the destructive regime, as well as what happens in rings where $R \gg \xi_{GL} (0)$ \cite{62}. They concluded that above $T_c$ pairs of electrons form for a limited time.

Motivated by the search for $h/e$-periodicity, instead of the typical LP $h/2e$-periodicity, Snyder et al. investigated small individual loops of superconducting Al \cite{63}. They found what they call a high-resistance state, with $R = 15 \times R_N$, that occurs at temperatures just below the $T_c$ of the Al nanorings. They posit that this effect is caused by a charge imbalance stemming from the non-equilibrium accumulation of quasi-particles near tilted NS interfaces, which occur naturally in constrained geometries such as the nanorings. They supported their argument...
with finite-element simulations, the results of which fit well with their experimental data. The model for their simulations was based on a model developed by Arutyunov et al. in which they proposed that the resistivity tensor close to the NS interface is anisotropic. However, this only predicted resistances up to 20\% higher than $R_N$. They proposed that further research in this area should move to measurements on Al films grown on sapphire substrates to achieve long-coherence-length Al samples, which may negate the effects caused by disorder and inhomogeneities in lift-off samples and give the first glimpses of $\hbar/e$ flux quantization in superconducting samples.
3.4 DOUBLE NETWORKS OF MESOSCOPIC LOOPS

In this section we will present the research performed on double networks mesoscopic superconducting loops. In addition we will also present the vortex/anti-vortex explanation of the enhanced LP oscillations that arose with this research. Finally, we present another vortex/anti-vortex explanation for the enhanced oscillation amplitudes seen in small two-dimensional superconducting loops.

In 2010 [64], Sochnikov et al. published a study on so-called “double networks” of high-\(T_c\) superconductors. An AFM micrograph of one of their samples can be seen in figure 3.14a. Double network is constructed by placing small squares rotated by 90° at the corners of the larger squares of the network. In figure 3.14a the large squares are 500 nm on a side and the small squares are 150 nm on a side. One of the motivating factors for using such a sample design geometry was that because as sample dimensions were decreasing, it was becoming harder to have measurements with good signal-to-noise ratios. By using a network like the one pictured in figure 3.14a they improve the statistics of their measurements and the samples are much more robust to small defects and damaged areas. As discussed below, they later showed that the small loops in the network are effectively decoupled from one another and do indeed behave as an ensemble of many individual small loops.

Their MR measurements on samples such as those shown in figure 3.14a not surprisingly exhibited oscillations as expected from the LP effect. What was surprising is that these oscillations had an amplitude that was an order of magnitude higher than that predicted by the LP effect.
Their explanation for the phenomenon was that the oscillations are caused by a periodic change in the interaction between thermally excited moving vortices and the oscillating supercurrent in the loop, based on the observations by Kirtley et al. and Kirtley et al. [65, 66] and theoretical work by Kogan et al. [4]. Despite the small size of the rings, they did not see oscillations with $h/e$ periodicities, as predicted for $d$-wave superconductors, or $h/4e$ as predicted for superconductors that exhibit stripes [67]. Sochnikov et al. later extended the model attributing the MR oscillations to interactions between vortices and anti-vortices with the supercurrent. This accounted for the interaction of magnetic moments of the vortices and anti-vortices with the external magnetic field. At the same time this also gave them a good model for the monotonic background upon which the LP oscillations are superimposed [68].

In a further study Sochnikov et al. describe the physics of a superconducting double network [69]. They perform numerical calculations to model the system and developed a mean-field approach in which the small loops placed at the vertices of the large loops are decoupled. They found that in both cases, which agreed with their previous experiments, the occupation of the large loops grows linearly with increasing magnetic field, as shown in figure 3.15. This is strong indication that the vortices are first distributed evenly among the large loops, and only later, when a certain energy threshold is reached, do the small loops begin to accept vortexes. This step-like behavior shows that the sub-lattice of small loops behaves like an ensemble of decoupled single loops.

Building on their model of superconducting double networks [69], Sochnikov et al. compared measurements on double networks of a high-$T_c$ superconductor with a single network of the same material [70]. They showed that a single network and double network behave differently in a magnetic field. With the single network they saw three different characteristics that indicate correlated behavior of the fluxoids, similar to previous findings [47, 52]. Their data show that single networks exhibit a finite slope of the MR curve at zero field, that the oscillation cusps are pointed downward, and that there are secondary dips in the MR behavior at half-integer values of $\Phi/\Phi_0$. The double network lacks these characteristics. Instead they have zero slope at zero field, their oscillation cusps are pointed upwards, an there is no secondary dip at half integer values of $\Phi/\Phi_0$.

Berdiyorov et al. offered a similar explanation for the enhanced oscillation amplitude based on MR measurements they performed on ladders of superconducting Nb, an example of which can be seen in the SEM image inset in figure 3.16. They created a model based on current-induced vortex motion to explain the anomalous MR amplitudes seen by Sochnikov et al. [64] in high-$T_c$ superconductors and tested it on specially prepared niobium samples [71]. Their measurements on the Nb ladders showed truly different behavior than LP oscillations. Although the periodicity was the same, they saw enhanced amplitudes at lower temperatures than what LP predicts. The sample size, relative to the sample’s coherence length, was such that the thermal fluctuations proposed by Sochnikov et al. [64] are not applicable. Their conclusion was that the large MR oscillations stemmed solely from vortex nucleation, motion and dissipation driven by an interplay between the dc current and shielding supercurrents induced by the external magnetic field.
Figure 3.15: Demonstration of the effect of the loop size ratio on the shape of the oscillations [69]. a) The normalized energy per unit-cell for different large loops side length $L$ to small loop size length $l$ ratios. b) The average vortex occupation of both large and small loops for increasing magnetic field strengths. One clearly sees the step-like behavior of the small loops, starting with zero vortex occupation and jumping up to an average of 1 vortex per loop when $\Phi = \Phi_0/2$.

Figure 3.16: A summary of the measurements on and a SEM image of a Nb ladder. The curve represented by the white circles is from a $R$-versus-$T$ measurement taken at zero magnetic field and 1 $\mu$A. The inset on the left shows the temperature-dependent oscillation amplitudes resulting from their measurements as well as from their GL dynamical model and the LP model. The inset on the right shows an SEM images of the Nb sample with the sample’s dimensions [71].
3.5 THE LITTLE-PARKS-DE GENNES EFFECT

As a modification to the LP effect, Paul de Gennes proposed that by adding side branches, through which no transport current flows, the superconductivity in and near the so-called destructive regime could be restored [72]. The idea behind his proposal is that one could use the LP effect to drive $T_c$ of a superconductor all the way down to zero, thus called the destructive regime. Adding, as he called it, a "dead end tail" would restore superconductivity in this regime.

Soon thereafter, Alexander studied networks of thin wires using the linearized Ginzburg-Landau equations [73, 74]. In the case of a thin superconducting ring he found that adding an open side branch attached to the loop increases the critical field [73].

Following this, Fink and Grünfeld theoretically examined a long superconducting wire with periodically placed dangling wires longer than the temperature dependent coherence length $\xi_{GL}(T)$ [75]. The presence of these side branches increases the critical-current density of the entire sample.

The first experimental confirmation of the destructive regime, was delivered by Liu et al. [76]. They fabricated hollow cylinders with diameters close to the temperature dependent coherence length of the material used, $d \approx \xi_{GL}(T)$. They were able to drive the $T_c$ of their cylinders all the way down to 0 $\Omega$ at magnetic field strengths equal to half-integer flux quanta through their cylinders.[76]

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Terai et al. studied a line, loop and disk [77]. They also saw an anomalous negative MR at low fields. Because they saw this same negative MR in all three types of samples, they conclude that it is not caused by sample geometry. Along the lines of the argumentation of [75], they argued that the side branches help enhance superconductivity at the nodes and that in the middle of the wire superconductivity is suppressed, creating an NS interface near $T_c$, which is known to cause a quasi-particle charge imbalance induced by the external current. They claimed the NS boundaries arise spatially near the nodes of the voltage probes and that the superconducting state within the ring or disk is still coherent, even when the negative MR is observed. This study further confirmed the prediction of de Gennes [72], that the destructive regime in small superconducting rings exist at certain external flux values.

Lopatin et al. theoretically explored quantum corrections to normal state conductivity near a quantum phase transition and predicted that for a cylinder with a small enough diameter that it should be possible to push $T_c$ down to zero at half-integer-flux [78], which agrees well with the experimental findings of Liu et al. [76]. Shah and Lopatin studied this further and predicted non-monotonic behavior when the superconducting sample (wire, thin cylinder, or thin film) is in the quantum regime [79].

Sternfeld et al. studied QPTs and the destructive regime of very small superconducting loops [80]. They actually achieve a range around $0.5 \Phi_0$ in which the samples return to the normal state resistance before transitioning back into the superconducting state. They found that a mean-field theory fits their data well, implying that inhomogeneities play a role in determining the cylinder resistance.

Staley and Liu study the Little-Parks-de Gennes (LPdG) effect, the destruction and restoration of the superconducting phase at half-integer-flux quanta through the addition of a superconducting side branch [81]. With their devices they experimentally verified for the first time the predictions of de Gennes [72] that there is a destructive regime and that adding a side branch suppresses this, or in other words restores superconductivity. As Staley put it, the side branch increases the energy required for entering the destructive regime. In other words, the
LPdG effect can be understood in terms of the proximity effect: the addition of a side branch raises the energy cost for entering the destructive regime, stabilizing superconductivity in the loop.
EXPERIMENTAL TECHNIQUES

In this chapter I will first outline the procedures used to fabricate the samples both at Bar Ilan University in Ramat Gan, Israel, and at the University of Konstanz in Germany. This will be followed by a discussion of the low temperature techniques employed to get high-quality magnetoresistance measurements of our networked samples. Following this general discussion, the two following discussions will look at the magnetic field independent thermometry we developed for more accurate temperature control and the use of a PI feedback control loop (see section 4.2.3) to hold steady temperatures while taking measurements.

4.1 NANOSTRUCTURED LOW TEMPERATURE SUPERCONDUCTORS

The first set of samples we examined were produced at Bar Ilan University using a dry etching technique. This was based on a process they developed for creating double networks of high-temperature superconductors, which are ceramics and not able to be prepared using the lift-off technique often used when creating nanostructured metallic samples. The equipment used to create the initial batch of samples were moved and replaced and a suitable recipe was not found on the new equipment. Attempts at creating a similar recipe with the equipment available at the University of Konstanz were also met with very limited success. This motivated the development of the lift-off recipe that was used for the final samples measured.

4.1.1 Dry Etching Samples

For all samples studied, the substrate used were silicon chips with a 200 nm oxide layer. All of the silicon chips originated from our supplies at the University of Konstanz. The aluminum films were deposited initially via thermal evaporation in our custom-built ultra-high vacuum evaporation chamber. These were then shipped to Israel, where Omri Sharon performed the remaining fabrication steps.

Using a dry etch method for nanostructuring requires a mask to protect the material that will have the final form. In the case of our samples we use cross-linked PMMA as a mask [82]. This is different from the normal use of PMMA, where it is used as a sacrificial layer when creating samples via lift-off. Cross-linking the PMMA requires much higher doses than normal use of PMMA, but it produces a very robust mask that stands up to argon sputtering and other reactive ion etching methods. The cross-linked PMMA allows for very fine structuring, down to the tens of nanometers, similar to other negative resists [83].

The negative resist procedure is illustrated in figure 4.1. First about 30 nm of aluminum is evaporated on the silicon oxide surface of the chip. The PMMA electron beam resist is then applied with spin-coating and then dried in an oven. Electron-beam lithography is used to write the pattern into the PMMA. Instead of using the normal doses for PMMA, a much higher dose—and thus longer exposure time—is required to get the PMMA to cross-link. This creates four regions in the PMMA that gradually transition from the over-exposed PMMA that describes the layout of the sample geometry, to the unexposed areas that are the predominant
Figure 4.1: Negative resistance procedure for sample fabrication. a) Aluminum is evaporated onto the substrate, followed by the application of the PMMA resist for electron beam lithography. b) The PMMA is exposed. The central area (red) is overexposed creating an extra-hard region in the PMMA. The bordering regions receive normal exposure or are under exposed (dark orange and orange). The surrounding region is not exposed at all (yellow). c) Development and rinsing remove the normally exposed regions, leaving the PMMA that was either over exposed and hardened, or the PMMA that was not exposed at all. d) Either argon sputtering or reactive etching with a chlorine-based gas removed the regions of Al uncovered during the development step. e) Acetone is used in the final step to remove the PMMA left after etching.

area on the surface of the chip. The development process then dissolves and washes away the PMMA that received a normal dose of electrons, which results in a small trench in the PMMA between the mask and the surrounding areas on the surface. Initially we tried to use argon sputtering in our reactive ion etch (RIE) machine to etch the samples. Unfortunately no stable etching rates were found and only a handful of working samples were ever produced. In some instances the sample was barely affected by the argon plasma and in other cases the entire etching mask and all aluminum were removed from the silicon chip.

The second etching process we tried employed a chlorine-base gas mixture in an inductively coupled plasma (ICP) generator from Oxford Instruments. This process was more successful than using the RIE to etch the samples, but the yield was low and the time investment was very high. For this reason we pursued a sample recipe based on the normal lift-off technique.

There were other problems associated with this sample fabrication procedure and the sample design that it dictated. Due to the long exposure times, the contacting pads had to be kept small. Instead of creating solid contact pads, a spiral-like shape was used to reduce the area that had to be exposed. The reduced size of the pads along with the fact that they were separated from the surrounding aluminum by a narrow trench made electrically contacting to the pads very difficult. Contact was achieved with a wire bonding machine that used a 50 µm thick aluminum wire. The foot created during the bonding process was just barely smaller than the bonding pad, requiring exact placement of the bonds. Any contact with the surrounding aluminum would prohibit successful measurement of the sample. Usually the samples were robust enough to allow for a few attempts at placing a bonding pad. However, beyond five
attempts the bonding pads began to degrade quickly. As a result many samples did not survive past the bonding process and a few samples had just two or three working bonding pads, so we had to give up the normal four point measurement technique and measure the samples using only two or three of the contact pads. The situation greatly improved once we switched over to the normal lift-off technique.

4.1.2 Lift-Off Samples

The more successful sample fabrication technique utilized lift-off. This process begins with spin-coating the clean silicon substrates with MMA-MAA EL6, the sacrificial layer, followed by PMMA A2. Next, electron beam lithography was performed, using normal PMMA doses. Development washed away the areas into which the aluminum of the final nanostructure would be evaporated.

Evaporation was initially done at room temperature with a thermal evaporator. About midway through this project we began having cross contamination problems with the magnetic materials in the evaporation machine. For at least a year we were unable to produce superconducting samples with this machine. Fortunately we had another evaporation machine available. Aluminum in the second machine was evaporated with an electron gun and the samples were cooled down to liquid nitrogen temperatures. We decided to cool the samples down based on some tests performed by another member of the lab.

The final step in the sample production process was the lift-off step. Initial tests on single-loop structures warned us that there may be problems getting the ‘caps’ removed from where the holes in the film are supposed to be. See figure 4.3 for an example of this.

However, once we began writing double network samples we found that we did not have to worry about this very much. At least 50% of the caps were washed away during the lift-
Figure 4.3: Lithography test structures where a few of the "caps" were not removed during the normal lift-off process. This was acceptable because, as stated earlier, the advantage of using the network samples was that inhomogeneities were averaged out. Figure 4.4 shows an electron microscope image of sample LOS003 on which not all of the small loops developed into viable loops. Half of the small loops were complete, however, about a third of the holes were completely filled in, and just under a third resulted in loops that were not completely closed.

Despite these problems, we were still able to manufacture a number of viable samples with good reliability.
Figure 4.4: SEM image of sample LOS003. Only half of the small loops were viable. The other half did not develop into loops at all, or were damaged.
One of the most important components in the measurement setup is the thermometer, as our goal was to investigate thermal processes in the NS transition. In order to do this we needed to achieve a fine-grained temperature control of under 1 mK near transition temperature of aluminum while sweeping an external magnetic field. This can be especially difficult as a time-varying magnetic field will induce eddy currents in the metal parts of the cryostat and other metallic components and produce heat. Furthermore, as we found out during our experiments, the temperature sensors can have magnetically induced transitions that not only cause an error in temperature measurement, but can also produce heat, effecting the superconducting state and behavior of the sample.

We were able to overcome both of these problems. There are many solutions for reducing eddy currents in a cryostat, for instance through smart design of the cryostat's parts such that no large currents can build up. The simplest solution is to sweep the magnetic field slowly. This effect of this is twofold: as the magnitude of the current is directly proportional to the rate of change of the magnetic field, this reduces the currents that can arise. Second, this gives the system enough time to dissipate any generated heat before it can greatly affect the sample. Solving the problem of magnetic effects internal to the thermal sensor was more difficult. We found nonlinear, field-dependent internal effects in our custom-built ruthenium oxide (RuO$_2$) thermometers that led us to build capacitive thermometers that did not have magnetic field dependencies. These had less sensitivity than the RuO$_2$ thermometers.

### 4.2.1 Resistive Sensors

Resistive sensors are the most popular sensors used in low temperature physics as they posses many beneficial qualities, including increasing sensitivity with decreasing temperature and ease of use [85]. As resistive thermometers are not primary, they must also be calibrated against other calibrated secondary thermometers or a primary thermometer.

In our lab we used RuO$_2$ thermometers as they have proven to be accurate even in high magnetic fields: only 7% deviation from the zero-field resistance even at 7.5 T [86]. We built our thermometers in the lab from four 5.32 kΩ SMD resistors soldered in parallel. These thermometers require thermal preconditioning before calibration as the thermal cycling stresses cause mechanical changes in the thermometer, which also change the resistance. After a few cycles the thermometer stabilizes and can be calibrated.

We calibrated our thermometers against other calibrated secondary thermometers available to us in the lab. The measurement device we used was a Model 370 resistance bridge from Lake Shore Cryotronics, Inc. This device uses a 4-point measurement method along with the lock-in technique to achieve very accurate measurements with a good signal-to-noise ratio. The excellent signal-to-noise ratio allows the use of very small sensing voltages, which significantly reduces self-heating effects, pushing the operating range of the sensor to temperatures below 1 K.

Despite the advantages and low magnetic field dependence of RuO$_2$ thermometers, ours proved to have magnetic field induced self heating effects at certain magnetic field strengths [87]. Figure 4.5 shows the anomaly seen with our thermometers and that it is dependent on the magnetic field sweep direction and the sweep rate. Because of these large anomalies we had to look for an magnetic field independent alternative to our RuO$_2$ sensors.
4.2 Low Temperature Thermometry

Figure 4.5: Magnetoresistance measurements of the RuO$_2$ sensor using different sweep rates [87]. A drop in resistance—which translates to a rise in temperature—of greater than 7% is seen in the fastest sweeps whereas the slowest sweeps exhibit negative resistance spikes of just over 1%. There is also a very clear hysteresis; the spike always occurs after the external magnetic field has passed through 0 T and at absolute field strengths just below 0.05 T.

4.2.2 Capacitive Sensors

A capacitive thermometer uses the fact that the susceptibility of various dielectrics have a relatively strong temperature dependence. The susceptibility is not magnetic field dependent, providing us with a means of magnetic field independent temperature sensing. But because the susceptibility is also dependent on the distance between the two electrodes, it will also change due to thermal expansion and contraction as one cycles the sensor through a wide temperature range. This means that a second thermometer in addition to a capacitive thermometer is often necessary to calibrate the capacitive sensor every time it is cooled down.

We built our sensors using the method developed by Murphy et al. [88]. They consist of a long, thin foil of kapton, 7.5 µm thick, sandwiched between two thin copper foils, each 15 µm thick. These were glued together with an epoxy, Stycast 1266, and rolled up into a small package. Depending on the geometry we used, each sensor had a capacitance in the range of 2–7 pF. Measurements were performed with both an Andeen-Hagerling 2700A and 2550A Capacitance Bridge.

Our measurements with the capacitive thermometer all but confirm our suspicions that the RuO$_2$ thermometer was the source of the heating we saw in the system. There was a spike in the capacitance that occurred simultaneously with the spike in the RuO$_2$ sensor. Again we found that the magnitude of the temperature spike was mitigated by slower sweep rates and in sweeps performed at higher temperatures. We found that using the capacitive sensor as the input for the PI heater control lead to more stable temperatures, as shown in figure 4.6.

In summary we found that we could avoid this large heating effect primarily by using the slowest sweep rate that the helium-3 holding time would allow, for the measurement
Figure 4.6: Temperature control using the signal from the capacitive sensor [87]. The sweeps rates were held at 5 mT/min from −100 mT to 100 mT and raised to 30 mT/min outside of this range.

temperature. We also used the capacitive sensor, but it seems as the RuO\textsubscript{2} sensor aged, the self heating effects diminished considerably.

4.2.3 Feedback Control with a PID Controller

PID control, for proportional-integral-derivative control, is a closed loop, feedback control method often used in temperature controllers [85]. This controller works by calculating the difference, or error, in real time between the desired temperature, the setpoint, and the actual temperature of the system. Through the application of corrections to the system, the controller minimizes the error and the system approaches the setpoint. In a temperature control system the corrections are applied to the system through a heater, the output of which is calculated by

\[
\text{Output} = P \left[ e(t) + I \int e(t) \, dt + D \frac{de(t)}{dt} \right],
\]

where \(e(t) = \text{setpoint} - \text{current value}(t)\) is the error of the system at time \(t\), \(P\) is the proportional coefficient, \(I\) the integral coefficient, and \(D\) the derivative coefficient. The proportional term counteracts the current error in the system and if acting alone can never completely remove the error from the system [85]. The integral takes past errors into account to counteract the current error. When added to a \(P\) controller acting alone, this term will remove the remaining error that a \(P\) controller cannot. Finally, the \(D\) term can be thought of as the term that tries to counteract anticipated error in the future, by reacting quickly to changes in error in time. In noisy systems the \(D\) term tends to amplify the noise and is often left out, as we did in our system, resulting in a PI controller, as we used.

There are many methods for tuning PID systems to find the ideal settings for the \(P\), \(I\), and \(D\) coefficients. We used the popular manual method as described in ref. [85].
4.3 MEASUREMENTS AT LOW TEMPERATURES

Measurements were performed in a number of different cryostats. Most of the measurements were done in one of two different HelioxVL helium-3 cryostats from Oxford Instruments. The initial cryostat used was the same as in my diploma thesis [89], the “Scheer Heliox”, which was iteratively modified as the requirements of the samples and chip carriers changed. The second Heliox was loaned from the Fonin group and will be referred to as the “Fonin Heliox”.

The details of how these cryostats work are beyond the scope of this dissertation. Those interested can review the sections on the measurement setup in [87] and [84].

4.3.1 Electronic Setup

The electronic setup consisted of the measurement rack and the cabling of the individual cryostats used.

ELECTRONICS RACK

The measurement rack contained the measurement electronics in five separate circuits. These were

1. the sample resistance measurement circuit,
2. the Heliox thermometry circuit,
3. the ruthenium oxide thermometry circuit,
4. the magnet power supply circuit,
5. and in some cases the capacitance thermometry circuit.

![Schematic of the cryostat along with the electronics rack used for control of the experiment and recording of the data](image.png)

Figure 4.7: Schematic of the cryostat along with the electronics rack used for control of the experiment and recording of the data [87].

A schematic of this setup along with the cryostat, dewar, and 1.5K-plate pump is shown in figure 4.7.
Most of the resistance measurements on the sample were performed with a bias current below 500 nA. As a result we used SR830 lock-in amplifiers to perform the four-point measurements. A Yokogawa 7651 served as the DC voltage and current bias. A custom-built adder combined the DC signal from the Yokogawa and the AC signal of 4 mV at 117.17 Hz, which was then fed into the cryostat on the bias input lead. The signal passed through the sample and was amplified by a Femto DLPV-200 current amplifier, from which it was then read by one of the lock-in amplifiers. To complete the four-point measurement two additional electrical leads, above and below the sample in the electrical potential landscape, lead off to a Femto DLPV-100 voltage amplifier, from which the voltage signal was then fed into the other lock-in amplifier. The output from both lock-in amplifiers was then fed into an ADWin Gold data acquisition system, where they were digitized and passed on to the computer to be saved.

The Heliox cryostats each came outfitted with an Allen-Bradley thermal element at the helium-3 sorption pump ($T_{\text{Sorp}}$), two 2.2 kΩ ($\text{RuO}_2$) thermal elements at the 1.5 K plate ($T_{\text{K}}$) and helium-3 pot ($T_{\text{He}_3}$), and heaters at the helium-3 sorption pump and the helium-3 pot. These five elements were controlled through the Oxford Instruments ITC 503 temperature controllers that were delivered with each cryostat.

Initially we only used a custom-built RuO$_2$ thermal element to measure the temperature near the sample. This element was read out with a LakeShore 370 AC Resistance Bridge. As we later found, this exhibited a non-negligible magnet field dependence at very low temperatures.

Finally, for some of the later samples we were able to use the custom-built capacitive thermometer that we made along with the Andeen-Hagerling 2550a Capacitance Bridge. Unlike the Scheer Heliox, the Fonin Heliox did not have individually shielded cables that we could use for the capacitive measurement. As a result the signal from the capacitance proved to be too unstable for use with a temperature control feedback loop.

**Scheer Heliox**

The experimental cabling in the Scheer Heliox was installed many years ago by Christian Schrim and Hans-Fritjof Pernau and has since remained largely unchanged [90]. The cabling included six 490 Ω twisted pairs of 50 µm-thick manganin wires, each 1.5 m long. Each twisted pair was placed in a stainless steel capillary with a 200 µm inner diameter where they were glued into place with silicone diluted with toluene. These twisted-pair wires also had a capacitance of about 375 pF, which made them effectively low-pass filters with a cut-off frequency of 5.44 MHz. In addition to the high-resistance lines, there were two low-resistance lines. These were constructed out of 112 µm thick brass and were also placed, individually, in stainless steel capillaries with a 200 µm inner diameter. These wires had a resistance of 15.4 Ω, a capacitance of 645 pF and a theoretical cut-off frequency of 101 MHz. These two wires were used to connect to the capacitor thermometers we later developed.

The original break-out-flange did have to be replaced as enough of the BNO plugs, which were made vacuum tight with a combination of Stycast Epoxy and a Vacséal resin, broke so that we did not have enough plugs for connecting to the sample, thermal sensors and heater. The original flange was replaced with a break-out-flange that used vacuum-rated Lemo and SMA plugs. The advantage of this is that when the plugs break they can be individually replaced. The Lemo plugs are also much more robust than the BNO plugs.

After passing the helium-3 pot, the custom cables were then passed through custom-built copper powder filters. The original filters were built using 50 µm thick copper wires but later models used 90 µm thick copper wires. The wires are wound in a special pattern to cancel
out self-induction and placed in the cylindrical cavity of a larger hollowed-out copper block. A copper powder with an average particle size of about 40\,µm was then filled in and the cavity is closed on both ends by the connecting plugs. The high frequency currents travel predominantly on the surface of the conductor and thus loose much of their energy to the oxide layer surrounding each grain in the powder closely packed around the wire. Further details on these filters can be found in [90] and in the Bachelor’s Thesis of [Thalmann 91].

Below the filters short lengths of wire soldered to the required plugs used to connect to the sample, sensors and heater. These were not built with the intention of providing further filtering to the signals carried over them.

Finally, the cryostat was equipped with multiple flat band cables to connect to the various sensors and heaters installed, as well as a free 24-lead (12 twisted-pairs) ribbon cable. Because the number of available working plugs on the original break-out-flange was limited, we used this to connect to our sample thermometer.

**Fonin Heliox**

The Fonin Heliox was almost identical to the Scheer Heliox. The main difference is that there was an additional 12 twisted-pair ribbon cable installed in the free port where the custom-built cables were installed in the Scheer Heliox. As a result, all measurements were moved to this cabling. Some tests were done with the copper powder filters used in the Scheer Heliox and no significant different was found between the signal-to-noise ratios with or without filters. The IVC of the Fonin Heliox had a smaller inner diameter and the size of the filters was such that there was light contact somewhere between the filters and the inside of the IVC. The base temperature of the cryostat was increased by this and its holding time was decreased. Most measurements in this cryostat were performed without any additional filtering above and beyond what the installed cables already achieved.

4.3.2 Magnetoresistance Measurements

The main type of measurements we took were magnetoresistance measurements at different temperatures, similar to what Little and Parks did in their original experiment and what others have done in later experiments on both low-$T_c$ superconductors and high-$T_c$ superconductors [13, 34, 65]. We found that we could also reliably extract the temperature behavior from the MR measurements which allowed us to find the $T_c$-versus-$B$ phase boundary.

4.3.2.1 Determining Resistance vs. Temperature Behavior

When determining the resistance versus temperature behavior of the samples there were three options available to us. The first is to use the data recorded during the initial cooldown as well as the subsequent condensation runs of the helium-3. The problem with this method is that due to thermal lag in the system there is usually a hysteresis and offset from the true transition temperature in this data.

The second option was to start at a temperature below $T_c$ and, using the PI feedback with the heater, to slowly raise the temperature above $T_c$. The advantage of this method is that the long measuring times allow temperature gradients relax back into equilibrium. The disadvantage of this method is that it takes a long time.
The third method is to extract the data from the magnetoresistance measurements taken at fixed temperatures. This method is only useful if temperature difference between subsequent MR measurements is small enough to give us a high enough resolution. As this was the case for some samples, this option was attractive as no extra measurements to determine the temperature behavior were needed. However, the question still remained whether this would produce the same results as the slow temperature sweep method. A comparison of temperature sweeps and extracting the temperature data from MR measurements is shown in figure 4.8. Here we compare slow temperature sweeps taken at specific magnetic fields to data extracted from the MR measurements at those field strengths. In figure 4.8a we see seven lines overlayed over the MR data to represent the magnetic field strengths where data was extracted. The data extracted along these lines is shown in figure 4.8b as dashed lines with the same color coding. For comparison we performed slow temperature sweeps at these same magnetic field strengths, the data of which are shown as solid lines in figure 4.8b. There is little difference between the extracted data and the directly measured data. Thus this proved to be a viable method of determining the thermal characteristics of the samples at various magnetic field strengths without having to perform additional measurements.

4.3.2.2 Determining the $T_c$-versus-$B$ Phase Boundary

Two different methods were also available to us for determining the phase boundary between the normal and superconducting phases of the sample. Again we wanted to test the difference between direct measurement and indirect extraction of the data from MR measurements. Figure 4.9 shows a direct comparison of the extracted data and the directly measured phase boundary.

To measure the phase boundary directly we set our PI heating circuit to hold the resistance of the sample at half the normal resistance of the sample, $R = 12.0 \, \Omega$, which was the definition...
of the so-called transition resistance that we used. This is in contrast to having the PI heating circuit hold a stable temperature during MR measurements. Once the system stabilized at the desired resistance and temperature, we slowly swept the magnetic field from −10 mT to about 53 mT, which is represented by the green trace in figure 4.9. We see the expected Little-Parks oscillations in the transition temperature $T_c$ from both the large and small loops in the network, which give rise to the short and long periodic oscillations respectively. We compare this with data taken from the MR measurements at different temperatures. The blue dots represent data within ±0.5 Ω of the transition resistance $R_T = 0.5R_N = 12.0$ Ω. We see that data extracted from the MR measurements follows the progression of the directly measured phase boundary closely. However, the small oscillations caused by the large loops are not resolved in this data. This would require measurements in smaller steps than the 5 mK that we used between 1.395–1.455 K. Furthermore, there is a gap in the measurements between 1.360 K and 1.395 K so that we do not have MR data around the local $T_c$ maxima near $1.8\Phi_0$, preventing a reliable fit to the data above $1.5\Phi_0$.

Despite these shortcomings, we still see very clearly the oscillations from the small loops superimposed on a parabolic background that closely follows the progression of the phase boundary that was directly measured.

Figure 4.9: Comparison of data extracted from all magnetoresistance measurements and the $T_c$-versus-$B$ phase boundary that was directly measured.
Our initial goal was to try and measure $h/e$ oscillations as predicted by theory in sufficiently small samples. The important constraint is that the temperature-independent coherence length of a Cooper pair, its physical size, must exceed the circumference of the loop. But there is a clear trade-off between having a long coherence length, but low critical field, and a short coherence length but high critical field. This is just one of many considerations that had to be accounted for when designing the samples we used. The following chapter starts off with the presentation and a discussion of the coherence lengths of our samples based on their resistivities and upper critical fields. These values will be used as a baseline for comparison to coherence length values obtained from the Little-Parks-like behavior of the samples in external magnetic fields. The remainder of the chapter details the progression of our sample design based on the measurements of our samples, culminating in the examination of the first fully functional lift-off sample.

### 5.1 Characteristic Lengths

For many of the structures studied here it may be difficult to get a good estimate of the relevant lengths. For example, while the design geometries of the samples are known, for many of the samples it was not possible to exactly determine the dimensions of the entire sample, i.e. that line widths were uniform over tens of micrometers. The estimates for the entire sample geometry that went into the calculation of the resistivity were taken from small sections of the networks and extrapolated to the whole network. Despite all of the problems associated with determining the characteristic lengths in our samples, calculating them gives us at least a general idea of the quality of our samples and at what behavior to expect from them.

As discussed in section 2.3.1, there are a number of methods of inferring the coherence length of a superconductor. The results obtained for our samples are summarized in table 5.1.1

### 5.1.1 Coherence Length From Resistivity

For the first few etched samples, samples SiO$_2$AlO$_2$d, SiO$_2$AlO$_2$e, Omri1 and SiO$_2$Al122, our initial estimates of the coherence length based on the resistivity and geometry of the sample indicated that these samples had coherence length smaller than 100 nm, a value for the coherence lengths often cited in literature for lift-off type samples [92]. This was just another reason for us to peruse a lift-off sample recipe after SiO$_2$Al122 was measured. The coherence length estimate for sample SiO$_2$Al122 is very small, probably due to a poor estimate of the elastic mean free path, $l$. The last etched sample, SiO$_2$Al143, showed a much longer coherence length than the others. Unfortunately, even in this sample the smallest loops were so small that we did not see oscillations in the MR behavior before the sample returned to the normal state. At this point our lift-off sample recipe was complete and we were quickly able to measure the first lift-off sample, LOT12. This sample also showed a long coherence length. The subsequent lift-off samples had even longer coherence lengths.
5.1.2 Coherence Length From Critical Field

We were also able to calculate the coherence length using \( \xi_{B_c \parallel} \) and our estimates of \( B_{c \parallel} (0) \). However, only a few samples were measured extensively enough to estimate \( B_{c \parallel} (0) \), so only calculated coherence lengths for these samples.

The extremely long coherence length calculated for sample \( \text{SiO}_2 \text{Al}_{122} \) is probably incorrect and can be attributed to difficulties estimating the average line width of the network on the sample and because there were was not a large set of measurements from which we could estimate \( B_{c \parallel} (0) \). For the last etched sample our calculations result in a coherence length that falls within the range of expected coherence lengths for these samples and is close to the coherence length calculated from the resistance.

The coherence lengths calculated from \( B_{c \parallel} (0) \) for the lift-off samples are more tightly clustered at lower values than the coherence lengths calculated from the sample resistances. It remains to be seen how these values hold up when compared to the values we obtain from the LP behavior of the samples, which is mathematically described by equation (3.3).

5.2 DOUBLE NETWORKS

5.2.1 Sample SiO2Al002

The first sample we measured, SiO2Al002 had multiple independent networks on a single chip, named a–e, of which we only measured networks d and e. As we were still in the design, construction and testing phase with the measurement setup we were unable to collect all
Figure 5.1: Magnetoresistance measurements performed on network d of sample SiO$_2$A1o2. This sample was measured using a two-point measurement configuration with aluminum wire bonds to electrically connect the sample, which explains the drop in resistance seen at low fields and low temperatures. The lead resistance was also subtracted from the measurement resistance. As the temperature rises, double peaks in the magnetoresistance begin to appear with increasing amplitude as the temperature increases. For a discussion on the origin of these double peaks see the text.

relevant data for a complete analysis of the sample and networks. However, our initial results helped us refine both the measurement setup and the sample design.

5.2.1.1 Network d

Figure 5.1 shows the MR sweeps at different temperatures. The range of the resistance is reduced to emphasize the oscillations. Figure 5.2 shows the same sweeps but zoomed in to narrower fields and smaller resistances to show only two peaks and the connecting trough of the oscillations. This sample was measured in a two-point configuration and the lead resistances have been subtracted from the measured resistance. The measurements were not taken close to $T_c$ for the network. Estimates for $T_c$ for this network were inferred from a measurement recorded during the condensation of the helium-3 in the cryostat and a fast temperature sweep measurement, where the sample was heated to 2.0 K and allowed to quickly cool back down, shown in figure 5.3. We can only narrow $T_c$ down to somewhere in the range from 1.167 K to 1.474 K.
The first problem with these temperature measurements, and why we can only estimate the $T_c$, is that the thermal properties of the cryostat are such that the actual temperature of the sample lagged considerably behind the temperature read out by the thermometers. The second problem is that for this measurement series we did not have a thermometer installed near the sample. All of the temperature data comes from the thermometer at the helium-3 pot, over 15 cm above the sample! One of the effects of this is that the temperature was not stable at the sample during the measurement series. This is evident by the fact that the sweeps are not symmetric around 0, which is very clear for the 1156 K sweep in figure 5.1.

Despite these problems with the measurements, it is still instructive to examine the MR behavior of the sample. For instance, we see “double peaks” at half integer values of the flux. Something similar to this was seen by Bruyndoncx et al. [51]. Their system consisted of two squares of equal size sharing a common strand. At certain field strengths the superconductivity in the strand was suppressed and the area was effectively doubled. In our case the small loops, which dictate this periodicity in the MR measurement, do not share a common strand and are in fact separated by 2800 nm-long strands, so the same mechanism cannot be responsible for this double peak.

Another possibility, as shown by Sochnikov et al. [69], is that when the ratio of the large loop side length and small loop side length is small, the small oscillations from the large loops...
can affect the shape of the large oscillations from the small loops, especially at the peaks of the later. Zooming in very closely to the troughs of the large oscillations, one could imagine that the small "noise" one sees is from the small periodic oscillations from the large loops. Based on the side length ratio of about 7:1, these would have a period at least 49 times smaller than that of the small loops. Obviously then the similar size argument is invalid. Furthermore, we can also see these small oscillations superimposed on the suppressed peaks of the large oscillations.

In any case, superconductivity is being restored at the peaks as opposed to being further suppressed as we would expect from the LP effect. In the LP picture this would mean that the circulating current is being suppressed, i.e. its velocity is reduced and thus its kinetic energy as well. One possibility may be that since we measured this network with the LakeShore Model 370 resistance bridge, there may be something else going on with the measurement setup that may cause a systematic error in the measurement like we see here.
Figure 5.3: $R$-versus-$T$ measurements for network d on sample SiO$_2$AlO$_2$. Because the measurement thermometer was not placed close to the sample, the thermal lag caused by the copper between the thermometer and the sample leads to a hysteresis in the $R$-versus-$T$ measurement.
Figure 5.4: Magnetoresistance measurement taken on network 2e of sample SiO$_2$Al002. The LP effect is clear to see as periodic oscillations occur with a frequency of $\Phi_0$. Thermal control of the system during this measurement was poor, leading to the asymmetry about $B = 0$ mT, evident when comparing the peaks of very different heights close to $-2.5\Phi_0$ and $2.5\Phi_0$.

5.2.1.2 Network e

After making some changes to the cryostat, including the installation of a sample thermometer, we measured network e on sample SiO$_2$Al002, which had the same dimensions as network d. Despite the changes we made to the cryostat, we still had problems bonding the sample and thus were limited to using a two-point geometry instead of a four-point geometry. The finite resistance from the leads was subtracted from the data shown in figure 5.4. The only MR measurement we were able to take is shown in figure 5.4. One sees again that the measurement was very asymmetric around zero field, which was due to poor temperature control. We used the temperature controller for the helium-3 system in the cryostat to try to regulate the temperature of the sample. The heater and sensors for this system were located almost 20 cm higher up along the cryostat than the sample and sample thermometer, which meant that we had very poor control over the temperature at the sample.

One also sees that just before the sample drops from its normal state into the transition to the superconducting state that the resistance increases above the normal state resistance. This result is to be expected when high bias currents are used, which was the case here. This measurement was performed with a bias current slightly above 0.5 $\mu$A. More discussion on this effect will come later when the MR measurement results for sample LOS003 are presented.
Figure 5.5: From the same magnetoresistance measurement as depicted in figure 5.4, this shows a zoomed in section of the measurement between $1.3\Phi_0$ and $2.3\Phi_0$. We see here in the trough between two peaks the small oscillations caused by the large 2800 nm loops in the network. As the area of these loops is 49 times larger than the area of the smaller loops, the oscillations are 49 time smaller.

The encouraging result from this sample was that we were able to resolve the small oscillations caused by the network of coupled large loops, which had 2800 nm on a side, in addition to the MR oscillations caused by the network of decoupled small loops with only 400 nm on a side.

Because the results from both 400 nm networks were so encouraging we tried to measure networks with smaller loops. We had two such samples, SiO$_2$Al$_{122}$ and SiO$_2$Al$_{143}$, which had estimated 210 nm and 245 nm side lengths, respectively.
5.2 DOUBLE NETWORKS

5.2.2 Sample SiO2Al122

The third etched sample we successfully measured was SiO2Al122, depicted figure 5.8. The sample was designed to have small loops with side lengths of 210 nm, as shown in figure 5.6, and large loops with side lengths of 1750 nm.

This sample was also measured in a two-point configuration, leading to a finite resistance from the measurement leads, which was subtracted from the data in figure 5.7 and figure 5.8. The MR measurements in figure 5.7 were taken with an absolute bias current below 0.5 µA, whereas the measurements in figure 5.8 were all taken with a bias current just above 0.5 µA. Discussion on the differences between these conditions will follow with the analysis of sample LOSO3 in section 6.2.1. Based on the low current measurements, we see no oscillations due to the 210 nm loops. Small oscillations due to the large 1750 nm loops do begin to appear above 1.7 K.

Despite the lack of large oscillations, there are however steps, which could indicate that different parts of the sample, possibly leads, bonding pads, and bonding wire, transition from the superconducting to the normal state at these various fields and temperatures.

The high bias current measurements also exhibit LP oscillations from the large loops, shown in figure 5.8. There are other features which one might conclude are LP oscillations from the small loops. The more likely explanation, which will be explored in depth in section 6.2.1, is that these isolated peaks in the MR sweeps are most likely occur during a transition from some part of the sample from the superconducting state to the normal state.

**Figure 5.6:** SEM micrograph of SiO2Al122 showing one of the small rings of the network, as well as the line width near the ring on sample SiO2Al122.
Figure 5.7: MR measurements with $I_{\text{bias}} \leq 0.5 \mu\text{A}$. LP oscillations from the small loops do not appear and LP oscillations from the large loops appear at temperatures above 1.7 K. "u" denotes the "up" magnetic field sweeps, from negative to positive magnetic field strengths and "d" denotes "down" magnetic field sweeps, from positive to negative magnetic field strengths. Regular plateaus appear at lower temperatures, which could indicate that different parts of the leads are transitioning from a superconducting to the normal state.
Figure 5.8: MR measurements with $I_{\text{bias}} \geq 0.5 \mu\text{A}$. LP oscillations from the large loops are visible as well as other features that appear to be LP oscillations from the small loops. These features are however not periodic and—as we explore later—arise at NS phase boundaries causing enhanced sample resistances.
5.2.3 *Sample SiO$_2$Al$_{143}$*

The last of the etched samples we measured was SiO$_2$Al$_{143}$ with a small loop side length of 210 nm and a large loop side length of about 1700 nm. This was the first sample with which we resolved the bonding issues and were able to perform four-point measurements. Based on this geometry we would expect MR oscillations with a period of 44.9 mT (i.e. peaks at ±22.49 mT) from the small loops and 0.7 mT from the large loops. We indeed see MR oscillations, however only from the large loops, as shown in figure 5.10.

Because the LP effect predominantly occurs around $T_c$ of a sample, we would not expect to see LP oscillations at such low temperatures as 645 mK, the lowest measurement in figure 5.10. We would expect to see oscillations in most of the measurements shown in figure 5.11, however, closer inspection of this data shows that the sample transitions from the superconducting state to the normal state at fields lower than 22.49 mT. This means that the small loops in this sample were too small.

We also extracted the temperature information from the MR data and compared it to data from a temperature sweep, shown in figure 5.12. The measurements give us two different $T_c$s for the sample. However, because the temperature is held constant (within a few mK at most) during a MR measurement, any skewing of the data due to thermal lag, as happens during a temperature sweep, is negated. Thus $T_c$ for this sample is probably close to 1.16 K, which is slightly below that of bulk aluminum. This is a surprising result as we usually expect the critical values of thin film superconductors to increase.
Figure 5.10: All MR measurements on sample SiO$_2$Al$_{143}$. Based on the geometry one would expect LP oscillation peaks just above ±20 mT as the temperature approaches the sample’s $T_c$. Unfortunately the sample transitions into the normal state at lower field strengths, preventing the observation of these peaks.

Figure 5.11: The MR measurements on sample SiO$_2$Al$_{143}$. Despite not being able to see the LP oscillations from the small loops, the LP oscillations from the large loops are visible.
Figure 5.12: Comparison of $R$-versus-$T$ measurement taken during the condensation of helium-3 and temperature data extracted from MR measurements at zero field. The curve recorded during helium-3 condensation is shifted to higher temperatures because of the thermal lag in the system.
Figure 5.13: SEM micrographs of sample LOT12. a) Zoomed in view of a small loop in the network. b) Zoomed in view of a large loop in the network.

5.2.4 Sample LOT12

The first lift-off sample and final testing sample was LOT12 with small loop side lengths of 545 nm and large loop side length of about 1800 nm.

This sample was also measured in a four-point configuration, however, a constant background resistance was always present in all of the measurement, indicating that some part along the sample circuit never became superconducting. Initial measurements with the sample showed a normal resistance of 277 Ω and a superconducting resistance of 227 Ω, as shown in figure 5.14a. Somehow after the sample warmed up to about 9 K, the resistance of the sample changed from 277 Ω to 1750 Ω in the normal state and from 227 Ω to about 1575 Ω in the superconducting state. The resistance values for the rest of the measurement are shown in figure 5.14b. We speculate that the sample further oxidized when it warmed up slightly, which led to this drastic increase in the resistance. The IV characteristics, measured in a four-point configuration at low temperature before the change, indicate that the sample was never in completely in the superconducting regime in the first place. The finite slopes in the IV characteristic in figure 5.15 and figure 5.16 proves this.
Figure 5.14: Temperature sweeps both before (a) and after (b) the jump in sample resistance. The jump occurred after the sample warmed to 9 K and is probably due to further oxidation of the sample at the elevated temperature.
Figure 5.15: LOT12 IVs taken after the resistance jump at low temperatures with a relatively wide range. We see that at intermediate bias currents, from about 1 µA to 4 µA, that the resistance of the sample is about 1600 Ω, at the lower of the resistances in figure 5.14b but not quite the lowest. At higher currents resistance jumps to about 1750 Ω, which is the resistance of the sample when it is completely in the normal state. The slope at lower currents is explored in figure 5.16. The conclusion we can reach from this plot alone is that the sample never completely becomes superconducting.

Despite the entire sample not becoming superconducting, we still see LP oscillations from both the small and large loops as shown in the plots in figure 5.17 and figure 5.18. As the LP effect is dominant close to \( T_c \), we do not expect to see oscillations in figure 5.17 as measurements were only taken at low and high temperatures. However, similar to other MR measurements on other samples, especially later ones, there are low amplitude LP oscillations during the transition from the superconducting to the normal state.

In figure 5.18 more measurements are made closer to \( T_c \) of the sample. We see that the amplitude of the oscillations is very small at low temperatures and increases as the temperature approached \( T_c \), in accordance with the LP effect. However, without measurements covering the entire temperature range it is impossible to perform a complete analysis. One must also keep in mind that some unidentified part of the sample does not enter the superconducting state, resulting in the very high resistance. The lower part of the resistance range, similar to the large dip in figure 5.17 is cut off to highlight the oscillations.
Figure 5.16: LOT12 IVs taken after the resistance jump at low temperatures with a relatively wide range. A linear fit to the data at very low bias currents shows a resistance of 1390 Ω. This is lower than the lowest in figure 5.14b but still not superconducting.
Figure 5.17: MR measurements on sample LOT\textsubscript{12} before the resistance of the sample changed. At the low and high temperatures at which these measurements were taken we do not expect to see large LP oscillations. On the flanks of the lower temperature measurements there are small amplitude oscillations with a period of approximately $\Phi_0$, which indicate that these may indeed be LP oscillations. More measurements would be needed for a more complete analysis.
Figure 5.18: MR measurements on sample LOT12 after the resistance of the sample changed. We see LP-like behavior in the sample as small oscillations begin at low temperatures and increase in amplitude as the temperature approaches $T_c$. 
Figure 5.19: SEM micrographs of sample omri01. The sample was designed with five different networks in series, a bias current would pass through all of them and the voltage drop would be measured across each network independently. One of the bonding pads for the current was damaged so the current had to be routed through a voltage bonding pad. SEM micrographs of the three networks through which the current passed can be seen in the insets and their location in the circuit is indicated by the green lines. A schematic of the configuration can be seen in figure 5.20.

5.3 NETWORKS IN SERIES

Another possible sample geometry we explored was placing multiple networks in series so that we could simultaneously measure many networks at once and greatly reducing the measurement time needed. This was inspired by the work of Sochnikov et al., who used it to place multiple high-$T_c$ networks in series.

The only sample with this type of configuration was omri01 and was fabricated using the etching technique. The idea of this design was to pass a bias current through the two contact pads at either end of the sample, as shown in figure 5.19, and use the six remaining contact pads to measure the voltage across each individual network. For clarity a simple schematic of the networks and measurement leads is shown in figure 5.20. Unfortunately, as with many of the etched samples, problems with bonding resulted in the current pad on the far right not working. To solve this problem we measured the sample using a three-point configuration with the upper contact pad serving double duty as a voltage and current contact. The current
Figure 5.20: Schematic of how the current was sourced through the networks of omri01 and where the voltage measurement was taken. Listed in each block are the small loop and large loop side lengths for each network.

was sourced through the three networks on the right and the voltage drop measured only over the middle-most network.

A slow temperature sweep was performed to try and determine the transition temperature of the sample, as shown in figure 5.21. Even at the lowest temperatures we could achieve with our cryostat, it is not clear that the sample completely enters the superconducting state, or if the resistance is still dropping. There is a slight drop in the resistance of the sample at 1.07 K, which could be attributed to the 50µm-thick Al bonding wires used to contact the sample. Otherwise the larger transition, which we attribute to the larger structure of the network, does not begin until the low temperature of 770 mK, which is very low for nanostructured Al.

Figure 5.21: Resistance of the sample as a function of sample temperature. One trace is from the initial cool-down of the sample. The second is from a temperature sweep during which the sample is heated for a short time and allowed to cool back down.
Figure 5.22: MR measurements on sample omri01. Visible are what appears to be one period of the LP oscillations caused by the smallest loops in all of the networks, namely the 90 nm loops, the peak locations of which are highlighted by the black dashed lines. Many other oscillations with smaller periods are also visible, these are discussed below.
After the sample was cooled to a base temperature of 270 mK, MR measurements were started at 300 mK and at higher temperatures in 100 mK intervals until 700 mK. These measurements are shown in figure 5.22. Because we were trying to measure the 250 nm loop network the calculations for the flux occupation are scaled to this loop size:

\[ n = \frac{\Phi_{250}}{\Phi_0} = \frac{(250 \text{ nm})^2}{\Phi_0} \cdot B. \]

The most striking feature is the large-period oscillation seen in the magnetic field sweeps at 300 mK, 350 mK and even 450 mK. This is surprising to see, because it is completely unlike previous samples with loop sizes less than 300 nm, where the oscillations from the smallest loops are not present in the MR data. We can attribute these large oscillations here to the 90 nm loops along the current path. These loops give rise to one single oscillation with a period of about 255 mT, which is highlighted by the black dashed line in figure 5.22.

There are more oscillations in the signal and zooming in closer, as shown in figure 5.23 and figure 5.24, we see, in addition to the oscillations caused by the 250 nm loops, the oscillations from the 500 nm loops with a period of 8.3 mT, from the 1100 nm loops with a period of 1.7 mT, from the 1500 nm loops with a period of 1.0 mT and possibly even the 2800 nm loops with a period of about 0.25 mT.

Looking at high frequency oscillations at the different temperatures we see a temperature dependence. At low temperatures the oscillations from the 1500 nm loops are the dominant feature and the oscillations from the 2800 nm loops are also clearly visible. As the temperature rises these 1 mT oscillations weaken and disappear. Above 600 mK the oscillations from the 1100 nm loops begin to dominate. This tells us that at different temperatures the different networks in series have stronger or weaker long range coherence of the superconducting condensate than their neighbors. In other words, the different networks have slightly different \( T_c \)s.

It would be instructive to perform a Fourier analysis on these measurements to try and pull apart all of the oscillations seen in the signal. However, one must keep in mind that the slight temperature instability caused by the magnetic field dependence of the thermometer leads to an asymmetrical MR measurement about zero field at these low temperatures. This is visible in some of the sweeps at higher temperatures in figure 5.22, but also made clear on the example of the 450 mT sweep, shown in figure 5.25.

To perform an FFT analysis one must remove the background of the signal without shifting the positions of the oscillations. We use a simple two-degree polynomial to fit the background on the measurement inside of the 90 nm-loop oscillations, i.e. between −127 mT and 127 mT, and subtract this from the signal. We expect simple quadratic behavior as, according to Little and Parks, the change in the resistance is proportional to the change in \( T_c \), which also has a quadratic background or envelope in a magnetic field. Subtraction of a quadratic background does not change the positions of the peaks, as demonstrated on the example of the 300 mK measurement in figure 5.26.

The results of the Fourier analysis on the background-reduced signals of all measurements are shown in figure 5.27a and figure 5.27b. Figure 5.27a shows a broader range of oscillations with a very strong signal at about 33 and another strong signal at lower values, which figure 5.27b shows in detail. The peak at 33 corresponds to the 1 mT oscillations from the 1500 nm loops, the signal from which is very strong in the 300 mK measurement. We would expect to see a signal near 20 in the 700 mK sweep as here the oscillations from the 1100 nm loops dominate on the small scale. We do not see this because the background removal on the
measurements taken at temperatures above 450 mK tends to add artifacts due to the asymmetry caused by the magnetic field dependent behavior of the thermometer. Looking at the slower oscillations, with a period below 5, as shown in figure 5.27b, we see a strong signal at 1 that decreases with increasing temperature, corresponding to the 250 nm loops. We also see a signal at 0.5 that, while it varies with temperature, is present at all temperatures. This is again most likely an artifact from the estimation and removal of the parabolic background. If we were to see $h/e$ oscillations, in this sample they would more likely be associated with the 89 nm loops, thus having a much longer period, appearing at a lower value in the plot. A summary of the results is located in table 5.2 at the end of this chapter. The network design sizes and expected oscillation periods are listed there. For comparison the same information is given for the network sizes determined from the SEM images. Finally, oscillation periods determined from the MR measurements are listed and the network sizes calculated from this information.

In conclusion, although we measure the voltage drop across one network, we see a modulation or signal in the current caused by each network through which we pass the current. We have a temperature and network size-dependent signal. In addition, according to the FFT anal-
Figure 5.24: MR measurements of sample omri1 zoomed in to show the fastest oscillations. At this level of detail we can now see that the period of the fastest oscillations changes with increasing temperature. At the lowest temperature there 1 mT oscillations from the 1500 nm and even smaller oscillations from the 2800 nm loops. These oscillations slowly disappear as the temperature rises and are replaced by oscillations from the 1100 nm loops.
Figure 5.25: Illustration of the temperature instability during a MR measurement at 450 mK. The difference in peak height is slight, yet apparent between the peaks at negative magnetic field strengths and the peaks at positive magnetic field strengths.

ysis, we do see an oscillation with a $h/e$ periodicity. However, we cannot rule out other confounding effects in this sample.

One reason why we see long range effects of different networks when measuring only one and Sochnikov et al. does not is that in the low-$T_c$ superconducting Al we have much longer coherence lengths and the effects from one network can persist over a finite distance into a neighboring network or structure.
Figure 5.26: Comparison of the original MR measurement on omrio1 at 450 mK to the background-reduced sweep. The background was fit using a simple quadratic function. Removing the background does not shift the peak positions. The dashed red lines show that the peak positions remain the same.
Figure 5.27: FFTs of the MR measurements at different temperatures. The plots for each temperature are shifted for clarity. Each plot is normed such that the variation in magnitude of the power spectra can be seen and that they all fit in the visible area of the plot. a) Looking at a broad range of frequencies, we see a very strong peak at about 33, which corresponds to an oscillation period of 1 mT or $0.03\Phi_{250\,\text{nm}}/\Phi_0$. This is the signal from the 1500 nm loops, which were paired with the smaller 500 nm loops. Because we were measuring the 250 nm loops, paired with the large 2800 nm loops in the same network, we would expect to see a strong signal from these near 120. This is not shown, but there are no more significant frequency peaks beyond the harmonic of the 33 peak. b) The narrower view of the frequencies shows a very strong signal at 1, which is from the 250 nm loops and is as expected.
Table 5.2: Summary of loop sizes and expected magnetic field periodicities they give rise to. Because the network we were trying to measure had small loops with side lengths of 250 nm, this is the basis for all calculations. In the first column the side lengths of the different loops are listed in network pairs, for example the smallest 90 nm loops are together in a network with the 1100 nm loops, the 250 nm loops with the 2800 nm loops, and the 500 nm loops with the 1500 nm loops. The second column lists the ratio of each loop area compared to the area of the 250 nm loop. The third column lists the expected and then measured oscillation period (peak-to-peak) of the magnetoresistance oscillations associated with each loop size. The fourth and final column converts the magnetic field oscillation period into the difference in magnetic flux quanta, based on the flux through the 250 nm loops.
5.4 CONCLUSIONS ON SAMPLE DESIGN

After testing different sample design strategies we were able to solve many of the problems we encountered. One of the more serious problems that had to be addressed was to allow for reliable bonding and connection of the sample to the measurement electronics so we could perform four-point measurements. This was achieved by adding a final evaporation step in which large gold contact pads were evaporated through a mechanical mask onto the aluminum leads. We also found that the lift-off samples performed just as well as the etched samples with the advantage of having a much faster fabrication process. Finally, we also found that with aluminum samples we cannot measure networks in series as the extremely long-range order of the aluminum causes the different networks to interact with one another and we see signals from all networks through which a current flows.
After taking the lessons learned from the previous samples and the experiments with the thermometry, we were able to successfully characterize two lift-off samples close to zero bias and the second with a dc current bias of 2.1 µA.

In the following chapter we will first look at the temperature characterization measurements, the IV characteristics and the MR measurements of sample LOS001, a lift-off sample with a small loop side length of just over 400 nm. Following this we will look at the same measurements for sample LOS003, which had a small loop side length just over 300 nm, and the phase boundary measurement we performed on this sample.

6.1 Sample LOS001

This sample was designed with small loops of 400 nm, large loops of 1750 nm and a line width of 50 nm. The actual parameters of the sample were close to the design parameters, with a small loop side length about 430 nm, a large loop side length of 1700 nm and an average line width just under 50 nm. SEM images of the large and small loops are shown in figure 6.1a and figure 6.1b respectively.

To determine the transition temperature we looked at the data collected during each condensation cycle of the helium-3 as well as temperature sweeps. The faster measurements show a $T_c$ ranging from 1.30 K to 1.36 K and the slow measurement shows a $T_c$ closer to 1.43 K, which is closer to the value obtained by extracting temperature data from the MR measurements. Note that the looping behavior of the temperature sweep giving us the 1.432 K estimate for the $T_c$ is due to instability in the temperature control, leading to large temperature oscillations, which—in combination with the poor thermal coupling between the sample, thermometer and heater—leads to loops in the $R$-versus-$T$ plot.
Figure 6.2: Measurements to find $T_c$ of LOS001. Fast sweeps show a $T_c$ ranging from 1.300 K to 1.360 K. A slower, more controlled sweep resulted in a much higher $T_c$, near 1.43 K, whereas the temperature data extracted from the MR measurements gives us a $T_c$ of 1.408 K.
The MR measurements show oscillations from both large loops, with a period of 0.7 mT and the small loops, with a period of about 11 mT. The maximum oscillation amplitude occurs in the sweep at 1408 mK, essentially the sample’s $T_c$, and is 40% of the sample’s resistance, or $\Delta dR \approx 14 \Omega$. The dashed red line in figure 6.4 show the lower and upper bounds of the largest oscillation. The oscillations from the small loops start off at very low temperatures with a rounded form that develops into an upward cusp. This upward cusp behavior is expected for decoupled loops as described by Sochnikov et al. Starting near the sample’s $T_c$ these peaks become more rounded again, displaying an almost sine-like form at the highest measurement temperatures. It could be that this is the midpoint of a slow progression from upward-pointing cusps of decoupled loops to loops exhibiting coupled behavior and downward-pointing cusps, as discussed in section 3.4. Another possible explanation for the changing of the oscillation shape may be that the loops start act like SQUIDs at higher temperatures and fields. The signature SQUID-like behavior is downward pointing cusps in MR measurements. The transition to this behavior would be gradual, first one would have sharp upward peaks in the low field and temperature MR measurements that would turn to sine-like oscillations at intermediate fields and temperatures. Finally, at higher fields and temperatures one would see pronounced downward peaks in the MR measurements [93]. We do not see the pronounced downward peaks at the samples become normal conducting first.

Figure 6.3: MR measurements of sample LOS001 at different temperatures. These measurements were conducted by sweeping the external magnetic field from a negative value, e.g. $-300$ mT for the measurements at the lowest temperatures, to a positive value, e.g. 300 mT.
Figure 6.4: The same MR measurements but zoomed in to a smaller magnetic field range to highlight the higher frequency oscillations caused by the large loops in the network. The dashed lines indicate the lower and upper bounds of the largest oscillation, which was recorded in the 1408 mK MR measurement. The magnitude of this oscillation is $\Delta dR = 14 \Omega$, about 40% of the sample’s normal state resistance.
An FFT analysis of these sweeps is presented in figure 6.6. The surprising result is that at low temperatures the oscillations have twice the period, corresponding to \( h/e \) oscillations, of what would be expected from the small loops. This is most likely an artifact from the background fitting and removal. For this sample we fit the background to a 12-degree polynomial, which near \( T_c \) delivers good results, but adds long oscillations to the background reduced signal before FFT is performed. An example of this can be seen in figure 6.5. We see in the lower panels that after estimating and removing the background we are left with a signal that clearly has an oscillation period much longer than \( 1 \cdot \Phi_0 \). There is no indication in the upper panels that there oscillations with this long period. These long oscillations have a maximum amplitude in the lower panels less than 0.2 \( \Omega \), which is less than 0.6% of the samples normal resistance. Thus these oscillations, although they have a very small amplitude compared to the sample's normal resistance, still show up in the FFT analysis and are most likely an artifact.

Above about 1.0 K these high-order polynomials begin to deliver background estimates that, at least to the eye, very closely follow what one assumes is the background of the measurement, without adding additional oscillations. Comparing the negative and positive halves of the MR measurements with the entire measurement in figure 6.6, we see similar behavior in all three analyses. The peaks in the FFT analysis of the entire sweep are located at the same flux values as in the FFT analyses of the negative and positive branches, but much larger. This is consistent with the increase in the amount of information when taking the entire sweep into account as opposed to only half of a sweep. Aside from the signal at extremely low temperatures, the behavior seen here is in agreement with what we would expect from the LP effect. We see the largest oscillations near \( T_c \) of the sample. Although the FFT cannot quantitatively determine
the oscillations’ amplitudes, we can see in figure 6.4 that the oscillation amplitudes reach a maximum of about $15\, \Omega$ at 1408 mK.
As demonstrated in section 4.3.2.2, we can extract the normal-superconducting phase boundary from MR measurements. The temperature steps in-between measurements for this sample were unfortunately too large to see the LP oscillations in $T_c$ from the small loops in the network. We do, however, have the parabolic envelope upon which the LP oscillations are usually superimposed. Using equation (3.6) to fit to the data, we gain valuable information about the sample. Using the $T_c$ from the MR measurement temperature characteristics ($T_c = 1.41$ K) the line thickness from the SEM images ($d = 50$ nm) and a coherence length guess of 120 nm gives us the parameters and envelope seen in figure 6.7. Using 136 nm as an initial guess for the coherence length, as estimated from mean free path calculations for the sample (see table 5.1), results in a line width of only 30 nm, which is much smaller than what we see in the SEM images, and a coherence length of 173 nm which is much larger than previous values reported in literature [92]. We have no reason to assume that our thin aluminum films are much cleaner than in previous reports. Thus we find that because the $T_c$ and $d$ fitted results agree so well with our measurements that a resulting coherence length of 100 nm is quite plausible, even if it deviates greatly from the value we determined from resistivity measurements.

![Figure 6.7: The LOS001 phase boundary extracted from the MR measurements and a fit to the data using equation (3.6). The temperature steps were unfortunately not close enough together to allow us to resolve the LP oscillations from the small loops in the network.](image-url)
Because we were measuring the differential resistance for MR measurements, we had to take care that we were not measuring with a bias current that was on an exotic branch in the IV landscape. To check this we recorded the IV characteristics at similar temperatures for which we took MR measurements. These data are shown in figure 6.8. We see that even at high temperatures, the critical current is well above the 0.5 µA maximum bias current that we used in most of our measurements. Figure 6.9 shows the IV data recorded during each MR measurement shown in figure 6.3. We see that, with the exception of one of the measurements recorded at very low temperature, most of the measurements were performed at bias currents much lower than the temperature dependent critical currents.

**Figure 6.8:** IV-characteristic measurements taken at similar temperatures as the MR measurements. Even at elevated temperatures as high as 1566 mK, the critical current is still above 1.0 µA, the maximum bias current we used in our measurements. See figure 6.9 for IV data from the MR measurements.
Figure 6.9: The IV behavior of sample LOS001 during each MR measurement. The data for each temperature is from the MR measurement at that temperature. For most of the measurements $I_{bias} < 0.4 \, \mu A$, for one measurement at a low temperature the bias current was slightly higher, $I_{bias} < 1.0 \, \mu A$. These values are still well below the temperature depended $I_c$ for the sample.
6.2 SAMPLE LOS003

This sample was designed with small loops of 300 nm and large loops with a side length of about 1575 nm. As figure 6.10 shows, the actual sample had small loop side lengths of 324 nm and average large loop side lengths of 1550 nm and a line width of about 60 nm.

Figure 6.10: SEM image of LOS003 with loop size and line width measurements.

This sample was cooled down and measured a number of times. During the first cool-down we applied a bias current 2.1 µA which lead to resistance enhancement, as will be presented section 6.2.1. A subsequent measurements series was performed at a zero bias and a much tighter temperature distribution of the MR measurements. These data are presented in section 6.2.2.
6.2.1 Measurements With High Bias Currents

Previous work had shown that one explanation for resistance anomalies, where the resistance during a superconducting transition can exceed the normal resistance of the sample, is that a high bias current can cause the nucleation of quantum phase slip centers (QPCs) in the superconductor \cite{92}. We explored this on sample LOS003.

The $R$-versus-$T$ behavior, is shown in figure \ref{fig:6.11}. As we saw with earlier samples, the faster measurements lead to a spread in results for $T_c$, nonetheless the average from these measurements is 1.31 K and is highlighted by the red vertical line in figure \ref{fig:6.11} This average is very close to the value we get when we extracted the temperature data from the MR measurement data, which is 1.315 K. What is different here is that before the sample transitions from normal state to the superconducting state, its resistance increases above its normal resistance. This confirms that, in this case, a high bias current leads to an anomalous resistance.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure6.11.png}
\caption{Resistance versus temperature measurements to find $T_c$ for LOS003 with a high bias current of 2 $\mu$A. As compared the the measurements taken at zero bias current we see a suppression of $T_c$ and resistance spike above the sample’s normal resistance indicating the dominance of a quasi-particle current at QPCs during the NS phase transition.}
\end{figure}
The MR measurements recorded for this sample with a bias current of 2 µA are shown in figure 6.12, and the behavior at low fields is highlighted in figure 6.13. As with the $R$-versus-$T$ measurements we see here enhanced resistance just before the sample transitions from the normal to the superconducting state. It would also seem that the oscillation amplitudes are also enhanced, as exhibited by the measurement at 1251 mK. This has an amplitude just below $2\Phi_0$ of over $10 \Omega$. This is larger than the amplitudes seen at similar temperatures on LOS001, a similar sample with a similar normal resistance. Examining the behavior at lower magnetic field strengths, shown in figure 6.14, we see that the anomalous behavior is not limited to an enhanced amplitude of the LP peaks at half-integral quantum flux values. We see that the features centered around $\pm1\Phi_0$ transition from troughs at intermediate temperatures between 1290 mK to 1307 mK to ridges above the sample’s $R_N$ with the short range oscillations of the large loops superimposed on this. Similar to this, a small ridge begins to appear in the trough centered around zero field at 1311 mK. This could indicate that at higher bias currents and as the temperature rises, a suppression of $T_c$ occurs that is counteracted at very small finite fields, beyond which the LP-like behavior returns. Figure 6.14 shows the MR measurements at low fields and shifted to more clearly depict the behavior of each individual measurement. It is difficult to construct a complete picture because we only have the first peak at low fields, where $A \cdot B = \frac{n}{2}\Phi_0$, on either side of the zero field, but it seems as though this peak, which appears at 1290 mK, is split into two peaks as the temperature rises and these peaks spread apart from one-another as the temperature increases.

This enhanced resistivity is nothing new, this has been seen before and discussed at length by Strunk et al.[92]. What is new is how this enhanced resistance arises in conjunction with LP oscillations. As the magnetic field approaches a strength corresponding to half-integer flux occupation of the small loops, $A \cdot B = \frac{2}{2}\Phi_0$, which is where the oscillations peaks occur in the ordinary LP effect, the sample begins to enter the NS phase transition. It is here that likelihood of QPSs increases and as the superconducting order parameter breaks down, the current is dominated by quasi-particles, giving us the enhanced resistance. Below a certain critical temperature (not $T_c$ of the sample), the LP oscillations are not strong enough to completely return the sample to normal state near magnetic field strengths that satisfy $A \cdot B = \frac{n}{2}\Phi_0$. In this case we simply see enhanced oscillations with larger amplitudes than for the ordinary LP effect. As the temperature increases the sample is driven to the normal state at $\frac{n}{2}\Phi_0$, thus the quasi-particle dominated current disappears and a normal current returns, restoring the resistance to the sample’s normal resistance. As the temperature increases the sample is driven normal further and further away from $\frac{n}{2}\Phi_0$, making the region where the sample has the normal resistance seem to expand, giving the appearance of a widening trough between the enhanced resistance spikes. At the same time this is going on in the small loops, the ordinary LP behavior of the large loops remains undisturbed where the magnetic field satisfies $A \cdot B = n\Phi_0$. The LP oscillations are just superimposed on the quasi-particle dominated current in these regions.
Figure 6.12: MR measurements taken at a finite bias current of 2 µA. The large bias current leads to resistance enhancement during transitions between the normal and superconducting state of the sample. We see in the MR measurements at all temperatures resistance enhancement above the sample’s normal resistance. These are caused by a quasi-particle-dominated current at NS phase boundaries that appear during the NS phase transition.
Figure 6.13: MR measurements taken at a finite bias current of 2 µA, highlighting the behavior at low fields. At this level of magnification of the small, positive magnetic field strengths we clearly see the enhancement of the resistance during the NS phase transition above the sample’s normal resistance.
Figure 6.14: MR measurements taken at a finite bias current of 2 µA, highlighting the behavior at low fields. The measurements are shifted up for clarity. This view of the data highlights the appearance of a "trough" at half-integer values of flux between the resistance enhancement peaks.
Finally, we were able to extract the phase boundary from this limited set of MR measurements, which is indicated by the blue dots in figure 6.15. The fit to the background indicates that the transition temperature is 1.32 K and the coherence length is 111 nm, both of which are plausible values and in close agreement with the $T_c$ ascertained from the temperature sweeps and the $\xi_{GL}$ obtained from the sample’s resistivity.

Figure 6.15: The phase boundary data for sample LOS003 extracted from the MR measurements taken at 2 $\mu$A. The data is then fit using equation (3.6). We see that despite having anomalous peaks, the phase boundary data extracted from the MR measurements follow the same model behavior as earlier samples. We see near $T_c$ smaller parabolas superimposed on a background parabola. Unfortunately the measurement density was too low to resolve the oscillations caused by the small loops, let alone the large loops.
Figure 6.16: An IV sweep measurement taken at low temperature to find IV-characteristics of LOS003. A fit to the ohmic behavior of the IV characteristics reveals a normal resistance of 22.6 Ω, about 1 % below 23.3 Ω determined from the temperature sweeps.

6.2.2 MR Measurements at Zero Bias

Finally, we measured the same sample again at zero current bias. With this sample we recorded slow temperature sweeps at zero field and at specific magnetic field strengths, shown in figure 4.86. The $R$-versus-$T$ behavior does not show anomalous resistance spikes above $R_N$ at any magnetic field. They also indicate that the transition temperature at zero field is about 1.46 K, which is much higher than the value from the phase boundary fit on the high current data. This is not surprising as it is known that among the three critical parameters, namely temperature, current and external magnetic field, that raising one will lower the others.

The IV characteristics show no unexpected behavior with a slope of 22.6 which differs by about 1 % from the measured differential resistance in the normal state of 22.8 Ω.
The MR measurements of this sample at zero current bias are shown with the full magnetic field range in figure 6.17 and a narrower magnetic field selection in figure 6.18. In these measurements at zero bias the resistance anomaly is not present. We see the normal LP oscillations for both the large and small loops. As described by Sochnikov et al., we expect sharp upward-pointing cusps for the decoupled small loops and sharp downward-pointing cusps for the coupled large loops, which has to do with the fact that the small loops exhibit a step-wise increase in flux occupation as the magnetic field increases and the large loops exhibit a linear increase.
Figure 6.18: These are the same measurements as shown in figure 6.17 but at a scale selected to highlight the LP oscillations of the 324 nm loops. We see a smearing out or rounding of the large peaks as the temperature increases. Also clearly visible are the LP oscillation from the large loops.
The periods of the different oscillations, however, match with the LP effect for both the large and small loops. This is demonstrated in figure 6.19 which shows periods below $1 \cdot \Phi_0$ at low temperatures and periods centered about $1 \cdot \Phi_0$ near $T_c$ with the amplitude increasing as the temperature approaches $T_c$ from below and falling off quickly above $T_c$. The peak at low temperatures is most likely an artifact of the background estimation and removal. This is demonstrated by the fact that when using polynomials of even degree between 2 to 12 to fit and remove the background the peak centered around $1 \cdot \Phi_0$ stays more or less in place with comparable amplitudes across all different background estimations. The peak at low temperatures, however, shifts to lower and higher values with an amplitude that varies non-linearly with the choice of background polynomial estimation.

Figure 6.19: FFT analysis of the MR measurements on sample LOS003. The MR measurements above 1460 mK were omitted because there were many of them that showed no periods at all, just like the 1455 mK and 1460 mK lines in this plot.
Finally, we were able to perform a direct measurement of the $B$-versus-$T_c$ phase boundary, which is shown in figure 6.20. Fitting to the background using equation (3.6) we get an agreement for the $\xi$ and $T_c$ from the resistivity measurements and the critical temperature determined from the temperature data in the MR measurements. In this direct measurement of the phase boundary we also have a good enough resolution to resolve the small oscillations from the large loops.

**Figure 6.20**: Direct measurement of $T_c$. During this measurement the PID was set to keep the resistance of the sample, $dR$ at the 12.0 $\Omega$, which is about half of the normal resistance, or the resistance of the sample at its $T_c$. 

\[
\text{differential Resistance } [\Omega] = (324 \text{ nm})^2 \frac{B}{\Phi_0}
\]
The measurements presented here are the results of our efforts at studying nanostructured superconducting aluminum devices. We performed magnetoresistance measurements on double network samples with loop sizes ranging from 90 nm to 500 nm. We showed in section 4.3.2.1 that we could extract the temperature-dependent behavior of the sample from the magnetoresistance measurements given that they were taken at small enough differences from one another to deliver sufficient temperature resolution. Further we showed in section 4.3.2.2 that we could also extract the phase boundary behavior of the sample from the magnetoresistance measurements. The important factor was again the temperature resolution of the data set. With lower temperature resolution only the background of the phase boundary could be reconstructed and with a higher resolution the LP oscillations from the smaller loops could also be reconstructed. The resolution was not high enough to see the small LP oscillations from the large loops.

Our goal at the outset of this project was to find oscillations with twice the LP oscillation period of $h/2e$, namely periods of $h/e$. To achieve this we wanted to fabricate samples where the BCS coherence length, or size of the Cooper pair, was larger than the circumference of the loop. Aluminum was a perfect candidate for such an investigation as it has a BCS coherence length of 1600 nm. We unfortunately were not able to overcome the limitation imposed by nanostructuring thermally evaporated samples, namely that the small structures and disordered nature of the superconducting films out of which our sample were made limit the coherence length to at least an order of magnitude smaller than that of bulk samples.

In a sample with multiple networks placed in series we found that a network not being directly measured could still affect the magnetoresistance behavior of a neighboring network that was being measured. This was made possible by the fact that aluminum has such a long coherence length, even when reduced in nanostructured devices. We also found a temperature dependence to this long range effect. Of the three large loops sizes measured, the larger ones, 2800 nm and 1500 nm dominated the fine structure of the magnetoresistance behavior at low temperatures and the smallest of these loop sizes, 1100 nm, dominated at higher temperatures.

Finally, we found anomalous resistance peaks and oscillations in the $R$-versus-$T$ and $R$-versus-$B$ field behavior caused by a high bias current. The resistance peaks, or enhanced resistance, where the resistance during a phase transition rises above the normal resistance of the sample is seen in both $R$-versus-$T$ and $R$-versus-$B$ behavior. In literature this is understood to be caused by quantum phase slip centers and the quasi-particle charge imbalances they create. The anomalous oscillations were unlike anything found in literature, as there was a curious temperature dependence to the behavior. At low temperatures the behavior of the system was the same as the expected LP effect. However, as the temperature rose two fundamental differences to the LP effect arose. First the peak positions seemed to change. It was as though the LP peaks at low temperatures were split in the middle as the temperature rose and these two new peaks moved away from one another. The second effect was that the troughs between peaks became bulges, eventually increasing above the normal resistance of the sample. We interpreted this seeming suppression of the peak to really be the sample fully entering the normal state, starting at half-integer flux values, and thus the resistance returning to it’s normal state value.
The NS phase transition regions are then squeezed together centered on the integral flux values. The smaller period oscillations cause by the large network loops are superimposed on resistance signal from the smaller loops.

The search for $h/e$ and $h/4e$ oscillations will still continue and aluminum double networks are a promising system for finding these oscillations. However, fine tuning of sample parameters is now required. The coherence length needs to be increased, keeping in mind that there is a trade-off between coherence length and critical field.
Wir haben Magnetowiderstandsmessungen an sogenannten Doppel-Netzwerken aus Aluminium mit Größen von 90 nm bis 500 nm durchgeführt. In section 4 haben wir gezeigt, dass wir das temperaturabhängige Verhalten der Proben aus den Magnetowiderstandsmessungen extrahieren konnten, solange die Messungen mit ausreichender Temperaturauflösung durchgeführt wurden. Weiterhin haben wir in section 4.3.2.2 gezeigt, dass wir die Phasengrenze Normalleiter und Supraleiter von den Magnetowiderstandsmessungen extrahieren konnten. Mit niedriger Temperaturauflösung konnten wir nur den parabolischen Hintergrund aus den Magnetowiderstandsmessdaten entnehmen und mit höherer Auflösung konnten wir die Little-Parks Oszillationen der kleinen Netzwerkschleifen aus den Magnetowiderstandsmessdaten entnehmen.

Unser Ziel war es die doppelte Periodizität der normalen Little-Parks Oszillationen zu finden, nämlich diejenige mit einer Periodizität von $h/e$, statt die normale Little-Parks-Periodizität von $h/2e$. Um dies zu erreichen, mussten wir Proben in der Größenordnung der BCS-Kohärenzlänge herstellen. Aluminium war hierfür der perfekte Kandidat, weil es eine Kohärenzlänge von 1600 nm in Bulk hat. Leider hatten wir das gleiche Problem, das bei anderen Gruppen auch aufgetaucht ist. In nanostrukturiertem Form ist die Kohärenzlänge von Aluminium um eine Größenordnung kleiner als im Bulk.

Wir haben eine Probe mit mehreren Netzwerken in Serie hergestellt und gemessen. Wir fanden, dass die benachbarten Netzwerke einen Einfluss auf das zu messende Netzwerk haben, obwohl sie nicht direkt gemessen worden sind. Das liegt daran, dass Aluminium, trotz Nanostrukturierung, immer noch eine sehr lange Kohärenzlänge hat, die den Einfluss der einzelnen Netzwerke weit über ihren Grenzen projizieren konnte.


BIBLIOGRAPHY


In ordinary life we hardly realize that we receive a great deal more than we give, and that it is only with gratitude that life becomes rich.
— Dietrich Bohnoeffe

At times our own light goes out and is rekindled by a spark from another person. Each of us has cause to think with deep gratitude of those who have lighted the flame within us.
— Albert Schweitzer

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