

## Experiment of the Century: Fabrication and Measurement of the Bimetallic Superconducting Loop with an Andreev Interferometer

A thesis for the degree Bachelor of Science in Engineering Physics

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# Preface

We would like to thank Leonid Kuzmin and Sumedh Hemant Mahashabde for their time and hard work to ensure that our thesis is of high quality. We would also like to thank the rest of the Bolometer group, Mikhail Tarasov, Ernst Otto and Natalia Kaurova, for their help and valuable discussions; Dmitri Golubev for the email correspondence, and our friends and families for their support and understanding during our work.

Claes would especially like to thank Petra for being as wise as she is beautiful.

The picture on the cover is a scanning electron microscope picture of the Andreev interferometer of sample L11-66. The normal metal is the faintest, followed by a layer of bright superconducting aluminium and finally a layer of aluminium evaporated in an oxidizing environment. The upper inlay shows modulation of the interferometer with injected thermoflux in the BL3-53 chip. The lower inlay shows the 35.6 % modulation of the interferometer's resistance, when a magnetic field is applied through the magnetic field loop.

#### Abstract

The experiment to measure the thermoelectric effect in a superconducting bimetallic loop was realised using an integrated mesoscopic circuit with an Andreev interferometer as readout. The aim was to apply nanofabrication technologies to this unsolved problem in physics. An additional goal was to examine the interaction between two bimetallic loops.

The Andreev interferometer had a normal resistance of  $R_N = 5.6 \text{ k}\Omega$ . The maximum resistance modulation was 35.6%. The interferometer displayed a quantized resistance close to the Sharvin resistance. Both the high modulation and the quantization are unexpected results.

To measure the thermoelectric effect, part of the loop was heated. Quasiparticles were injected through the Josephson junctions into the loop. A non-trivial effect was observed in the readout. With increasing current, the resistance decreased. In the loop interaction experiments, a part of the loop not connected to the readout was heated. The results from the loop interaction experiments are inconclusive.

We recommend that more samples are fabricated in order to repeat the result. The observed results should be explained theoretically as soon as possible. In future experiments, the induced temperature gradients must be measured.

#### Sammanfattning

Ett experiment för att mäta den termoelektriska effekten i en supraledande bimetallisk loop realiserades i form av an integrerad mesoskopisk krets, med en Andreevinterferometer. Målet var att använda nanofabrikationsteknologier på detta olösta problem i fysiken. Ytterligare ett mål var att undersöka växelverkan mellan två bimetalliska loopar.

Andreevinterferometern hade en normalresistans på  $R_N = 5.6 \,\mathrm{k}\Omega$ . Den maximala resistansmoduleringen var 35.6 %. Interferometern uppvisade en kvantiserad resistans, nära Sharvinresistansen. Både den höga moduleringen och kvantiseringen var oväntade resultat.

För att mäta den termoelektriska effekten, värmdes en del av loopen. Kvasipartiklar injicerades in i loopen genom Josephson-övergångarna. En icke-trivial effekt observerades i interferometern. Resistansen minskade med ökande ström. I växelverkansexperimenten värmdes en del av den loop som inte var ansluten till interferometern. Resultaten från växelverkansexperimenten är inte definitiva.

Vi rekommenderar att fler kretsar tillverkas, så att resultaten kan bekräftas. De observerade resultaten bör förklaras teoretiskt så fort som möjligt. I framtida experiment måste den inducerade temperaturgradienten mätas.

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## Nomenclature

	Symbols
Symbols	Description
е	Elementary charge, $1.60217646 \cdot 10^{-19}$ As
$\mu_0$	Magnetic constant, $1.25663706 \cdot 10^{-6} \mathrm{N/A^2}$
т	Electron mass, $9.10938188 \cdot 10^{-31}  \text{kg}$
$k_B$	Boltzmann's constant, $1.3806503 \cdot 10^{-23}$ J/K
$\hbar$	Planck's reduced constant = $h/(2\pi) = 1.05457148 \cdot 10^{-34}$ Js
$\epsilon_k$	Bloch energy compared to Fermi energy
$E_k$	Energy of a particle with wave number k
$k_F$	Fermi wave vector
H	Magnetic field
В	Magnetic field strength
Α	Magnetic vector potential
Q	Amount of charge
$\Delta(T)$	Energy gap
$\Delta_0$	Energy gap at 0 K
$n_s$	Electron density of superconductor
$n_{s}(0)$	Density of states at temperature $T = 0 \text{ K}$
σ	Spin
$\theta$	Superconductor phase
$\varphi$	The phase contribution from the Andreev reflection
Φ	Magnetic flux
j	Current
js	Supercurrent
jт	Thermoelectric current
$\eta_T$	A temperature dependent transport coefficient
ξ	Coherence length
λ	Penetration depth
κ	Quotient of penetration depth and coherence length

- Total quasiparticle population  $n_{><}$
- Amount of quasiparticles below the Fermi level  $n_{<}$
- Amount of quasiparticles above the Fermi level  $n_>$

Resistance of *SIS'*-junction when both turn to normal metal.  $R_{SIS'}$ 

- Interferometer normal resistance  $R_N$
- R Resistance
- S Superconductor
- Ν Normal metal
- Insulator Ι
- $T_c$ Critical temperature
- $H_c$ Critical magnetic field
- Interferometer arm length L
- Mean free path  $l_e$
- W Interferometer arm width
- I VCurrent-voltage characteristics

Abbreviations

	Tibble Tiddetib
Abbreviation	Description
BCS	Bardeen-Cooper-Schrieffer
PMMA	Polymethyl methacrylate
CAD	Computer Aided Design
SEM	Scanning Electron Microscope
DC	Direct Current
AC	Alternating Current
BL3 – 2	Bimetallic loop CAD design number 2
<i>BL</i> 3 – 3	Bimetallic loop CAD design number 3

## Chapter 1

# Introduction

This bachelor thesis covers the fabrication and measurements of an experimental setup with two bimetallic superconducting loops and an Andreev interferometer. The background and a brief theory are introduced in the following section to prepare for the statement of purpose. The project scope and delimiters makes the problem more clearly defined, leading up to the approach to the problem. The chapter also includes reading instructions to guide the reader through the thesis.

### 1.1 Background

The initial discovery of superconductivity was at the beginning of the 20th century. The principal investigator Kamerlingh Onnes envisioned lossless power lines when investigating superconductivity [1]. Technologies using superconductivity have emerged, among them high-temperature superconducting transmission cables [2] and power generators [3]. The science has matured into a technology.

Superconductors can be very useful in measurement techniques through utilising the Josephson effect. The effect describes a superconducting current between two superconductors where a region is isolated from any superconducting effects. The Josephson effect states that the phase of the current is synchronized between the two superconductors if the isolating region is thin enough. This breakthrough has led to very sensitive measurement equipment [4].

Magnetometers can be constructed on the basis of the Josephson effect. They are called Superconducting Quantum Interference Devices or SQUIDs. A SQUID can be used to measure biomagnetic fields and internal organs in humans, such as the brain [5].

The Andreev interferometer is an alternative to the SQUIDs used in experiments

concerning magnetic measurements. The interferometer resistance has been shown to vary with an applied magnetic field. The interferometer has an interface of a normal metal (N) part and a superconducting (S) part. An electron incident to the interface is reflected as a hole, which retraces the path of the incident electron, and creating a Cooper pair inside the superconductor [6].

#### 1.1.1 The Andreev interferometer and thermoelectric currents

Nakano and Takayanagi proposed a new interferometer design with a Y-shaped waveguide in 1993. The Andreev interferometer consisted of a Josephson junction and the Y-shaped normal metal [7]. This is the design used in this project.

In 1995 Petrashov, Antonov, Delsing and Claeson studied *NS* interfaces as mirrors to control the magnetoresistance with a phase shift in the superconductors. They used a cross geometry for their Andreev interferometer. An applied magnetic field caused resistance oscillations in the range of  $\Omega$ s [8].

Stoof and Nazarov presented a successful model on the oscillations in resistance of the Andreev interferometer. They published it 1996, only a year after the experiment by Petrashov, Antonov, Delsing and Claeson. Stoof and Nazarov concluded that the resistance oscillations were due to the temperature dependence in the *NS* interface reflection and that the system was diffusive. Their model had a good fit to the data obtained in Petrashov, Antonov, Delsing and Claeson's experiment [9].

Petrashov, Chua, Marshall, Shaikhaidarov and Nicholls used an Andreev interferometer to measure currents in mesoscopic devices in 2005. Currents in their circuit were associated with a macroscopic phase gradient. An external magnetic field was applied perpendicular to the surface of the circuit [10].

This experiment uses an Andreev interferometer for readout of the resistance oscillations. These can be caused by thermoelectric effects in the bimetallic loop and interactions between the bimetallic loop and the applied magnetic field. The resistance modulation is not in the  $\Omega$  range like in the mentioned experiments, instead the oscillations are in a previously unattained range of k $\Omega$ .

The authors of this thesis have observed resistance quantization in an interferometer. This will be presented more stringently in the chapter on theory and also in the results. Kuzmin, Mahashabde, Shaihadarov, Tarasov and Petrashov are going to present the results at a conference later this year [11].

In the idealized case, all thermoelectric effects and currents inside a superconductor should be cancelled by the supercurrent  $\mathbf{j}_s$ . This does however not hold for all superconductors. In his Nobel lecture V. L. Ginzburg writes "When the superconductor is nonuniform or anisotropic, the currents  $\mathbf{j}_s$  and  $\mathbf{j}_T$  do not in general compensate each other completely, and an observable thermoelectric magnetic field emerges" [12].

There is a substantial mismatch between superconducting theory and experiments of the field's temperature dependence. Van Harlingen and Garland [13] found a discrepancy between the expected and observed temperature dependence of the magnetic flux. The difference is very significant and cannot be explained with classical theory.

Thermoelectric currents are important for the experiments. Almost all measurements will be in a regime where the thermoelectric currents dominate the superconducting currents. In this current range, a thermomagnetic field will be created [12] and the interferometer resistance will be affected.

## **1.2** Statement of purpose

The project purpose is to measure the thermoelectric effect in a superconducting bimetallic loop and to have an understanding of the effect. The aim is to apply nanofabrication technologies to this unsolved problem in physics. Hopefully the results can be used to resolve the lack of theoretical understanding of the effect.

The project also aims to measure the effect using an on-chip Andreev interferometer. This requires that the fabrication process is developed further and that a series of experiments can be conducted at temperatures low enough to achieve superconductivity. The modulation of the Andreev interferometer resistance is measured in three experiments.

In the first an external magnetic field is applied. In the second experiment, the thermoelectric effect is studied. A temperature gradient is induced in the bimetallic loop and the resistance modulation is measured. In the final experiment a temperature gradient is induced in another bimetallic loop, to which the interferometer does not connect. This experiment is intended to test for interaction effects between nearby bimetallic loops.

### **1.3 Project scope and delimiters**

The project focuses solely on the thermoelectric effect in superconducting bimetallic loops. Only Andreev interferometers with the Y-shape proposed by Nakano and Takayanagi [7] are considered for measurements. The experiment setup is in the form of mesoscopic thin film structures on a chip. Aluminium is the only material used for superconductors, while silver and titanium are used for normal metals.

The project is limited to the two chip layouts assigned by the supervisor, Dr. Kuzmin. Both chip layouts include two bimetallic loops, each equipped with five Josephson junctions, which can be used for both heating a part of the loop and measuring the temperature gradient. There is one Andreev interferometer, connected to the smaller loop. The layouts also include a magnetic induction line, which is used to apply an external magnetic field. The layouts includes two distinct junction sizes, large and small, with associated resistances. The dimensions used are specified in the layout. The difference between the two layouts is small, and is mainly important for the fabrication process.

The fabrication follows given recipes. The parameters are varied to develop the process, but the basic structure is not changed. This report covers the various fabrication results from the fabricated samples, but only provides experimental data for one chip.

Only the experiments outlined in the statement of purpose are included in the project. The experiments are limited in scope by the laboratory equipment of the Bolometer group. The measurements are done around 0.3 K and only small magnetic fields are considered. The induced magnetic fields are not measured but can be estimated from the current. This is not included in the project.

The theory chapter is limited to only consider practically measurable quantities. That makes it more useful in the fabrication process. The considered theory is meant to provide a context for the experiment and the results rather than aiming to construct a model in which the results can be explained. The explanations are left for future work. Among other things the Usadel equations in the dirty limit [14] are not considered here, though it is possible that they can solve the problem [15].

The chapter provides the necessary foundations of superconductivity theory to understand the field. Both the Bardeen-Cooper-Schrieffer and the Ginzburg-Landau models are considered in an applied fashion. They are used to give a basic understanding of the subject and to explain the later sections, which describe the structures on the chip: the Josephson junctions, *NS* interfaces and Andreev interferometer. The chapter also introduces the thermoelectric effect to be studied.

## **1.4** Approach to the problem

Extensive literature studies and fabrication work are undertaken to achieve the purpose. The experimental samples are realised in the MC2 cleanroom at Chalmers, Gothenburg. The samples are inspected optically and electrically at room temperature. Promising samples are then measured in a cryostat at temperatures below 1 K, which is referred to as subkelvin in the report.

The fabrication process starts with a clean silicon wafer. The large structures of the layout are constructed through the fabrication steps of photolithography, etching and evaporation. The wafer is then divided into samples. The smaller features are created in similar steps, but using electron beam lithography instead of photolithography. The shadow evaporation technique is used for these structures.

The dependence of the interferometer resistance modulation on the external magnetic field is measured by applying a DC voltage sweep over a magnetic induction line on the sweep. The resulting current creates the magnetic field. An AC bias voltage is applied over the bimetallic loop and the interferometer. The resistance is calculated from the

bias voltage and the measured output voltage over the chip.

In the experiments on the thermoelectric effect the DC sweep was applied between two Josephson junctions. The resulting current establishes a temperature gradient, creating a thermomagnetic current and field. This is described in more detail in the chapters on theory and experiment realisation.

## 1.5 Reading instructions

This section gives an overview of each chapter in the report, with the intention to guide readers through the thesis. The second chapter, *Theory*, covers the theories of superconductivity, the important structures on the chip such as junctions, the thermoelectric effect and the Andreev interferometer.

Chapter 3, *Fabrication*, describes methods and technologies used during the cleanroom fabrication and provides a overview of the junction fabrication. The *Experiment realisation* is covered in chapter 4 where the restrictions, the chip design, and the steps going from design to results are explained. This is followed by *Measurements* in chapter 5, which covers the room temperature screening of faulty samples and the actual subkelvin measurements.

The *Results* are presented in chapter 6, where fabrication results together with results from the three experiments are presented. These results are later discussed in the following chapter, *Discussion*. The *Appendix* has further information about fabricated samples and technical information on tools used in the cleanroom and the cryostats. The circuit diagrams for the measurement setups are also included in the appendix.

Citations are noted with square brackets and a number [2]. If specific pages in a book or longer text should be considered they are noted with page numbers. For example, referring to pages 202 to 204 in Tinkham's *Introduction to Superconductivity* would look like, see pp. 202-204 in [16].

## Chapter 2

# Theory

The theory chapter is about establishing fundamental properties of superconductors for understanding and further on, discussion. The properties concerning applied magnetic fields, temperature dependence on superconductivity and transport theory concerning the currents in a superconductors. Superconducting currents and even other kinds of currents are important for this experiment. A large number of pages has been devoted to how these currents behave in different junctions and depth of the materials used. The main interest of this report is the thermoelectric current, which creates thermomagnetic fields.

A solid theoretical understanding of superconductivity is necessary to draw good conclusions from measurement data. The chapter on theory begins with the foundational Bardeen-Cooper-Schrieffer model, henceforth denoted as the *BCS* model. After the foundations have been laid, the chapter goes quickly from the introductory material towards the dense *Ginzburg-Landau* model of superconductivity. This model is important since all superconductors in use are of thin films, and therefore granular.

## 2.1 Fundamentals of superconductivity theory

Superconductivity emerged as a field of study in the early 20th century. Kamerlingh Onnes tried to measure the conductivity of mercury at very low temperatures. In 1911 he discovered that below 4.19K there was no resistance at all. Infinite conductivity is one of the defining features of superconductivity [1].

Another important milestone was the Meissner-Ochsenfeld effect. When a material is cooled below the critical temperature  $T_c$  a magnetic field cannot in penetrate the material. The superconductor then starts behaving like a diamagnet. The material which exhibits perfect diamagnetism has a magnetic susceptibility of  $\chi \sim -1$ . The

magnetic field is effectively nonexistant inside the superconductor.W. Meissner and R. Ochsenfeld discovered this effect and published it in 1933 [17].

The main idea was that superconducting electrons with opposite spin pair up and lower the total energy of the system. These electrons are bound to each other in a Cooper pair which in turn are coupled to the lattice by phonon-electron interactions. This interaction is the cause of superconducitivity according to the BCS theory which was published in 1957 [18].

The Cooper pairs break up in a magnetic field with a field strength larger than a certain critical value. The breakdown also depends linearly on the magnetic induction [19]. This breakdown is not the same in every superconductor. Generally there are two types of superconductors, type-I and type-II [20].

$$\kappa = \frac{\lambda}{\xi} \tag{2.1}$$

The dimensionless parameter  $\kappa$  in equation (2.1) determines the type of superconductivity in a sample.  $\lambda$  is the penetration depth of a magnetic field from the surface to the inside of the superconductor.  $\xi$  is the coherence length of the superconducting electrons in the sample [21].

$$\kappa < \frac{1}{\sqrt{2}} \tag{2.2}$$

Type-I is defined by having a coherence length  $\xi$  which is much smaller than the penetration depth  $\lambda$  inside the phase below  $T_c$  as shown in (2.2). Magnetic properties are very different in these types. The first type exhibit more of the Meissner type magnetization. Despite this the magnetic flux can penetrate a certain distance inside the superconductor but dissipates quickly.

$$\kappa > \frac{1}{\sqrt{2}} \tag{2.3}$$

Type II superconductors are defined by equation (2.3) [20]. As shown in condition (2.3) the penetration depth of the magnetic field is larger than the coherence length of the superconducting electrons. The second type of superconductor exhibits strange magnetic properties, the interface between normal and superconducting phases have a negative energy. This makes it favourable for magnetic fields to penetrate the sample and creating a mixture of the superconducting phase and the normal phase. This is opposite to how a Type-I superconductor behaves [20].

Gorkov published an article in 1959 which linked the BCS model with Ginzburg-Landau theory near the critical temperature. On one hand the initial guess of the electric charge in the Ginzburg-Landau model was Q = e. By taking Q = 2e, Gorkov could use Ginzburg-Landau theory to explain superconducting currents inside the superconducting phase [22].

In addition Gorkov found that BCS and GL theory were linked through the constant  $\kappa$  by evaluating BCS theory close to the critical temperature. Put together it can be shown that  $\kappa_{BCS} \iff \kappa_{GL}$  [22].

#### 2.1.1 Formulation of Bardeen-Cooper-Schrieffer theory

The BCS model builds on the Bloch model for metals [23], adapting it to also include interactions between electrons and phonons as well as the Coulomb interaction between electrons. The electron-phonon interactions lead to electron-electron interactions, with the exchange of virtual phonons. From now on it is referenced to as phonon interaction. Together with the screened Coulomb repulsion between electrons, it gives rise to the energy difference between normal and superconducting phases. It is assumed that the interaction between a single particle and the system is the same in both phases, thus not contributing to the energy difference [18].

The electrons of the system are characterized by their Bloch states, specified by wave vector **k** and spin  $\sigma$ . The phonon interaction process can be seen as a scattering from a Bloch state specified by **k** to **k**' = **k** ± **K** with a phonon specified by wave vector **K**, absorbed or emitted. Using second quantization creation and annihilation operators,  $c_{k\sigma}^{\dagger}$  and  $c_{k\sigma}$ , the Hamiltonian of the system can be written in the form of (2.4) [18].

$$\mathcal{H} = \sum_{k > k_F} \epsilon_k n_{k\sigma} + \sum_{k < k_F} |\epsilon_k| (1 - n_{k\sigma}) + H_{Coul} + \frac{1}{2} \sum_{k, k', \sigma, \sigma', K} \frac{2\hbar\omega_K |M_K|^2 c^{\dagger} (\mathbf{k}' - \mathbf{K}, \sigma') c (\mathbf{k}', \sigma') c^{\dagger} (\mathbf{k} + \mathbf{K}, \sigma) c (\mathbf{k}, \sigma)}{(\epsilon_k - \epsilon_{k+K})^2 - (\hbar\omega_K)^2}$$
(2.4)

In the notation used,  $\epsilon_k$  is the Bloch energy relative to the Fermi energy,  $k > k_F$  denotes states above the Fermi surface and  $k < k_F$  those below. Thus the two first terms of (2.4) signify the contribution from the Bloch energies of the particles to the energy of the system. The third term is the Coulomb interaction. The fourth term is the phonon interaction term, including a matrix element  $M_K$ . The interaction is attractive for energies  $|\epsilon_k - \epsilon_{k+K}| < \hbar \omega_K$ , while the Coulomb interaction is repulsive. When the phonon interaction dominates the Coulomb term, the system is in the superconductive state [18].

Assuming that this criterion holds and that momentum is conserved, it is advantageous in energy if the electrons form pairs and that all pairs have the same net momentum  $\mathbf{k}_1 + \mathbf{k}_2 = \mathbf{q}$ . The energy is reduced further if the pairs consist of electrons with opposite spins and if  $\mathbf{q} = 0$ . The most desirable ground state is thus  $(\mathbf{k} \uparrow, -\mathbf{k} \downarrow)$ , where  $\uparrow$  and  $\downarrow$  denote the direction of the spin  $\sigma$  [18].

The problem is then reduced to only considering such pairs, where both states are occupied. New creation and annihilation operators  $b_k^{\dagger}$  and  $b_k$  for pairs are defined in terms of the single-particle operators. The part of the original Hamiltonian (2.4) that concerns pairs with  $\mathbf{q} = 0$  can be rewritten as (2.5), where most interactions are included in the new matrix element  $V_{kk'}$ , which will be positive for a superconductor. Pairs with total momentum  $\mathbf{q} \neq 0$  have little influence and may be treated as perturbations [18].

$$\mathcal{H}_{red} = 2\sum_{k>k_F} \epsilon_k b_k^{\dagger} b_k + 2\sum_{k< k_F} |\epsilon_k| b_k b_k^{\dagger} - \sum_{kk'} V_{kk'} b_{k'}^{\dagger} b_{k'}$$
(2.5)

#### 2.1.2 Free energy and the Ginzburg-Landau model

The thermodynamical free energy is a vital concept in superconductivity. A number of other thermodynamical quantities are functions of the free energy. These quantities include the specific heat, the critical magnetic field and the groundstate energy, see [18] and pp. 546-568 in [24].

#### Free energy in the Bardeen-Cooper-Schrieffer model

The energy of a particle with wave number k is obtained by applying the Hamiltonian (2.5) to a wave function. The energy is approximated by equation (2.6). The energy gap  $\Delta(T)$  is dependent on the current temperature. When minimizing the free energy, F, with respect to the energy distribution function one arrives at the solution (2.7), which is the Fermi-Dirac distribution. For  $k > k_F$  it describes electron distribution and for  $k < k_F$  hole occupation [18].

$$E_k = \sqrt{\epsilon_k^2 + \Delta(T)} \tag{2.6}$$

$$f_k = \frac{1}{e^{\frac{E_k}{k_B T}} + 1} = f(E_k)$$
(2.7)

In the limit  $\epsilon_k \to 0^+$  the electron energy  $E_k$  approaches  $\Delta(T)$ . Correspondingly for  $\epsilon_k \to 0^-$  the hole energy becomes  $\Delta(T)$ . The electron energy is then  $-\Delta$ . Subsequently the energy gap of  $2\Delta$  is centered around the Fermi energy.

The modified density of states then becomes (2.8), which is singular at the gap edges. The density of states is plotted in figure 2.1, in which the energy gap of  $2\Delta(T)$  is clearly visible. When  $\Delta(T)$  approaches 0, the superconductors density of states approaches that of a normal metal, and the quotient in (2.8) approaches 1.

$$\frac{dn(E)}{dE} = n(0)\frac{E}{(E^2 - \Delta(T)^2)^{\frac{1}{2}}}$$
(2.8)

The transition temperature  $T_c$  is defined as the boundary where there is no real, positive value of  $\Delta(T)$  satisfying (2.8). Above  $T_c$  the value of  $\Delta(T)$  is therefore zero, and the energy distribution simplifies into the distribution of Bloch theory – the metal returns to the normal phase. Below  $T_c$ , it is required that  $\Delta(T) \neq 0$  to minimize the free energy and the specimen is in the superconducting phase [18].

This suggests that the transition temperature and the energy gap are closely related, as the equations (2.9) and (2.10) confirm. Equation (2.9) shows the energy gap at T = 0 K. When the temperature is near  $T_c$ , meaning  $1 - \frac{T}{T_c} \ll 1$ , the gap is instead given by equation (2.10).

$$\Delta_0 = \frac{3.50}{2} k_B T_c \tag{2.9}$$

$$\Delta(T) = 3.2k_B T_c \sqrt{1 - \frac{T}{T_c}}$$
(2.10)



**Figure 2.1** – The density of states of a normal metal (green, dashed) and that of a superconductor (blue, solid). Both are normalised with the density of states of a normal metal.

#### Free energy in the Ginzburg-Landau model

The free energy expansion of a superconducting phase transition exhibits the characteristics of a second order phase transition. Landau investigated phase transitions of the second order (see pp. 193-216 in [24]) which are defined by a continuous change in the lattice sites of the atoms and particles, but discontinuous in the symmetry change of the sample. Thus a second order phase transition creates a symmetry break at a critical temperature  $T_c$ , also called the point of transition. Symmetry changes discontinuously, as described in section 2.1.3, see p. 447 in [25].

From the general theories of Landau (see pp. 193–216 in [24]) there exists a generalized thermodynamic function  $\Xi$  which is a function that depends on temperature and pressure. The transition is modelled as a thermodynamic potential in the following way:  $\Xi = \Xi_0 + A\gamma^2 + B\gamma^4 + \dots$ 

According to the original article by Landau and Ginzburg (see pp. 546-568 in [24]) the order parameter is closely related to the amount of superconducting electrons, as in condition (2.11). In a similar way this applies to superconductors, which means that  $\gamma = |\Psi|$ . This could in the superconducting state be considered as the free energy, *F*, near and below the critical temperature, *T<sub>c</sub>*, from the original article by Landau and Ginzburg (see pp. 546 - 568 in [24]) and addendums from the review article by Cyrot [21] :

$$n_s = |\Psi|^2 \tag{2.11}$$

$$F_{S} = F_{0} + \alpha |\Psi|^{2} + \frac{\beta}{2} |\Psi|^{4}$$
(2.12)

Free energy contribution

Because only two phases are considered only three terms appear in equation (2.12). The three terms are as follows:  $F_0$  is the free energy significantly over  $T_c$  in the disordered room temperature state.  $\alpha$  is a constant. This constant fluctuates with temperature as  $\alpha = \alpha'(T - T_c)$ . On the other hand  $\beta$  is constant with respect to temperature, see [21] and pp. 546-568 in [24]. The constants are present in energy exchange in the Ginzburg-Landau model. They connect the different parts of the model to form a coherent theory.

$$F_{S} = F_{0} + \alpha |\Psi|^{2} + \frac{\beta}{2} |\Psi|^{4} + \frac{\hbar^{2} |\nabla\Psi|^{2}}{K_{\text{inetic energy contribution}}}$$
(2.13)

Note how the kinetic energy changes from  $\hbar^2 \nabla |\Psi|^2 \Rightarrow |(-i\hbar \nabla - 2e\mathbf{A})^2 \Psi|$  in equation (2.13) to (2.14). The assumption made is basically that the vector potential in the kinetic energy is gauge invariant. Physical fields often have phases which affect the physical properties of said field. If a property is gauge invariant, it does mean that any arbitrary phase does not affect the physical properties of the system, see pp. 135–136 in [26]. The fourth term with  $\nabla |\Psi|^2$  means the kinetic energy of the order parameter. The superconducting electrons cannot have too rapid changes in kinetic energy, since this model would not continue to be valid [21].

$$F_{S} = F_{0} + \alpha |\Psi|^{2} + \frac{\beta}{2} |\Psi|^{4} + \left| \left( -i\hbar\nabla - 2e\mathbf{A} \right)^{2} \Psi \right| + \underbrace{\frac{|\mathbf{H}(r) - \mathbf{H}_{a}|^{2}}{2\mu_{0}}}_{\text{Magnetic field contribution}}$$
(2.14)

If a weak magnetic field is applied to the equation, an additional fifth term comes about which is the magnetic field strength, see equation (2.14). The considerations made are for a varying magnetic field strength with regards to position and also the critical field strength. The situation when applying a weak magnetic field is also gauge invariant [21]. If no external magnetic field is applied, the Ginzburg-Landau equation is only considered with the first four terms. These terms are the ground state free energy and the kinetic energy, just as in the previous equation (2.13). If the applied field is very weak, the same equation applies for that situation [21].

#### 2.1.3 Critical fields and specific heat

The critical field  $H_c$  for a superconductor can be expressed in terms of the free energy of the normal phase,  $F_n$  and that of the superconducting state,  $F_s$ , as in (2.15), see p. 21 in [27]. The relation (2.16) shows the temperature dependence of the critical field  $H_c$  at temperatures near  $T_c$  (that is  $1 - \frac{T}{T_c} \ll 1$ ), see [18] and p. 21 in [27]. As can be seen,  $H_c$  depends on the critical field at zero temperature  $H_0$ , which can be taken as a parameter.

$$F_n - F_s = H_c^2 \frac{\mu_0}{2}$$
(2.15)

$$H_c = H_0 \left[ 1 - \left(\frac{T}{T_c}\right)^2 \right] \tag{2.16}$$

Since the entropy is related to the free energy as  $S = -\frac{\partial F}{\partial T}$  we arrive at the difference of entropy between the normal and superconducting phases in (2.17). The righthand side is always positive, implying that the order is greater in the superconducting state than in a normal metal. The one exception is just at the transition temperature where the

critical field is zero, and the entropy difference is continuous.

$$S_n - S_s = -\mu_0 H_c \frac{\mathrm{d}H_c}{\mathrm{d}T} \tag{2.17}$$

The specific heat is related to the entropy through the thermodynamic relation  $C = T\left(\frac{\partial S}{\partial T}\right)_{H}$ . Applying it to the entropy difference in (2.17) the difference of specific heat at  $T_c$  becomes (2.18), see p. 22 in [27]. Since the righthand side always is positive, there is a jump in specific heat at the critical temperature. This jump is consistent with a second order phase transition. Combined with the critical field situation, this is stringent evidence for a transition of the second order.

$$(C_s - C_n)|_{T_c} = \mu_0 T_c \left(\frac{\mathrm{d}H_c}{\mathrm{d}T}\right)\Big|_{T_c} > 0$$
(2.18)

#### The critical magnetic field in the Ginzburg-Landau model

The critical field in a type-II superconductor has two critical points in an external magnetic field. The first point just lowers the amount of superconducting electrons, but does not destroy the superconducting properties of the sample like the second critical field strength does. The starting point is the free energy difference in the superconducting and the disordered phase. The penetration depth is defined as in equation (2.20); it can be transformed from the constants  $\alpha$  and  $\beta$  to the measureable quantities of the magnetic field strength and electric charge.

$$F_s - F_0 = -\frac{\alpha^2}{2\beta} \Rightarrow H_{c1}^2 = \frac{\mu_0 \alpha^2}{\beta}$$
(2.19)

$$\lambda^{2} = -\frac{mc^{2}\beta}{2\mu_{0}e^{2}\alpha} \Rightarrow \kappa = 2\sqrt{2}\frac{e}{\hbar}H_{c1}\lambda^{2}$$
(2.20)

The first critical magnetic field strength is coupled to the  $\kappa$  constant. This constant varies as the penetration depth  $\lambda$  and coherence length  $\xi$  vary with temperature. Further developed for the second critical field gives the following dependence  $H_{c2} = \kappa H_{c1} \sqrt{2}$ . This holds if the superconductor is of type-II and  $H_{c2} > H_{c1}$  [21]:

$$\int \left( b |\Psi|^4 + \frac{e\hbar}{m} |\Psi|^2 \left( H_a - H_{c2} - \frac{\mu_0 e\hbar}{m} |\Psi|^2 \right) \right) d^3r = 0$$
(2.21)

$$\frac{\int |\Psi|^4 \, \mathrm{d}^3 r}{\left(\int |\Psi|^2 \, \mathrm{d}^3 r\right)^2} = b_A \tag{2.22}$$

According to Cyrot [21] the geometric factor,  $b_A$  in equation (2.22) is only a function of the lattice. In our experimental setup aluminium is used as the superconductor. Aluminium has a fcc-lattice and this is a square lattice [28]. The following general assumption for the square lattice holds because of this.

$$\Rightarrow F = \frac{1}{2\mu_0} \left( B^2 - \frac{(H_{c2} - B)^2}{1 + (2\kappa^2 - 1)b_A} \right)$$
(2.23)

$$\Rightarrow H = \mu_0 \frac{dF}{dB} \Rightarrow M = \frac{B - H}{\mu_0} = \frac{H_a - H_{c2}}{\mu_0 b_A (2\kappa^2 - 1)}$$
(2.24)

The magnetization increases like a linear function of the magnetic field strength in both type-I and type-II superconductors [19],[21] ; however, a magnetic field inside a

granular or type-II superconductor varies with an arbitrary periodicity. The periodicity depends on both experimental setup and what kind of field is induced, this will be further elaborated upon in the chapter about results [21].

#### Quantization of magnetic flux in superconducting rings

F. London postulated that the current density should be proportional to the average of the momentum, **p**, and magnetic vector potential as in (2.25) [29]. Inside the superconductor the Meissner effect renders the current density non excistant. This leads to the condition for a the quantization through the geometry, see (2.26).

$$\mathbf{j} \propto \mathbf{p} - Q\mathbf{A} \tag{2.25}$$

$$\oint \mathbf{p} \cdot d\mathbf{x} = -nh \tag{2.26}$$

From conditions, (2.25) and (2.26) the magnetic flux through a ring is evaluated. The conditions are combined and integrated throughout the volume of the ring. Magnetic flux is obtained when integrating with in regard to the path. Q is also substituted with the electric charge of the Cooper pair, taken as Q = -2e.

$$\Phi_n = \oint \mathbf{A} \cdot d\mathbf{x} = -\frac{nh}{Q} \tag{2.27}$$

$$\Rightarrow \Phi_n = \frac{nh}{2e} \qquad n = 1, 2, 3 \dots \tag{2.28}$$

Onsager writes about the origins of a very peculiar effect, the magnetic flux in a closed path can be measured without examining the enclosed field [29]. He further states that to achieve a Meissner effect in a multiply-connected superconductor it needs a closed geometry. A closure like that will not take place unless the free energy which matches the phases exceeds both the superconducting current and the applied magnetic field [29]. The experimental results which confirms this was published by Deaver and Fairbank [30] and Little and Parks [31].

Every integer n in the flux quantization represents a different pairing in the superconducting phase. Furthermore these effects vary periodically with the flux, as shown by Little and Parks [31]. These periods will be an important effect to measure in the superconducting bimetallic loop in chapter 4.2.2 on page 46.

#### 2.1.4 Electrodynamics and currents

Superconducting currents can persist in the superconducting phase for long periods of time [1]. In a type-I superconductor, the currents are modelled according to equation (2.30) and create perfect diamagnetism inside the superconductor. For this case, F. and H. London constructed a theory which has similar traits of Ohm's law but is different in a superconductor [32]. The expression of the penetration length is given in equation

(2.29).

$$\lambda_L = \sqrt{\frac{\epsilon_0 m c^2}{n_s e^2}} \tag{2.29}$$

$$\mathbf{j} = -\frac{1}{\lambda_L^2 \mu_0} \mathbf{A} \tag{2.30}$$

A postulate from the London derivation is the prefactor before the vector potential in the magnetic field  $\mathbf{A}$  in equation (2.31). If the curl operator acts on both sides of the previous equation (2.30), the following magnetic vectorfield is obtained [32] :

$$\nabla \times \mathbf{j} = -\frac{1}{\lambda_L^2 \mu_0} \qquad \nabla \times \mathbf{A} = -\frac{1}{\lambda_L^2 \mu_0} \mathbf{B}$$
(2.31)

$$if \begin{cases} \nabla \cdot \mathbf{A} = 0\\ \nabla \cdot \mathbf{j} = 0 \end{cases}$$
(2.32)

The explanation of the Meissner effect is the following; the magnetic field does not penetrate the sample with boundary conditions (2.32) [18]. In any region where the superconducting current is zero, the magnetic field is also identically zero.

Disordered materials like alloys have the traits of different components. They are of varying sizes and position relative to each other in the material. In the Pippard limit the equations by F. and H. London have limited validity because of additional scattering of the electrons in the material caused by impurities. Instead Pippard's formulation of the superconducting current should be considered in Type-II superconductors. The following equation by Pippard (2.35) is viable for alloys and thin films [33], which we are using in our experimental circuit as stated in section 4.2.1 on page 42.

The proposed equations by Pippard [33] contain a vector potential, which varies spatially within the potential. The coherence length  $\xi$  given in (2.34) is an additional variable in the Pippard formulation of the superconducting currents. The property of  $\xi_0$  is the coherence length in the BCS model. The following equations are in the Pippard formulation on a similar basis of the London gauge:

$$\mathbf{j} = -\frac{\xi\mu_0\lambda}{\xi_0}\mathbf{A}$$
(2.33)

$$\xi = \sqrt{\frac{\hbar^2}{2m|\alpha|}} \tag{2.34}$$

$$\Rightarrow \mathbf{j} = -\frac{3\lambda}{\xi_0} \int \frac{\mathbf{r}(\mathbf{r} \cdot \mathbf{A})e^{-\frac{t}{\xi}}}{r^4} dV$$
(2.35)

The strength in Pippard's theory is that it is valid for much more complicated granular materials, which the London theory is not. As can be seen in the last equation, the current varies exponentially with the temperature dependent coherence length. One important aspect in our experiment is that the Pippard model clearly states that the current can vary on the surface of the superconductor [33].

The London limit is defined in (2.36), in this limit the Pippard equation (2.35) is valid. The Pippard limit governs the range where the London equations hold [34]. These limits are also another way to define type-I and type-II superconductors.

A Pippard superconductor obeys the Pippard limit which makes it a type-I superconductor. Type-II superconductors obey the London limit and are also called London superconductors [35].

$$\lambda \gg \xi \tag{2.36}$$
  
$$\xi \gg \lambda \tag{2.37}$$

If the mean free path,  $l_e$ , is short compared to  $\lambda$ , then all electrons contribute to the current. However, if the mean free path is very long compared to the penetration depth of the sample then the current is mainly on the surface. A small fraction of electrons flowing on the superconductors surface can carry the greater part of the current [33].

### 2.2 Superconducting interfaces and junctions

Junctions and boundaries of superconductors with other materials give rise to important effects. Superconductors, normal metals and insulators are denoted as *S*, *N*, and *I* respectively. To explain the bimetallic loop circuit the *SIN* and *SIS'* junctions and the *NS* interface are needed. The *SIN* junction is covered in appendix E.

The *SIS*' and *SIN* junctions can be explained through a theory presented by Tinkham [16] while the *NS* junction is used in a different manner, and a different theory and model are used. To understand *SIS*' junctions, the concept of a weak superconductor is useful. A superconductor is characterized by the superconducting energy gap discussed in both Bardeen-Cooper-Schrieffer theory and Ginzburg-Landau theory in 2.6 on page 9.

A weak superconductor, denoted *S*' has a smaller gap energy  $\Delta' < \Delta$ . This is due to the strength of the coupling between the phonon-electron and electron-electron interactions in the superconductor [36]. A small coupling constant results in a low critical temperature, lowering the energy gap of the superconductor [37]. The conclusion of Allen and Dynes's article is that the density of phonon states determines the strength of the interaction.

#### 2.2.1 The electron density

The density of states is discussed in detail in section 2.1.2 on page 9 and is important to understand the electron density on each side of the interface. Tinkhams model (see p. 70 in [16]) is useful to describe the current and voltage, *I-V* characteristics of superconductors interfaces. The *SIS'* and *SIN* junctions are presented using the electron density approach from Tinkham has a more detailed theoretical discussion.

The electron density, as a function of energy is obtained by multiplying the density of states in the superconductor and the Fermi-Dirac distribution. The Fermi-Dirac distribution, discussed in section 2.1.2 on page 9, determines the proportion of electrons with a certain energy, given the systems average energy. The density of electrons is an integral part of Ginzburg-Landau theory, where the order parameter  $\Psi$  is related

through  $\Psi = \sqrt{n_s} e^{i\theta}$ , see p. 118 in [27]. This phase is important for considering superconducting loops and the interaction of two superconductors. A phase difference in a superconducting loop needs to take into account the presence of a magnetic field to be gauge invariant, see p. 202 in [16]. Figure 2.2 shows the electron density for different temperatures. When the temperature approaches the critical temperature  $T_c$  the density of electrons approaches that of a normal metal.



**Figure 2.2** – The electron density depends on the product of the density of states and the Fermi-Dirac distribution. The density is normalised with the electron density of the normal metal.

#### 2.2.2 The SIS' junction

The interface of two different superconductors, one strong and one weak, separated by an insulating layer is referred to as a *SIS'* junction. Josephson published an article in 1962 describing how a Cooper pair tunnel current passes through the interface. The current depends on the phase difference of the superconductors, as shown in equation (2.38) [4].

$$I_s = I_0 \sin\left(\theta - \theta'\right) \tag{2.38}$$

Tinkham's model describes the tunnel current by equation (2.39). It is similar in form to that of the *SIN*-junction, but considers the density of states of both superconductors, see p. 77 in [16]. The two superconductors *S* and *S'* have different energy gaps, giving rise to the differing densities of state  $n_s$  and  $n'_s$ .

$$I_{SIS'}(V,T) = \frac{1}{e R_{SIS'}} \int_{-\infty}^{\infty} n_s(\epsilon) n'_s(\epsilon + e V) \left[ f(\epsilon,T) - f(\epsilon + e V,T) \right] d\epsilon$$
(2.39)

The I-V characteristics of the *SIS'*-junction are shown in figure 2.3. For low temperatures, when both metals superconduct the dashed line shows the current–voltage relation. The resistance  $R_{SIS'}$  is shown for comparison, which identifies the system when superconductivity is lost.



**Figure 2.3** – The I-V characteristics of the *SIS*-junction, around  $T \approx 0$ . The tunnelcurrent (green, dashed) has a sharp jump at the sum of the two energy gaps  $\Delta + \Delta'$  and approaches the I-V characteristic of two normal conductors with impedance  $R_N$ 

A *weak link* is a term commonly used in the context of *SIS'* and *SIN* junctions. Likharev defines the term as "the conducting junction between bulk superconducting specimens ("electrodes"), the critical current which is much less than that in the electrodes" [38]. A weak link is thus different from the tunnel-type junctions discussed earlier, since a weak link can have a non tunnelling conductivity.

*SIS'* junctions provide many important functions for the bimetallic loop. The design discussed in section 4.2 on page 41 covers more details on what their purpose is. Junctions with both small and large cross sections are used on the samples, and the finished fabricated product can be seen in the scanning electron microscope figure 6.6 on page 60.

### 2.2.3 The NS interface

The normal metal–superconductor interface is of extra interest for this experiment. The interface consists of two metals joined by a common two dimensional surface, one metal superconducts and the other is in the normal phase. The characteristics provide the realisation of an Andreev interferometer discussed later in section 2.3. Tinkhams model is incapable of describing the phase shifting effect discovered by Andreev in 1964 [39]. The reflection effect is often referred to as Andreev reflection.

Virtanen and Heikkilä provide a description of the *proximity effect*, protruding into the normal metal from the superconductor [40]. Their article takes into account both the

transport of the carriers of electrical current and the carriers of thermal currents. These models are too comprehensive and complex for further elaboration, but should be considered for further theoretical studies. The thermoelectric effects influencing the interface are to some extent covered in section 2.4 on page 21.

Nakano and Takayanagi published an article [7] providing the necessary theory to describe the phase shifting and the application to interferometers. Their analysis started with the Bogoliubov-de Gennes equations (2.40), which illustrate the electron hole pair of incident and reflected particles on the interface, see pp. 137–145 in [41]. The actual interface is modelled with a change in the energy gap, similar to a potential  $U(\mathbf{r})$  both by de Gennes and Pincus [41] and Nakano and Takayanagi [7]. In the normal metal, no energy gap exists and the potential is zero, while in the superconductor the energy gap  $\Delta$  is nonzero. The junction is shown in figure 2.4.

$$i \partial_t \psi_e = \left(-\frac{\nabla^2}{2m} + \mu\right) \psi_e + i U(\mathbf{r}) \psi_h$$
$$i \partial_t \psi_h = \left(\frac{\nabla^2}{2m} + \mu\right) \psi_h - i U(\mathbf{r})^* \psi_e$$
(2.40)



**Figure 2.4** – The geometry of reflection interfaces. An electron traveling in the normal metal towards the interface is reflected as a phase shifted hole, in the normal metal and to conserve charge a Cooper pair is created in the superconductor [7].

Nakano and Takayanagi use a two dimensional approach, where *x* is the line of reflection, and the potential  $U(\mathbf{r})$  simplifies to equation (2.41). This means that the area close to the interface (x = 0) is omitted from further consideration. The normal metal is then at x < 0 and the superconductor at x > 0, as in figure 2.4.

$$U(x,T) = \begin{cases} 0 & x < 0\\ \Delta(T) e^{i\theta} & x > 0 \end{cases}$$
(2.41)

Nakano and Takayanagi propose plane wave solutions to equation (2.40) ignoring reflection and transmission normally associated with scattering. The reflected wave has a phase contribution which is determined through continuity conditions. This phase is a factor  $e^{i(\theta+\varphi)}$ , where  $\theta$  is the phase contribution from the potential equation (2.41) and  $\varphi$  in equation (2.42) the phase gained in the reflection [7].

$$\varphi = \arctan \sqrt{\left(\frac{\Delta(T)}{E}\right)^2 - 1}$$
 (2.42)

 $\varphi$  approaches  $\frac{\pi}{2}$  when the energy of the incident electron is near the Fermi energy. The Andreev reflection occurs when the energy of the incident electron *E* is small in comparison to the superconductors energy gap  $\Delta$ . At low temperatures most electrons in a normal metal have energies close to the Fermi energy, in accordance with the Fermi-Dirac statistics. Normal reflection and transmission effects occur at higher energies, but these manifest without the phase shift.

#### Magnetic influence

The *NS* interface is susceptible to the influence from external magnetic fields, a phenomenon which Little and Parks demonstrated and published in 1962 [31]. The influence from the magnetic field changes the energy binding the Cooper pairs, thus decreasing the energy gap and through that the critical temperature. Little and Parks discovered the periodicity of the oscillations, which have maximums at integer values of the flux quantum  $\Phi_0$  discussed in section 2.1.3 on page 13.

By changing the size of the energy gap, the free energy of Ginzburg-Landau model changes and equation (2.29) relates this to a change in penetration depth. With an increase of the penetration depth, surface currents may be induced in the superconductor, and a current induced in the normal metal. Any interfering currents in the interface would only be induced by changes in the magnetic field.

## 2.3 The Andreev interferometer

The Andreev interferometer, presented by Nakano and Takayanagi [7] is designed to measure the phase difference of the two superconductors in an *SIS'* interface. Through constructing *NS*-junctions on each side of the Josephson junction, as depicted in figure 2.5, each interferometer arm will enable Andreev reflection. An electron wave, from *W* or *X*, will be split at the join in the Y-shape, and each wave will get Andreev reflected in the junction. The two "hole"-waves will thus have a phase shift around  $\frac{\pi}{2}$  from the reflection, but also a phase from the superconductor  $\theta$  and  $\theta'$ , as presented earlier.

The design proposed by Nakano and Takayanagi, as depicted in figure 2.5, allows for voltage and current to be measured through four connections, U, V, W, X. The impedance of the interferometr can then be measured while the phase difference of the superconductors  $\theta - \theta'$  is varied. The resistance would then vary in accordance with equation (2.43) allowing the resistance to vary from  $\frac{R_N}{2}$  to infinity [7]. Checkley, Iagallo, Shaikhaidarov, Nicholls, and Petrashov, proposed a smaller variation, as equation (2.44), though this was for a different interferometer geometry [6].

The resistance  $R_N$ , common for both forms is formed by the smallest restriction of the interferometer. In the ideal case of the Nakano and Takayanagi design (figure 2.5), the narrowing of the waveguide causes a so called Sharvin resistance, which will be discussed further on page 21. In the realistic case the waveguide has no narrowing and



**Figure 2.5** – The Andreev interferometer as the yellow Y-shape and the *SIS'* which forms the Josephson junction.

the normal resistance  $R_N$  will be smaller than the Sharvin resistance.

$$R = \frac{R_N}{1 + \cos\left(\theta - \theta'\right)} \tag{2.43}$$

$$R = R_N - \delta R(T) \left(1 + \cos\left(\theta - \theta'\right)\right) \tag{2.44}$$

The Andreev reflected quasiparticles can be assumed to have an energy close to the Fermi energy because of the low temperature needed for superconductivity. Since the temperature is approximately constant in the interferometer, particles in both arms have similar energies, but the superconductors *S* and *S'* could have slightly different energy gaps. The phase contribution from the reflection,  $\varphi$  from equation (2.42), could be different. This contribution could cause problems when measuring the absolute phase difference of the superconductors.

Considering the phase from the Andreev reflection for small variations in the quasiparticle energy, the expression is series expanded according to equation (2.45). Regardless of energy gap, the phase contribution will for small energies be close to  $\frac{\pi}{2}$ .

$$\varphi = \arctan \sqrt{\left(\frac{\Delta}{E}\right)^2 - 1} \approx \frac{\pi}{2} - \frac{E}{\Delta} + \dots$$
 (2.45)

If the energy of the incident quasiparticle where to be too large, approximately the same size as the energy gap of the superconductor, the interface would stop Andreev reflecting and reflect or transmit particles similar to a normal reflective surface. This can be explained using a model similar to the *SIN* I-V integral in equation (E.1).

The coherence of electron-hole waves in the normal metal of the interferometer is vital for its function. The phase information of the quasiparticle must be conserved throughout the round trip, up and down the interferometers arms. This requires that the arms must be shorter than the coherence length [7].

Cadden-Zimansky, Wei, and Chandrasekhar published experimental results confirming the coupling of the electron-hole waves for distances smaller than the superconductors coherence length [42]. In practical terms, the length of each interferometer arm (*L* in figure 2.5) must be smaller than the coherence length of the superconductor  $\xi$ .

The small narrowing of the interferometer close to the branching of the Y-shape causes what is known as a Sharvin point contact [43],[44]. The requirements to attain this gap is that the width and length of the gap both are smaller than the mean free path length  $l_e$ , closely related to the Fermi energy. The two dimensional electron gas is a useful model, since the thickness of the metal is small and the conductance of the narrow point can, quasi-classically be written as (2.46).

$$G_{\rm Sharvin} = \frac{e^2}{\pi\hbar} k_F \frac{W}{\pi}$$
(2.46)

Where  $k_F$  is the Fermi wave vector and W the width of the point contact under the approximation that the width of the point contact is much smaller than the mean free path length [44]. The mean free path length of silver is  $l_{e,Ag} = 52 \text{ nm}$  [45]. The quantized conductance leads, in accordance to Büttiker to conducting channels in the material which contribute to a total conductivity [46]. For structures larger than a Sharvin point contact the conductivity would then depend on which channels are conducting under the given conditions. If the branching point is a point contact, the Sharvin resistance will dominate the resistance of the whole interferometer and the resistance,  $R_N$  in equation (2.43) can then be written as the inverse of the Sharvin conductance in (2.46).

### 2.4 The thermoelectric effect

In this subchapter a background will be given to the thermoelectric paradox and certain fundamental relations will be shown. Ginzburg proved the existence of a thermoelectric current in a superconductor [12]. Van Harlingen and Garland conducted a seminal study on this effect in the beginning of 1978 [13]. The conclusions from the study was that the thermoelectric flux did not behave like previous theory had predicted.

This previous research is important for this study. There is a good reason for this. The first one is because the experiment will investigate any coupling between an applied magnetic field and the thermomagnetic field. The thermomagnetic field is caused by the thermoelectric current.

Another important thing is if the phase difference in the Andreev reflection will cause an oscillation in the magnetoresistance of the interferometer. This phase difference depends on temperature, applied magnetic field and the currents inside the superconductor [47]. Petrashov, Antonov, Delsing, Claeson have previously conducted a study on Andreev interferometers with modulating resistance because of an applied magnetic field [8]. This is also called magnetoresistance.

#### 2.4.1 Background to the thermoelectric effect

Meissner concluded early in the 20th century that a thermoelectric effect could not occur in the superconducting phase. Gorter and Casimir stated that there should not be any thermoelectric currents,  $j_T$ , when using their two-fluid model to describe the superconducting phase.

Ginzburg studied the thermoelectric problem in 1943 and 1944. The thermoelectric current should be cancelled out by the superconducting current,  $\mathbf{j}_{s}$ , if  $T < T_{c}$  like the condition in (2.47) which gives the equality (2.48) [12].

$$\mathbf{j}_S + \mathbf{j}_T = 0 \tag{2.47}$$

$$\Rightarrow \mathbf{j}_S = -\mathbf{j}_T = -\eta_T \nabla T \tag{2.48}$$

When a superconductor is granular, the currents are not uniform and therefore do not compensate each other in the superconductor. This thermoelectric current gives rise to a thermomagnetic field. This effect is pronounced in the interface between the normal and superconducting phases [12]. If a temperature gradient,  $\nabla T$ , exists across the junction then the effect gets even more visible.  $\eta_T$  is a temperature dependent constant [12]



**Figure 2.6** – The picture shows a bimetallic loop with two *SIS'*-junctions. At the bottom of the loop there is an Andreev interferometer. The thermomagnetic field  $H_T$  is perpendicular to the mixture of thermoelectric and superconducting current denoted by *J* [12].

According to Ginzburg, the existing thermoelectric current can be quenched by the supercurrent. If this happens, the thermomagnetic field,  $\mathbf{H}_T$ , will also be quenched. Despite this, if an uncompensated thermoelectric current is in an anisotropic or granular superconductor, then  $\mathbf{H}_T \neq 0$  as in figure 2.6 and this field is temperature dependent, or  $\propto (\nabla T)^2$  [12]. The magnetic flux  $\mathbf{\Phi}$  is also  $\propto (\nabla T)^2$  according to Lawrence, Pipes and Schwartzman [48].

The magnetic flux is caused by a thermoelectric current as previously mentioned, this current is created by a temperature gradient. The Meissner effect requires  $\nabla \times \mathbf{j} = 0$ , which is not true in granular metal films. A granular metal film is a type II superconductor. The flux is predicted to be observable near  $T_c$  [49].

Consider a bimetallic loop with an applied  $\nabla T$  in two *SIS'*-junctions, on the top of the

loop. The  $\theta - \theta'$  induced by the different superconductors in the loop is shown in equation (2.49). The first contribution is from the supercurrent and the second is from the quantization of magnetic flux in the vector potential [49]. The following equation was proposed by Galperin, Gurevich and Kozub [47] :

$$\nabla \theta = \frac{2m}{e\hbar n_s} \mathbf{j}_s + \frac{2e}{\hbar} \mathbf{A} + \frac{2m\eta_T}{en_s\hbar} \nabla T$$
(2.49)

Assume the thermoelectric current in (2.48) is combined with equation (2.49). This combination evaluates as equation (2.50) [49]. The first contribution  $\Phi_0$  is the flux quantization effect.  $\Phi_T$  is the thermoelectric flux which can give the thermoelectric effect.

$$\mathbf{\Phi} = n \, \Phi_0 + \Phi_T = n \, \Phi_0 + \int_{T_1}^{T_2} \left( \frac{m \eta_{T_2}(T)}{e^2 \, n_s(T)} - \frac{m \eta_{T_1}(T)}{e^2 \, n_s(T)} \right) \mathrm{d}T \tag{2.50}$$

#### 2.4.2 Granular superconductors

In this experiment thin films are used of various metals and thicknesses. A thin film is a very thin layer of any material. The superconducting thin films in our experiment are made of aluminium. These superconductors are clearly disordered when viewed in a scanning electron microscope, as in figure 6.6 on page 60. Aluminium is a type-II superconductor in its thin film form, a fact known for a long time [50].

Type-II superconductors do not exhibit a perfect Meissner effect, but instead different electromagnetic properties as discussed in 2.1 on page 7. The coherence length varies in the sample with both temperature and geometry, which affects the critical field of the thin films. The grains in the superconductor are an intrinsic material quality in our experimental situation. A number of grains form internal pathways connected to other grains inside the thin film. These grains form a inner network of weak links [50]

Most of the current will be on the surface or edges of the aluminium thin film and not in the bulk of the superconductor [50]. The defects, such as dislocations and grains alter the superconducting properties of the thin film. The defect interface has a certain area and if the area of these defects increases, the critical current density increases. This yields a very low critical current if the grains and other defects have a small scale and are evenly distributed in the sample. Minimization of the grain size divided by the coherence length lead to extremely low critical-current densities and thus minimize the  $j_c$  in the sample [51].

#### Thermoelectric currents in superconductors

The thermoelectric current resides in the surface layers of the superconductor. This experiment considers the Pippard equation (2.35) on page 14 and especially the surface currents in this model. In the thin film case of aluminium, it is even more pronounced. When the currents are large in the edges of the thin film, or the surface and a few orders of magnitude less in the bulk material [51].

If condition (2.47) regarding total conservation of the currents holds, there exists two cases. The case in condition (2.51) has a larger contribution to the superconducting current than the thermoelectric current. The other case (2.52) has instead a larger contribution to the thermoelectric current.

$$\mathbf{j}_S > \mathbf{j}_T \tag{2.51}$$

 $\mathbf{j}_{S} < \mathbf{j}_{T} \tag{2.52}$ 

When the superconducting current is larger than the thermoelectric current, this should quench the thermomagnetic effect. The supercurrent does not display a perfect Meissner effect in the granular superconductor. The thermoelectric current does however not obey the Meissner effect [12].

#### 2.4.3 Charge imbalance in superconductors

Ginzburg stated that the current is carried by electrons. This means that the current also can be carried by quasiparticles of the hole type [12]. In Andreev reflection, holes are one kind of quasiparticle reflected from the superconductor. Electrons can also be modelled as quasiparticles [52].

$$n_{><} = n_{>} - n_{<} = 2n(0) \int_{\Delta}^{\infty} n(E_{k}) (f_{>} - f_{<}) dE_{k}$$
(2.53)

Charge imbalance can be seen in equation (2.53).  $n_>$  denotes the quasiparticle population above the Fermi level and  $n_<$  the population below. These different populations have different charges. The different contributions to the total population,  $n_>$  and  $n_<$ , are not in equilibrium. This imbalance in charge gives a quasiparticle current [53].

To interpret the physical meaning of the experiment it is important to consider the quasiparticles and their charge imbalance inside the superconductor. In the experiments quasiparticles are injected into the junction. This causes a charge imbalance. A short time after heating, the quasiparticle population starts to decay [52].

If only energies of  $E > \Delta(T)$  are considered, the Boglioubov-de Gennes equations give the solution for the excitation spectrum as equation (2.54) [54]. The difference from BCS theory is the quadratic dependence on the energy gap. The quasiparticle decays rapidly into lower excited states with the mechanism of phonon emission. The quasiparticles recombine in pairs after decaying [53].

$$E_k = \sqrt{\epsilon_k^2 + \Delta(T)^2} \tag{2.54}$$

Phonons with  $E_k \ge 2\Delta(T)$  do not have any probability to achieve pairbreaking. Then the phonons increase the quasiparticle population even further, to the point where the quasiparticles excite even more phonons. It should be noted that the relaxation time of an excited quasiparticle in a superconductor is extremely short. Similarly at  $k_B T \ge \Delta(T)$ , a fraction of quasiparticles propagate into the superconductor. The quasiparticles in the superconductor dissipates power and this is seen as an interface resistance [53].

$$I_{inj} = \frac{1}{e R_{NS}} \int_{\Delta}^{\infty} n(E_k) \left( f(E_k - e V) - f(E_k + e V) \right) dE_k$$
(2.55)

The injected current into a junction can be seen in (2.55). Comparing that equation to equation (2.38), there is a difference in the Fermi-Dirac distribution by the applied voltage, (V) [53]. The injected quasiparticle current behaves substantially different from the *SIS*'-current; this will be shown in the results chapter.

#### Andreev reflection and oscillations in magnetoresistance

Quasiparticles can propagate into both the *S* and the *N* phase. As temperature increases from the heated junction, more quasiparticles travel into the superconductor. A consequence of this is that the boundary resistance between *N* and *S* increases [53]. The diffusive transport of quasiparticles gives rise to an oscillatory behaviour in the resistance of the interferometer. This is because of the change in  $\theta - \theta'$  [55].

The positive phase in the reflection,  $\theta$ , is from a electron and the negative  $-\theta'$  from a quasiparticle hole [56], as discussed in section 2.3. For this to be clearly visible in the experimental situation, the current needs to be subcritical or have condition (2.52). This thermoelectric current controls the phase gradient in equation (2.49). The gradient is closely related to Andreev reflection.

When a quasiparticle current goes into a disordered superconductor from a normal metal, a resistance arises from scattering processes inside the superconductor. If the amount of superconducting electrons fluctuates, this can lead to enhanced Andreev scattering. This enhanced scattering can cause quasiparticles to penetrate deep into the superconductor, before being reflected back into the normal metal [57].

The quasiparticle interference during Andreev reflection causes oscillations in the interferometers resistance [8]. Injected quasiparticles above the Fermi level can be reflected in the far side of the superconductor as a quasiparticle below the Fermi level. The two quasiparticles mix with each other despite being at different levels and cause interference [53].

Stoof and Nazarov investigated the magnetoresistance oscillations in 1996. They found that the resistance in their model oscillated with an modulation of around 10% near  $R_N$ . The oscillations depend on the phase and the coherence length, which is a function of temperature according to equation (2.34). This should according to Stoof and Nazarov hold in general for any geometry. When using a conventional proximity effect, they find that the magnetoresistance of a diffusive transport in normal metal should be phase independent [9].

The proximity effect has a re-entrant behaviour on resistance with respect to temperature. As mentioned earlier in the section, the energies of the quasiparticles are at  $E > \Delta(T)$ . In a superconducting loop this may also add to the conductance at high temperatures. In large loops the resistance gets enhanced at the energies discussed [58].

## Chapter 3

# Fabrication

The samples realising the bimetallic loop are fabricated in the cleanroom at MC2, Chalmers. The process, going from an empty silicon wafer to a circuit on the chip requires many steps and several different machines. The chemicals chosen for fabrication are allowed in the cleanroom, have the desired effect and are well known to the Bolometer group. This chapter gives an overview of the fabrication process including a brief introduction to the machines used.

Depending on the size of on-chip structures, different methods are used to create them. Large structures are created on the wafer before it is diced into individual chips, using the relatively fast *photolithography* process. Small structures, need to be created using *electron beam lithography*, since they are too small for photolithography.

Figure 3.1 shows the procedure necessary to prepare a clean chip (step A) for creation of large structures. A liftoff and a photoresist layer is applied to a silicon wafer by spinning and baking (step B). The wafer is exposed in a photolithography process. Thereafter, the photoresist layer is removed in chosen areas by development (step C). After *ashing*, a swift *dry etching*, the wafer is clean (step D) and ready for creation of large structures.

Figure 3.2 displays how the desired structures are *evaporated* onto the wafer (step E). The liftoff and photoresist layers as well as any evaporated metal in areas not exposed in photolithography, are removed in a *liftoff* process, rendering the wafer bare with the large metal structures (step F).

Figure 3.3 describes the preparations for creating small structures. A copolymer layer is spun onto the wafer and germanium is evaporated on top to create a hard mask. A *electron beam resist* is spun on top of the germanium layer (step G). To create small structures on the chips they are first exposed in electron beam lithography. Thereafter the wafer is scribed into chips. Each sample is then developed so that the exposed resist is removed (step H). For some wafers the scribing has been done before the preparation for the electron beam lithography.


**Figure 3.1** – Fabrication steps A-D: Preparation for large structures. A: A clean wafer; B: Liftoff and photoresist layers are spun; C: After photolithography, etching creates an undercut; D: Ashing cleans the wafer.

The now uncovered germanium is removed by etching, which also creates an *undercut* in the copolymer beneath (steps I-J). When two openings are sufficiently close to each other, etching will eat away all the copolymer between, thus creating a *bridge* as featured in step J. This bridge can also be seen in figure 3.4, which is a cross section too.

As figure 3.5 illustrates, the small structures are created by evaporation. The technique of using several metals and utilising several angles to create the desired structures (steps K-M, see also figure 3.6) is called *shadow evaporation*. Again using a liftoff process, excess metal films are removed together with the copolymer and germanium mask (step N, see also figure 3.7). The chip is now fabricated and ready for *measurements*.



**Figure 3.2** – Fabrication steps E-F: Creating large structures. E: Evaporation of Ti, Au and Pd; F: Liftoff.



**Figure 3.3** – Fabrication steps G-J: Preparation for small structures. G: Copolymer, germanium and electron beam resist layers are applied; H-J: Electron beam exposure and development followed by two kinds of etching creates the desired undercut (figure 3.4).

# 3.1 Photolithography

Photolithography can be used to make small structures, but not for the smallest structures of the design. The features fabreated with photolithography are shown in



**Figure 3.4** – 3-D view of fabrication step J. Colors are as in figure 3.3.

figure 4.3 on page 45 (section 4.2.2). The wafer is covered with a photoresist layer, which is sensitive to light. A photomask controls which areas will be exposed. The photoresist will react where exposed, leaving openings for evaporation after development. The layout is thereby transferred from the mask to the photoresist.

#### 3.1.1 Liftoff and photoresist

Before exposure a liftoff and a photoresist layer are applied, which can be seen figure 3.1 step B on page 27. There are two commonly used resist types: positive and negative. A positive photoresist becomes soluble in a developer liquid after exposure, while a negative photoresist becomes insoluble after exposure.

The choice of resist depends on the type of mask used for exposure. If the mask is the inverse of the desired pattern, thus blocking the pattern, a negative resist should be used. Alternatively, if the mask is the same as the desired pattern a positive photoresist is needed. Positive resists give improved control of small structures according to Jaeger [59]. This is the resist used during fabrication of the samples.

The liftoff layer and positive photoresist used in the fabrication are LOR3A and S1813 which are applied using a resist spinner. The thickness of the layer is inversely proportional to the square root of the spinning speed (see p. 22 in [59]) and proportional to the viscosity of the liquid. For a two-inch wafer the thickness of LOR3A is just over 360 nm [60] and the thickness of S1813 is  $1 \mu m$  [61],[62]. For more information about the



**Figure 3.5** – Fabrication steps K-N: Creating small structures. K-M: Metals are evaporated from different angles to create the desired structures, utilising shadow evaporation (figure 3.6); N: Liftoff uncovers the finished structures (figure 3.7).

processes read about the recipes in appendix A.

Directly after each layer is spun, the wafer is baked on a hotplate. This is done to improve adhesion and remove solvents in the liftoff and photoresist layers, see p. 22 in [59]. When the liftoff layer has been applied and baked it is important to directly apply the resist. If the wafer has been stored it must be carefully cleaned and dried before applying the resist, see p. 21 in [59].

#### 3.1.2 Exposure and development of the photoresist

In photolithography a mask consisting of glass with chromium protects the covered part from exposure. The masks are generally fabricated with optical or electron beam systems, see p. 28 in [59]. The masks used were made with electron beam lithography [62], where the layout was exposed directly in the electron beam resist.

To improve the resolution a vacuum is applied between the mask and the wafer before exposure. The improved resolution is due to the minimized gap between the mask and the wafer [63]. Each time a mask is used in direct contact with the wafer, the wear on the mask can damage the details in the features. This is why multiple masks often are



Figure 3.6 – 3-D view of fabrication step M. Colors are as in figure 3.5.



**Figure 3.7** – 3-D view of fabrication step N. Colors are as in figure 3.5.

fabricated, see p. 21 in [59].

The mask must be carefully aligned with the wafer, see p. 22 in [59]. When the wafer is

positioned in exact reference to the mask, the photolithography can start. The layout is transferred using high-intensity blue light. The layout includes positioning marks for the electron beam lithography, see section 3.2.3 on page 33.

When the desired layout has been exposed onto the chip, the resist must be developed. The development dissolves the exposed photoresist, exposing the underlying silicon. The wafer is immersed in a bath of Microposit MF-319 developer [62]. Since positive resist is used, any resist that has been exposed is washed away with the developer. The underlying silicon is then uncovered, as shown in figure 3.1 step D on page 27. The development process is described in appendix A.

After the development, the wafer is ready for evaporation. For further reading about evaporation see section 3.4. The bigger parts of the layout are then constructed. Due to the resolution the smaller parts are left for electron beam lithography.

## **3.2** Electron beam lithography

Deep sub-micron scale features are too small for traditional photolithography in the MC2 Cleanroom [62]. Another technique is therefore required. In 1964 the first structures of electron beam lithography with linewidths beyond the capability of photolithography were produced [64]. Electron beam lithography takes considerably longer time to use than photolithography; one reason is the small beam size used to get the better resolution.

The electron beam technology uses accelerated electrons fired at the sample. The sample is covered with a layer of resist. Selected parts of the resist are exposed and the features are revealed on the wafer after development [65]. The features written by the electron beam lithographer can be seen in figure 4.4 on page 46 (section 4.2.2).

#### 3.2.1 Copolymer and electron beam resist

Copolymer is used as a liftoff layer, similar to S1813 in photolithography. It works by expanding when put in remover solvent, causing the germanium to break.Further, the liftoff is more effective with the layout in figure 4.2 on page 43 (section 4.2.1). To the top left in the figure, dots are shown. These will be exposed by the electron beam lithography. The liftoff liquid will enter these dots and thereby help the liftoff.

Electron beam lithography exploits electron scattering in the resist. The fabrication recipe uses PMMA as the electron beam resist. Electrons penetrating the resist at a perpendicular angle induce an inelastic type of scattering, called forward scattering. The scattering is modelled by a narrow Gaussian distribution. While using a thin resist layer and a high accelerating voltage, the width of the distribution can be neglected [64].

In contrast backscattering electrons are scattered elastically. It mainly occurs in the

copolymer beneath the resist. Backscattering is the dominating mode of exposure when a high acceleration voltage, such as 50 kV or 100 kV, is used [64]. In the fabrication process these two voltages have been used.

The incoming electrons have too much energy to expose the resist. After being scattered several times their energies are low enough to expose the resist. This is the type of exposure primarily used during fabrication. The exposure can be modelled well with a Gaussian approximation [64].

#### 3.2.2 Resolution limits and error sources

The resolution limit of electron beam lithography depends mainly on the resolution of the resist on the wafer and on the following development process [64]. The resolution reached with PMMA is 20 - 25 nm [62]. The resist dependence is partly due to the molecular structure of the resist. It also depends on the Coulomb interaction between the beam electrons and the resist molecules [66]. The resolution is highly improved with increased acceleration voltage.

If the exposed area is wider than the feature, the surrounding resist will also interact with the electron beam [67]. This effect is known as the electron beam proximity effect and can result in a variation of exposure between the features or inside them. The effect deteriorates the features, which can even merge together. Backscattered electrons cause the effect by being scattered away from the incident beam [67].

The proximity effect is one of many problems in writing small features. Electron beam lithography is also affected by drift and astigmatism. For the fabrication these error sources are even bigger than the proximity effect, but are easier to correct. The drift is caused by movements inside the electron beam lithography machine. This can be compensated for during the calibration [62].

The astigmatism is a type of aberration and occurs when electrons that propagate in perpendicular planes have different focus. The shape of the beam will be deteriorated by the astigmatism and the electrons will not be projected correctly [68]. In calibration of the electron beam the astigmatism can be reduced.

#### 3.2.3 The electron beam lithography machine

The electron beam lithography machine used in the fabrication of the small features is the JBX-9300FS and is further described in appendix D. It focuses and shapes the beam using magnetic lenses and deflection coils. The desired layout is designed using a CAD program [69].

Before exposure many control systems have to be used, including control of displacements and height. The accuracy is measured utilising a laser [69] and the

difference of the actual beam position and the designated position is calculated. The error is subsequently forwarded to the electron beam for correction of its position.

The sample is placed on a workstage, equipped with detection marks for calibration. It is detected as a peak in the backscattered electron intensity [69]. The errors from the drift are calibrated from these marks [62]. Moreover two electron-absorbing detectors exist on the workstage. These enable measurement of the beam size and therefore automatic adjustment of the focus can be performed [69].

After focusing on the workstage, the positions of the samples need correction. During photolithography positioning marks have been written into the samples. In electron beam lithography they are used to correlate the distances on the chips and calculate where to direct the beam.

# 3.3 Etching germanium and undercut

The two main etching techniques are wet chemical etching and dry etching. The etchant has to remove the unprotected layer faster than it removes the resist, see p. 25 in [59]. Accordingly the selection of technique and chemicals is important. Wet etching requires a large amount of chemicals relative the amounts of gas required for dry etching, see p. 26 in [59]. The chemical processes are isotropic in direction.

In contrast to the wet etching, dry etching can obtain highly anisotropic profiles in thin films, see p. 26 in [59]. The germanium layer, seen in figure 3.3 on page 28, has to obtain sharp edges and therefore requires a highly anisotropic etching profile. The fabrication process involves dry etching for both the germanium layer and the undercut. The etching rate depends on the temperature, thus temperature control is important during the process, see p. 25 in [59].

After etching, lines and openings in the resist layer have the desired undercut and the chip is ready for evaporation. It is important to protect the sample from liquids and dust before evaporation. The features are not formed properly if the undercut is destroyed or the bridges are broken.

#### 3.3.1 Dry etching

Dry plasma etching systems ionize the etchant gas,  $CF_4$  or  $O_2$ , in a vacuum using radio frequency excitation at 13.56 MHz, see p. 26 in [59]. During fabrication the Oxford Plasmalab System 100 is used to etch the germanium layer. Then Plasma Therm BatchTop PE/RIE m/95 is used for isotropic etching of the undercut [62]. Both systems are reactive etching systems using a radio frequency power source [70],[71].

Reactive ion etching or RIE, is highly anisotropic when applying an electric field. The

plasma ionizes the reactive gases and the ions accelerate towards the surface where the sample is placed. Both chemical reactions and collisions can contribute to the etching, see p. 26 in [59]. During fabrication, only chemical etching is used, not physical. This is due to the low energies used, which are chosen because they are less destructive [62].

The plasma is generated independently in the upper part of the chamber and near the lower electrode. The process in the upper part of the chamber has capability to generate a high density of ions and radicals for a high etching rate. The directional anisotropic etching is provided by the electrical field near the bottom electrode [70].

The Oxford Plasmalab System 100 has two chambers intended for different processes. The first is designated for chlorine, methane or hydrogen based processes. The other chamber is used for fluorine based processes and deposition of silicon, silicon dioxide and silicon nitrite [70]. For germanium etching,  $CF_4$  is used in chamber two. The Oxford Plasmalab System 100 etching process etches mostly downwards, which is preferable for the profiles of the edges of the germanium. Sharp edges result in a more precise layout of the evaporated metals.

The Plasma Therm BatchTop PE/RIE m/95 system is designed for ashing of photoresist and electron beam resist. The gases used are oxygen, tetrafluoromethane, argon and hydrogen [71]. During the fabrication process it is used for creating undercut and for ashing, both after photolithography and electron beam lithography.

The undercut is a cavity leaving a greater area of silicon uncovered than the corresponding area of the opening in the germanium layer, see step D in figure 3.1. The oxygen plasma reacts with germanium, creating germanium oxide which is solid and stays where it is. It also reacts with the copolymer, but since the copolymer is carbon based, the products are gaseous and are pumped out. This leaves a cavity known as an *undercut*.

# 3.4 Evaporation

*Thin film evaporation* is the process of letting a thin film form on a sample by evaporation. This is done in a vacuum chamber where evaporated metal is condensed on the chip, see p. 95 in [72].

In our process, evaporation is used three times. First, it is used to create the large structures; contact pads and large connection lines. Before the electron beam process, it is used to create the germanium mask for the small structures. Last, it is used after the electron beam process to evaporate metals through the openings in the germanium mask. This creates small structures directly on the silicon surface. The copolymer allows removing of metal film formed on areas where the silicon surface is thus covered.

#### 3.4.1 Shadow evaporation

*Shadow evaporation* is a technique of using varying evaporation angles to control on what areas on the chip the film is formed. It is made possible by the undercut created in the etching processes. Different metals can be condensed onto the chip without having to expose the chip to oxygen between evaporations. This is a great benefit since oxygen reacts with metals, creating insulating oxide layers, see [62] and p. 134 in [59].

Shadow evaporation makes it possible to evaporate several metal layers forming different structures on the chip. All metals can be evaporated in the same vacuum cycle. It is therefore unnecessary to repeat the long-lasting vacuum pumping and lithography processes for each metal. Since this process involves fewer steps it is also less prone to failure [62].

#### 3.4.2 The chemistry and physics of evaporation

The evaporation has to be performed in a vacuum in order to avoid chemical reactions and physical collisions with air. Chemical reaction with air would prevent an unoxidized layer of the metal to form in the desired area. Physical collisions would prevent the atom clusters to travel without change in direction, which would distort the evaporated pattern [62].

The *source* is a bulk amount of the metal to be evaporated. It is placed at a certain distance from the chip, where the thin film is to be formed, see p. 95 in [72]. The chip is then rotated to a certain angle to allow shadow evaporation.

The source metal is caused to evaporate, by resistive heating or electron beam exposure. The gaseous metal close to the source will have a pressure greater than the surrounding vacuum and small clusters of gaseous atoms will therefore begin to move in all directions from the source. By the time the atom clusters get close to the chip, the pressure is sufficiently low to disregard all interactions between them, see pp. 132-134 in [59].

Due to the high vacuum, the atom clusters will be mostly chemically intact when they hit the chip. They will hit the chip traveling in a direction straight from the source. Therefore, they will hit the chip at a point that is geometrically reachable from the source as shown in figure 3.3. See also figure 6.5 on page 135 in [59].

Due to the geometry, certain areas of the uncovered silicon are not geometrically reachable from all angles. This makes it to some extent possible to control what areas are covered with what metal films, when evaporating several metals in one cycle.

Small clusters of metal atoms hitting the silicon surface will condense on the chip. They will also condense on other clusters already on the chip, tending to organize themselves into the polycrystalline structures intrinsic for the metal in question [62].

# 3.5 Inspection of samples

To produce a fully working chip many different procedures have to be successful in all steps of the fabrication. An error in one step can result in an unusable chip and the following fabrication steps will be worthless. For instance if the photoresist is uneven, the photolithography exposure will vary in different parts of the wafer. This can result in features that do not match the desired layout.

The fabrication process is very time-consuming and therefore the inspection will minimize unessential work. The information received from inspection can be used to rationalise the processes to get as many working samples as possible. Some errors that are detected can be corrected with an additional fabrication step. In the example the photoresist can be removed and replaced.

The *optical microscope* and the *scanning electron microscope* are suitable for examining the surface of chips. The optical microscope is used during the fabrication for inspection after important steps. In contrast, the scanning electron microscope is highly destructive and therefore not appropriate before measurement of the sample. The scanning electron microscope is useful after fabrication if a chip gives unexpected results.

#### 3.5.1 Optical microscope and fabrication process inspection

There are two types of optical microscopes: bright field and dark field. The principle of imaging in the microscope is that the surface materials reflect light of different wavelengths. The bright field, utilising light perpendicular to the sample, is usually used, see p. 37 in [59].

If some features reflect light in similar ways to the surroundings, they may not be seen in the bright field. These features can be clearly observed with dark field microscopy. This is because the surface appears dark with bright features. For inspection with dark field the sample is illuminated from an oblique angle, see p. 37 in [59].

The resolution for typical optical microscopes is 250 nm, see p. 37 in [59]. With a maximum magnification of a thousand times, the smallest parts of the layout cannot be seen. The smallest features have widths of 40 nm [62]. Though they cannot be seen, they can interfere with the surrounding materials and the interference can be observed.

An example optical microscope picture is shown in figure 3.8. Parts of the bimetallic loops are visible in figure 3.8(a), where the thin superconductor (blue arrow) can be observed. The Andreev interferometer (orange arrow) can be observed in figure 3.8(b).

Through changing focus, letting the focus fall at different heights, the features widths are revealed. The undercut would show wider lines, when the focus is closer to the silicon wafer, and get thinner closer to the top of the mask layer. The size of the undercut can be thereby be approximated.



(a) The upper part of the primary bimetallic loop is shown with a yellow arrow and denoted with a P. The secondary bimetallic loop is marked with a red arrow and S, while the thin superconductor is marked with a blue arrow.



(b) The Andreev interferometer is shown and marked with an orange arrow.

**Figure 3.8** – With inspection in the optical microscope these figures of BL3-43 are received with highest magnification. The upper parts of both of the bimetallic loops are visible in 3.8(a). The Andreev interferometer (orange arrow) can be observed in figure 3.8(b).

The optical microscope is a non destructive inspection method, so it can be used continuously through fabrication, see p. 37 in [59]. If some structure is detected to not be fully formed, this can be compensated in the next fabrication step. If a sample has uncorrectable errors, the sample can be discarded.

#### 3.5.2 Scanning electron microscope

The scanning electron microscope works similarly to the electron beam lithography machine. As in electron beam lithography the surface of the sample is bombarded with electrons with energies in the range 0.5 - 40 keV, see p. 37 in [59]. The picture is formed by scanning the surface with the incident electrons and detecting secondary electrons.

Because of the interest in the surface topography, detection of secondary electrons are used for inspection. Secondary electrons are generated when the incident electrons ionize sample atoms. The magnitude of the current depends on the material, but mainly on the curvature of the surface.

The scanning electron microscope is destructive and may destroy the samples while inspecting them. Because of the destructiveness, it is never used before measurements on the samples [62]. The microscope can give information about why the chip was not working and if the fabrication process needs to be changed.

The maximum resolution of the scanning electron microscope is about 2 - 3 nm corresponding to a magnification up to 300 000 times, see p. 37 in [59]. The resolution is much better than in the optical microscope. The features on the sample are clearly visible in the scanning electron microscope. Accordingly the scanning electron microscope is highly appropriate for analysing why the chip gives unexpected result

from measurements. If an error is detected, the fabrication process can be corrected.

An example scanning electron picture is shown in figure 3.9. The smallest features of the layout can clearly be seen and measured using the length scale. The shadow evaporation is visible as shadows of the interferometer Y-shape (orange arrow). Another shadow is visible as an error, which will be discussed further in section 6.1 on page 54.



**Figure 3.9** – With inspection in the scanning electron microscope the Andreev interferometer (orange arrow) and the thin superconductor (blue arrow) is clearly visible. The red arrow points out a parasitical structure which is created during shadow evaporation, it has no function. Above the thin strong superconducting line a shadow is visible, which can destroy the result of the sample. The scale is shown in the lower part of the picture.

# **Chapter 4**

# **Experiment realisation**

In order to realise the experiment, theoretical and practical limits were identified and overcome. These requirements are discussed in section 4.1. The proposed and used solutions that enabled the experiments are the chip setups described in section 4.2. The samples and the actual experiments were realised as described in section 4.3.

# 4.1 **Requirements for the experiment**

In order to observe the thermoelectric effects described in section 2.4 the experimental setup has to include superconducting bimetallic loops and some form of readout. To achieve superconductivity the setup has to be cooled below the critical temperature  $T_c$ . For aluminium in bulk form  $T_c = 1.2 \text{ K}$  [73], but in thin films the  $T_c$  can be higher [50],[73]. Nevertheless, it is desirable to reach as low temperatures as possible, to minimize the thermal noise. In order to achieve temperatures below 1 K a cryostat is used.

Smaller, controlled geometries improve accuracy. The project aims to fabricate an onchip experimental setup, so it should be possible to reach higher accuracies than the former bulk material experiments described in the introduction chapter. To further improve the precision the readout is placed on the chip. The readout is made in the form of an Andreev interferometer.

The experiment also requires the possibility to induce a temperature gradient, see section 2.4. There should preferably be a way to measure the temperature gradient as well. To test the dependence of the thermomagnetic field on external magnetic fields, there should also be some way to apply and control an external field. To improve the control characteristics these parts should also be placed on-chip. All parts on the chip, from the readout to the magnetic control, should be accessible from macroscopic measurement instruments and control electronics. This can be done using on-chip contact pads and

connections inside the cryostat.

The limits of different fabrication technologies are described in the fabrication chapter. The smallest possible feature sizes are on the order of 20 nm, so this creates a limit as to how small the setup can become. In practice, differents parts of the setup must have their individual dimensioning. This is described in more detail in the next section.

The arm length *L* of the Andreev interferometer has to be shorter than the coherence length  $\xi$  of the superconductor [7], as noted in section 2.3. To allow for error margins  $L \ll \xi$  is a reasonable condition. For aluminium  $\xi_{Al} = 1.2 \,\mu$ m [74], so a reasonable arm length is around 100 nm.

The interferometer has to be made in a conducting metal that remains in its normal state at the temperatures considered [7]. For temperatures on the order of 300 mK silver and titanium are two possible choices. Titanium actually has a critical temperature of 400 mK [73], but the superconductivity can be suppressed using the proximity effect by placing a metal with a lower critical temperature in contact with the titanium layer, see p. 22 in [75]. Furthermore, due to the evaporating machine used, Edwards Auto 306, it is not possible to achieve superconducting titanium at all [76]. Titanium can therefore be used as a normal metal by itself.

To achieve the Sharvin resistance in the interferometer there is also the condition that the arm width *W* must be shorter than the mean free path  $l_e$  in the interferometer metal [43]. For silver we have  $l_{e,Ag} = 52 \text{ nm}$  [45]. With the limits of fabrication taken into account we arrive at 20 nm < *W* < 52 nm and that the interferometer must be fabricated using electron beam lithography. We have not been able to find experimental data for the mean free path of titanium, but conjecture that it is similar to the value for silver. Samples with titanium have been tested, as well as samples with both titanium and silver.

# 4.2 Chip description

The general idea of the chip's function can be seen in figure 4.1. The figure shows which parts are required on the chip, as discussed in the previous section. There are two bimetallic loops, the primary and the secondary. The secondary loop allows for interaction experiments, to test the effect in the primary loop from currents in the secondary. There is also a magnetic induction line, to allow measurements of the dependence on external fields.

Furthermore there are *SIS'* junctions connected to both loops, to allow heating of parts of one or both loops. They also allow temperature measurements (see p. 1885 in [77]) and are useful when screening samples. The interferometer is connected to the primary loop, allowing phase shift measurements in the loop. The interferometer, induction lines and junctions are connected to contact pads, enabling signal transfer to and from macroscopic measurement equipment.

Together these features should meet the general requirements in section 4.1. The parts



**Figure 4.1** – A schematic overview of the chip functions and the interaction between the different parts. For example, the phase in the superconductor of the primary bimetallic loop affects the resistance in the Andreev interferometer. The resistance is measured using the relevant contact pads.

Table 4.1 – Number of important parts for the experiment on the chip layout

Number of parts							
16	Contact pads						
16	Connection lines with interfaces						
2	Bimetallic loops						
10	SIS' junctions						
2	Weak links						
2	SN-contacts						
1	Magnetic induction line						

of the chip are described in more detail in the next section, along with the chip layout. The differences between the existing layout designs are described in section 4.2.2. The specific parts are of course subject to be manufactured in a way as to meet the other requirements. The realisation of the parts is described in section 4.3.

#### 4.2.1 Chip overview

Figure 4.2 shows the chip in different zoom levels. There is an overview of the entire chip, an overview of the loop circuit, the primary bimetallic loop and the *SIS'* junction numbering. This numbering corresponds to the contact pad numbering. Table 4.1 lists the different important features and their quantities in the layout.

The outer rim of the chip consists of contact pads. They are numbered clockwise from 1 in the upper left corner to 16, as shown in figure 4.2. The pads are large and can be seen with the naked eye. Each contact pad is connected to a specific feature on the chip. The magnetic induction line, is for example connected to contact pads 10 and 11. The pads and corresponding connection lines are marked with green in figure 4.2.



**Figure 4.2** – The chip in different zoom levels, with important parts pointed out. To the top left the entire chip is shown, with the contact pad numbering. The second picture shows an overview of the loop circuit and the third shows the primary bimetallic loop. The bottom picture shows the junction numbering.

Thick connection lines leave the contact pads to connect to inner chip functions. They are denoted in the figure in green color, like the rest of the contact pad layer. Closer to the bimetallic loops, they become black in the layout and this means that they are getting a lot thinner and are part of a different fabrication layer. The green lines are  $\approx 20 \,\mu\text{m}$  wide and the black  $\sim 1 - 2 \,\mu\text{m}$ . The line width of the black lines depends on the specific layout used. See section 4.2.2 for the differences between the used layouts.

In figure 4.2, the wafer ID number is also seen. It is a fairly large structure which denotes with the first letter what kind of circuit is on-chip and a number corresponding to model number. These ID numbers may seem inconsequential, but are very important to follow up the different fabrication paramters and conducting quality control of fabricated samples.

There are other parts also visible in figure 4.2. For instance there is the dot array, which can be seen in the little black dots, uniformly dispersed over the chip. They help with the germanium mask liftoff by making it more efficient and therefore taking less time. Without the dots, there is a considerable difference in time during liftoff. Liftoff is covered in more detail in section 3.2.1 on page 32.

The circuit includes two bimetallic loops, both having 5 *SIS*' junctions at the topside. The junctions of the primary loop connect to contact pads 3, 6–9, while those on the secondary loop, placed to the left, connect to 1,2,14–16. The junctions and their numbering are shown in figure 4.2.

Each loop has an additional feature, which is a weak link. The weak link in the secondary loop is not connected to any contact pad, while the one in the primary loop is connected to both contact pad 4 and 5. They are important as they establish a difference between the two loop sides, in effect making the loop a bimetallic loop. *SIS'* junctions and weak links are discussed more in section 2.2.2.

By running a current between two junctions a heat gradient is established. Quasiparticles are injected into the loop as a result of the gradient, as described in section 2.4.3. There are both small and large junctions, which leads to different resistances. The different junction sizes allow symmetrical and asymmetrical quasiparticle injection. Junctions 14, 15, 2, 3, 6 and 9 are regarded as small, while the other *SIS*' contacts are large.

The properties of the *SIS'* junctions depend on the temperature, as seen in equation (2.39) on page 16. This means that the junctions can be used for thermometry measurements [77]. This is useful for the experiment of the century, where a temperature gradient is established by means of two junctions. The gradient can be estimated using the current and models for the quasiparticle current, but using two more junctions the gradient can be measured directly.

The primary bimetallic loop has an area of  $1296 \,\mu\text{m}^2$  and the secondary  $1380 \,\mu\text{m}^2$ . The ring from the top to the bottom of the primary loop and the L shape are weak superconductors. Otherwise there would not be any phase difference because of different superconductors in the *SIS'* junctions, which is needed for the creation of quasiparticle-currents, see section 2.4.3.

The temperature gradient in a loop gives rise to a phase difference  $\theta - \theta'$ . This can be measured in the Andreev interferometer, as has been discussed in section 2.3 on page 19. The current from the *SIS'* contacts which travels throughout the loop will be affected by the thermomagnetic field and the generated thermoflux. This will be read out in the interferometer. Our experimental goal is to measure the dependence of the generated thermomagnetic field with the external applied magnetic field. An external field is induced from the magnetic induction line, connected to contact pads 10-11. The induction line is basically a very robust conducting line.

The Andreev interferometer is beneath the primary bimetallic loop and denoted in red in figure 4.2. It can be seen in more detail in figure 4.5 as a red Y-shaped structure. The interferometer is 515 nm long in this design, which correlates to being 57% smaller than the coherence length of aluminium. The arm width is 40 nm, which is 23% less than the mean free path of silver at  $T \sim 300mK$ . Since these dimensions are very small this structure is extremely delicate. If it breaks it is impossible to conduct the experiment, as the sample would lack readout.

#### 4.2.2 Layout comparisons

The chips have been made according to two layouts by Leonid Kuzmin, the BL3-2 and the BL3-3 designs. Figures 4.3(a) and 4.3(b) show the overview of the Bl3-2 and BL3-3 CAD designs, respectively. They operate the same way, but the BL3-3 layout is designed to improve reliability and repeatability in fabrication. The lack of dot array in figure 4.3(b) is not final, it is created later in a script controlling the electron beam lithography machine.



Figure 4.3 - Overview of the BL3-2 and BL3-3 designs.

The other difference visible on this scale is in the contact pads. To ensure connectivity between the contact pads, which are 150 nm high and the connection lines, which are 15 nm high, an extra layer of 70 nm is evaporated on top. The change should drastically improve the chance of good connection in the pad-line interface.

Furthermore, the contact pads have been observed to "roll up" one end, effectively cutting the contact between the two metals. The chance of that happening is decreased by reinforcing the connection and making it thicker. Then the contact pad would have to "roll up" a lot further.



**Figure 4.4** – The primary bimetallic loop, with associated junctions and the Andreev interferometer, in the BL3-2 and BL3-3 CAD designs. The connection lines are reinforced in the BL3-3 design (b).

On a smaller scale the two designs are compared in figures 4.4(a) and 4.4(b). It is clear that the connection lines are reinforced, as to improve reliability and decrease sensitivity to fabrication errors. This also lessens the number of potential infinite resistances.

On a yet smaller scale the Andreev interferometers of the two designs can be compared. For the BL3-2 design see figure 4.5(a) and for BL3-3 see fig. 4.5(b). Both the interferometer and the *SIS'* junctions are reinforced to further decrease sensitivity to fabrication errors and improve reliability. The interferometer connection line is also substantially reinforced.

## 4.3 Realisation of the experiment

The samples were made according to the layouts described in section 4.2, using the fabrication techniques described in the fabrication chapter. Contact pads and main connection lines were made using photolithography, see section 3.1. This technology was in fact used for all parts visible to the eye in the CAD layout overviews, see figure 4.3. The *SIS'* junctions, bimetallic loops, smaller connection lines and the Andreev interferometer were made using electron beam lithography, see section 3.2.

After lithography, etching was done to achieve undercut and allow for shadow evaporation. See section 3.3 for details on the etching and section 3.4 for details on the



**Figure 4.5** – The Andreev interferometer in the BL3-2 and BL3-3 CAD designs. The new design has larger contacts between small and large structures.

evaporation process. The fabrication recipes is available in appendix A. See appendix B for a list of the fabricated samples and their specific fabrication parameters.

Besides the differences in layout outlined in section 4.2 there were also differences in fabrication technology. The angle for shadow evaporation was changed to avoid problems with debris beneath the germanium mask. An additional thick layer was evaporated on top of the structure to reinforce the connection between contact pads and lines.

During the entire process the samples were continuously inspected and screened using the inspection techniques described in section 3.5. The samples that made it through the process were then measured electrically at room temperature according to the process of section 5.1 to test for basic conductivity. In the next step of the measurement process, chips with satisfactory readings were placed in a cryostat and measured at room temperature again to ensure the basic function. The results from the fabrication process and the process development are provided in section 6.1.

The samples were then cooled to subkelvin temperatures, where they were characterized electrically according to section 5.2. At these temperatures the actual experiment was also conducted and it was possible to test the thermoelectric effect using the magnetic induction line. The results from these bimetallic loop measurements are provided in section 6.2.

# Chapter 5

# Measurements

After the many steps of fabrication and inspection with the optical microscope, the promising samples were measured. The experimental data can provide information that proves or reforms the theory. It is important to use an appropriate measurement setup and technique.

The measurements were done with two different setups in room temperature and with two setups in the subkelvin regime. In the first room temperature measurement the samples were put in a "pen"-setup. If the measurement results indicated that the sample was not broken, the second room temperature measurement was performed. The second room temperature measurement used the same sample holder as used in the cryostat for the subkelvin measurement. The second room temperature measurement is important, because it confirms that the pads on the sample are in contact with the cryostat sample holder.

The sample holder was then loaded into a cryostat, the subkelvin measurement setup. Two different measurements has been done in the cryostat; one to receive the current-voltage curves and one with a induced magnetic flux. This chapter describes the different measurement methods and the information that we were interested in. For further reading see appendix C and D. Appendix C describes more technical information about the techniques used, while appendix D describes the cryostat used.

## 5.1 Room temperature measurements

The room temperature measurements are important for selecting promising samples to place in the cryostat. The cryostat takes long time to load and is expensive to cool down. Therefore the room temperature measurements makes the selection more effective.



**Figure 5.1** – A DC voltage sweep was applied over the bias resistance  $R_{Bias}$  and desired junctions on the chip, to create a current through the sample. The output voltage and the sweep voltage were measured (green loop) to provide I-V data (marked in blue). The I-V measurement indicates if the junctions work according to the requirements.

The setup consists of a pen, where the sample was placed, and a holder for the pen, connected to an amplifier. The setup has a sweep generator and an oscilloscope. The voltage was measured with a voltmeter and can be converted into current (for a known resistance). Figure 5.1 shows an overview of the the setup and the connection between the equipment. The sweep generated a current sweep and the bias resistance was tuned in the amplifier holder, see p. 39 in [78]. This electrical setup was also used for the second room temperature and first subkelvin measurements.

The pads on the sample were connected to the amplifier trough sixteen Pogo pins on the pen holder. The samples can be measured with two, three and four points in the setup. To measure voltage and current over the sample four wires were used. Depending on what lines to measure and what type of measurement to conduct the wires were connected in the pen holder. The voltage was either measured over the same or other contacts than the current.

If the connection was the over the same contacts, a *two point measurement* was performed. In figure 6.6 on page 60 (section 6.1) this can be exemplified as a measurement over 6 and 7. The resistances, measured then includes both the connection lines and junctions.

If the pins had one contact in common, it was a *three point measurement*. The current can then be measured over for instance 6 and 7, while the voltage can be measured over 7 and 8. This resulted in a measurement only including the resistance of connection line 7 and junction 7.

The tunnel junctions on the samples had resistances in the order of magnitude of  $k\Omega$  and was therefore difficult to distinguish from the resistances in the connections lines. When a *four point measurement* was performed, utilising four connections in figure 6.6, only the junction resistance was measured. Thus a *four point measurement* was more convenient [62].

The measurement in room temperature gave information about the electric character-

izations of the metals on the sample. The information was useful to detect errors like broken lines or short circuits. However, it could not be used to conclude that the sample will result as expected.

Some of the detected errors could be corrected, like the broken lines. The lines were repaired with silver paste and then measured again. If the errors were too major to be corrected, the scanning electron microscope was used to analyse why the errors occurred. The information that could be received by the scanning electron microscope is described in section 3.5.

When the room temperature measurement of a sample indicated that it had working connections, it proceeded to the next room temperature measurement setup. The measurement was made in the same sample holder as later used in the cryostat. This measurement confirmed whether the connection between the sample and the setup was correct.

The samples were easily damaged while loading them into the measurement setups. Static electricity from humans can burn connection lines and result in an unusable sample. Both the sample and the person in contact with it were connected to ground to prevent this from happening. Furthermore, the connection lines can be broken by scratches from tweezers used to hold the sample. These damages is unnecessary because they can be avoided by being careful.

## 5.2 Subkelvin temperature measurements

For the subkelvin measurements of the samples, the temperature was held just below 300 mK, often at 280 mK. This temperature is called the base temperature. At the base temperature the resistance of the metals and junctions on the samples differ from room temperature. This is according to Mattihiessen's rule [79].

The result of the change in resistance is illustrated in figure 2.3 on page 17 (section 2.2). The curves in the figure represent the current - voltage (I-V) characteristics at different temperatures. By heating the sample a few hundred mK and then cooling it back down to base temperature, I-V curves could be measured at many different temperatures. The result could then be compared with the I-V characteristics in figure 2.3.

The electric characteristics of a sample determined whether it was useful or not. If it was comparable to figure 2.3, the most interesting measurement was conducted of the sample: with an applied magnetic field. For this measurement a different electric setup was used.

#### 5.2.1 Measurements with an applied magnetic field

The measurements with applied magnetic fields are illustrated in figure 5.2. In contrast to the overview of the I-V setup in figure 5.1 there are two voltage sweeps, one induced magnetic field and a lock-in amplifier. In this case the current data is from the green loop and the voltage data is from the red loop.



**Figure 5.2** – A DC voltage sweep was applied over the magnetic bias resistance  $R_{Magnet}$  and desired junctions on the chip, to create a current and magnetic field (green loop). Simultaneously, an AC voltage was applied over the bias resistance  $R_{Bias}$  and desired junctions. The output voltage is amplified and put through a lock-in amplifier (red loop). This voltage and the magnetic current provide the I-V data (marked in blue) for the LabVIEW program.

The induced magnetic fields are weak compared to the critical magnetic field of aluminium [50]. The measurement setup can be placed in a magnetic shield. The data from measurements without a magnetic shield has a high level of ambient noise.

The magnetic fields were induced by applying a DC voltage sweep over a bias resistance  $R_{Magnet}$  and two contact pads on the chip. These contact pads connected to the magnetic induction line (described in section 4.2) for the experiments that test the resistance modulation of the Andreev interferometer. For the experiments on the thermoelectric effect in the loops and the interaction experiments the sweep was applied over the relevant junctions. The current and hence the magnetic field strength were controlled by adjusting the sweep length or the bias resistance. The voltage over the contact pads was measured and sent to LabVIEW.

An AC voltage sweep was applied over the bias resistance  $R_{Bias}$  and contact pads 5–13. Contact pad 5 connects to the weak link of the primary bimetallic loop and contact pad 13 to the interferometer. The sweep resulted in a current through the loop and the interferometer. The voltage over junctions 4 and 12 was amplified 1000 times and sent

to a lock-in amplifier. This is a four-point measurement.

The output voltage was mixed with the AC voltage in a phase-locked loop to decrease the noise. The phase-locked loop has been tested with different frequencies and has proved to show low interference at frequencies of 135 Hz and 5 Hz. Therefore these frequencies were used. The voltage was measured after the amplifier had locked in and sent to LabVIEW. There is an additional gain of 20 times in the lock-in amplifier.

### 5.3 Cryostats

Cryostats are vessels constructed to provide cryogenic temperatures for experimentalists for prolonged times, often hours to days or months. Both cryostats used in the experiments, Triton<sup>™</sup>DR and Heliox<sup>™</sup>AC-V are closed-cycle cryostats. These utilise pulse tube refrigeration technology and are covered in more detail in appendix D.2 on page 91.

The heat produced by currents through the sample can to some extent be removed by the cooling power of the cryostat. *The heat budget* is an overview of dissipated heat by resistances and junctions on the chip [76], which become important for prolonged measurements.

The Heliox<sup>TM</sup>AC-V has a base temperature of 300 mK, and a cooling power of 100  $\mu$ W at 350 mK [80]. The Triton<sup>TM</sup>DR on the other hand has a cooling power of 200  $\mu$ W at the 400 mK-stage and 200  $\mu$ W at the 3 K stage. This enables more complex samples and measurement equipment or a lower temperature [81].

#### 5.3.1 Problems with measuring at subkelvin temperatures

To measure the samples in the cryostat, connections from the sample to the outside equipment are required. The wires used for this conducts heat and therefore heat were produced in the system. The wires also transfer noise to the sample, and therefore additional contribution of heating of the sample.

A solution is to use wires with low heat conductivity, but these materials also have low electrical conductivity. The solution to the two problems therefore is optimised. Because of this limit the cooling power in the cryostats is low. Since the cryostats utilise different stages, the heat load was minimized system wise to maintain the low temperature, see p. 56 in [82].

To minimize the effects of heating, the wires in our cryostat setup are wired in twisted pairs. In a wire with twisted pair, each induced current gets cancelled by the next twist in the cable, see pp. 57-58 in [82]. This does not result in the induced current, which would be produced in a straight wire and heat the system.

In addition, the heat effects from the wires are reduced with heat sinks. The wires are bound in spirals on thermal dumps, which works as heat exchangers, see pp. 58-59 in [82]. Heat sinks are also used when cooling the system as described in D.2 on page 91.

The problems with the heating have effected the measurements on the samples. Many times the measurements have had to wait for the cryostat system to cool down again before continuing. When the system was heated too much during a measurement the results were effected because of the resistance characteristics of the metals on the sample.

# Chapter 6

# Results

The chapter presents the results from the two main areas of interest, the fabrication of samples and the experiment on the bimetallic loop. Fabrication results are discussed in section 6.1 while the bimetallic loop experiments are discussed in section 6.2. The progress with the Andreev interferometer is discussed in section 6.2, where the amplitude modulation and quantization of its resistance are discussed.

The fabrication process has changed through the project. The geometry of the sample together with materials have been the major changes. This section will cover the changes of the fabrication process, the design changes, discuss materials and their thickness. The different factors all come together in the measured resistance of each samples junctions.

Figure 6.4 shows a broken interferometer from the BL3 wafer with titanium as normal metal. In figure 6.3 an overview of the circuit is visible, with important features marked. Each sample which was measured at room temperature is displayed in figure 6.1, providing an overview of all measured samples.

# 6.1 Fabrication

When a new wafer is actuated, the photolithographic process is applied using the same parameters as for other wafers. Regardless, two samples from the same wafer turn out with different results, even when fabricated with identical materials, etching times and development times. Comparing samples is perilous since conclusions must be drawn from so many different parameters, but can still give indications on how to change the process.

Junction resistances are measured at room temperature using two, three or four point measurements, as described in the measurements chapter. There are four types of

components on the chip which are measured. Large junctions, around 400 nm wide overlapping the thin superconductor, have a typical resistance of  $1 \text{ k}\Omega$  [62]. Small junctions are only 100 nm wide, thus providing a quarter of the surface area compared to large junctions. The resistance is therefore close to four times as large.



**Figure 6.1** – Each chip of the BL3 wafer with the materials used. The unused green samples on the wafer have either not been used or have no recorded results.



**Figure 6.2** – The BL3 wafer with each chip, marked by color if the chip completed room temperature measurements, cryostat measurements or was inspected in the scanning electron microscope (SEM).

The magnetic induction line, from contact 10 to 11, is a layering of metals and should have a low resistance. Since there are only two points of contact, the resistance of the two cables is added in series summarising to around 600  $\Omega$  [62]. The Andreev interferometer is a new component, with no experience regarding the expected resistance. The

Sample	Material	j14	j15	J16	J1	j2	j3	J4	J5	j6	<b>J</b> 7	<b>J</b> 8	j9	MIL	AI
BL2-45	Ag 56, Al 45	17 k	11 k	18 k	9.5 k	46 k	В	Ν	18 k	58 k	52 k	47 k	1 k	681	Ν
BL2-46	Ag 56, Al 45	13 k	23 k	20 k	1.1 k	4.6 k	121	121	4.2 k	9.8 k	9.6 k	7.6 k	8 k	635	Ν
BL2-53	Ag 42, Al 35	437	307	305	382	342	Ν	150	250	372	335	381	500	513	419
BL2-54	Ag 42, Al 35	470	329	321	429	403	Ν	251	252	382	528	14 k	5.6 k	554	3.6 k
BL3-15	Ag 30, Al 40	155 k	347 k	99 k	82 k	20 k	Ν	20 k	9 k	13 k	15 k	14 k	12 k	580	8 k
BL3-25	Ag 30, Al 40	40 k	80 k	235 k	10 k	31 k	Ν	386	Ν	Ν	22 k	21 k	14 k	660	53
BL3-31	Ag 42, Al 35	В	В	127 k	2 k	В	Ν	52	5	19 k	В	8 k	4 k	559	1.6 k
BL3-32	Ag 42, Al 35	23 k	28 k	6 k	10 k	337 k	Ν	161 k	118	192	161 k	158 k	203 k	1 k	175 k
BL3-34	Ag 42, Al 35	Ν	В	8 k	45 k	В	Ν	140 k	274	23 k	110 k	94 k	24 k	960	47 k
BL3-22	Ti 30, Al 25	6.2 k	5.5 k	1.3 k	1.5 k	5 k	В	Ν	1.6 k	76 k	2 k	1 k	5.1 k	900	1.4 k
BL3-23	Ti 30, Al 25	В	6.5 k	1.3 k	2.6 k	В	7.9 k	Ν	Ν	10 k	1.5 k	1.6 k	4 k	970	1.2 k
BL3-24	Ti 30, Al 30	В	25 k	3 k	В	В	В	1.5 k	1.5 k	8.2 k	3 k	3 k	1.3 k	817	1.4 k
BL3-42	Ti 30, Al 30	В	270 k	35 k	2.4 k	757 k	26 k	500	Ν	474	5 k	5 k	285	1.2 k	1 k
BL3-53	Ti 3 + Ag 42, Al 35	21 k	17.5 k	1.3 k	548	11 k	8.6 k	2.5 k	2.5 k	20 k	4.3 k	4.4 k	В	416	1.2 k
BL3-62	Ti 3 + Ag 42, Al 35	10 k	В	273	834 k	В	Ν	В	В	844 k	78 k	5 k	1 k	Ν	100

resistance of the interferometer is measured with a three or four point technique. The resistance of the interferometer for functioning samples will be discussed on page 59.

**Table 6.1** – Samples which have been measured at room temperature. Unmeasured junctions are marked with "N" and infinite resistances, bad contacts and short circuits "B". Large junctions are marked with a capital "J" in boldface, while the smaller junctions are marked with a lowercase "j". All resistances are in  $\Omega$  or k $\Omega$ , as marked in the figure. The material column has the normal metal, its thickness in nm followed by the strong superconducting material and thickness. Samples BL3-53 and BL3-62 have two layers of normal metal, both Ti (3 nm) and Ag (42 nm).

#### 6.1.1 Resistance and materials

The samples fabricated from wafer BL2 all had the same metals; silver, aluminium and aluminium evaporated in an oxidizing environment, their thicknesses are shown in figure 6.1. In general the BL2 wafer has either junctions with very low resistance, suggesting that the current shunts passed the junction in the underlying silicon, or too large resistances. The large resistance could be due to some reaction between silver and aluminium or a smaller than expected contact surface. Samples from BL3 have different materials, which are shown in figure 6.1. More of the BL3 samples were measured, and some were put in the scanning electron microscope for fabrication feedback, this is all shown in figure 6.2.

Pure titanium becomes superconducting at 400 mK [73], which is why it on two samples has been combined with a thicker layer of silver to form the normal metal. The BL3 wafer in general has many samples with well defined structures malfunctions. The problems have been the Andreev interferometer, which has been broken on some samples, see for example figure 6.4, and the resistances of mainly small junctions. Large junctions with silver as normal metal, have to some extent been bad, but better for titanium. Figure 6.1 gives an overview of samples and their room temperature resistances. Silver has not worked well with the BL3 design.



**Figure 6.3** – BL3-34, an overview of the circuit, comparable to the CAD-design in figure 4.4(b). Both loops are visible together with some debris remnant from the etching of the hard germanium mask. The magnetic induction line is marked with MIL, while the Andreev interferometer is marked with a orange arrow.

The BL3-3 design is exemplified in the scanning electron microscope picture 6.3, where the circuit of the both bimetallic loops of BL3-34 is shown. Comparing this with figure 4.4(b) the structure looks good. The BL3-34 was measured at room temperature and proved to have many high resistance junctions, but the chip overview picture is typical for all fabricated samples.

In total 15 samples have been measured at room temperature. The resistance of each measured junction is shown in figure 6.1, unmeasured junctions are marked with "N" and infinite resistances, bad contacts and shorts "B". Large junctions are marked with a capital "J" in boldface, while the smaller junctions are marked with a lowercase "j".

The table gives an overview of trends in fabrication and any systematic errors which could be a result from the process. The large junctions are paired, so junctions 16 and 1, 4 and 5, and 7 and 8 which are geometrically close to each other should be similar in their resistance.



**Figure 6.4** – The BL3-41 has a small bleed (marked with arrow) of superconducting aluminium from the second (middle) layer. The thin strong superconductor (horizontal) is near 75 nm wide, and looks good in this figure. Other parts of the sample show shadows from evaporation, implying that the undercut was too big.

Looking at small junctions on BL3, junctions 14 and 2 have failed more often than others. This could be due to a design fault, when converting the CAD-design to the electron beam readable format some information will be lost. Both junction 14 and 2 are on the outside of the secondary loop as shown in figure 4.4(b), and not get the same amount of exposure due to the proximity effect as junction 15 in the middle.

The resistance of the junction is highly dependent on the oxidation time of the pure aluminium layer. All samples with silver as normal metal have been exposed to oxygen for 5 min, so that the pure aluminium layer should have a thick layer of oxide. The samples with titanium are exposed longer due to the titanium's ability to react with the gas and decreasing the amount reacting with the aluminium. The layer of aluminium evaporated in an oxidizing environment S' is the same for all samples. Since only the S and S' materials are in contact the resistance should be characterized by the oxidation time and the thickness of these materials.

Looking at junctions 7 and 8 on samples BL3-22, BL3-23, BL3-24 and BL3-42, a slightly thicker layer of aluminium has increased the resistance. The influence of material thickness is not clear, since there are few other working junctions to compare the samples. If there was a trend, it would appear for both the primary and the secondary loop.

The magnetic induction line should have a resistance in the range of  $500 \Omega$  to  $1 k\Omega$  which is realised in all save one sample, BL3-42. The structure is simple, merely a line of

metal close to the circuit, so any problems should be detectable a room temperature. An interesting point is the stability of the line's resistance, samples fabricated with the same materials and thickness have a tendency to have very similar resistances. The resistance of other junctions can sometimes differ by an order of magnitude.

The Andreev interferometer has a range of resistances. Room temperature measurements have shown good resistances for BL3 samples, with titanium, but not as good with silver. The main problem despite the resistances is breaks. Many of the interferometers have been broken during liftoff on the pure titanium BL3s.

Only a few samples placed in the cryostat have shown successful results. Figure B.1 on page 84 in the appendix shows measured data for samples in the cryostat. Clearly both BL2-45 and BL2-46, with identical fabrication parameters have both had many infinite junctions when measuring in the cryostat. The similar chip, BL2-53 had thinner layers of silver and aluminium which yielded slightly better junction resistances. After moving to the cryostat, BL3-25 had no functioning junctions. The Andreev interferometer's resistance at room temperature was small at room temperature, but gave a bad connection in the cryostat. This chip illustrates that samples which work fine at room temperature can fail disastrously in the cryostat.

#### 6.1.2 Geometrical considerations

Figure 3.9 on page 39 shows a problem in fabrication. The pale shadow, above and below the thin strong superconductor is caused by an undercut which is too big, the sample has been over etched. Considering step K in figure 3.5 on page 30, there is no undercut to catch the shadow evaporated material, which instead falls on the wafer. If the lower line in figure 3.9 was more clear, the extra shadow could short circuit the interferometer and render the sample useless. In figure 6.5 the upper shadow has been lifted away correctly and there is no risk of a short circuit. Bridges in the germanium mask give similar consequences as shadows. The bridge can be caused by insufficient germanium etching or poor exposure of PMMA in the electron beam. Its consequences can be seen in particular in figure 6.5 since the extra bit of hard mask has left openings in the evaporated interferometers right arm. The changed layout, referred to as BL3-2 in section 4.2.2 used on BL2-22 gave surprisingly good junctions, but was not measured in the cryostat. The Andreev interferometer was damaged, as shown in figure 6.5. The break is visible in both the normal metal part and the shadow lower in the picture, and is due to a small bridge in the germanium hard mask which has not been etched away. The new design with triangular shapes increased the overlapping area of different layers.

Figure 6.6 shows the *SIS*' junctions on the primary loop of BL3-64. The aluminium strong superconductor, visible as the thin line is unbroken and the *SIS*' junctions look good. The fine grain is the last layer of aluminium evaporated in an oxygen environment which has formed on top of the underlying aluminium. This chip used 10 nm of titanium as normal metal, but was fabricated to be used only in the scanning electron microscope, and was never measured. The etching has left debris around the structures, and the size of the undercut can be appreciated.



**Figure 6.5** – The Andreev interferometer (orange arrow) on BL4-22 has a broken arm. From the shadow (red arrow) it is clear that the break is due to a small bridge in the germanium hard mask which has not been etched enough. The thin strong superconductor, marked blue, is broken on the left.



**Figure 6.6** – The *SIS'* junctions connected to the primary loop of BL3-64. The junctions are shown in the CAD-design in figure 6.3. It has a clear strong superconductor *S* (thin horizontal line) and tunnel junctions number 3, 4, 5, 6, 7, 8, 9 with connection lines. The grain of the last layer, aluminium evaporated in an oxidizing environment is visible.

# 6.2 Experimental results

The fabrication gave several samples that underwent measurements in the cryostat. One sample the authors would especially like to present is the BL3-53. More information of its material makeup is in figure 6.1. At subkelvin temperatures there was an infinite resistance in junction 6. A number of the small junctions did not work as required on the chip. However, the loops, the interferometer and magnetic induction line were functional. On this chip only parts of the experiments could be done since not all of the junctions were working according to our requirements. Sample 53 is of a Bl3-2 design.

The BL3-23 and BL3-24 did also have functional Andreev interferometers. Because chip number 23 was not measured with a magnetic shield in the cryostat, the measurement results are not useful. As seen in table 6.2 the BL3-53 had substantially higher normal resistance  $R_N$  of the interferometer, giving clearer modulation.

The interferometer on BL3-53 was different from the ones on samples 23 and 24. The BL3-53 had an additional silver layer of 42 nm. The only difference between samples 23 and 24 is the aluminium layer, which is 5 nm thicker on BL3-24. Additionally the BL3-24 had 4 junctions with infinite resistance. Thus we concentrate on the BL3-53 results in this section.

 Table 6.2 – Normal resistances for Andreev interferometers.

Chip	Junctions	Sweep direction	Resistance ( $k\Omega$ )
BL3-53	4–12, 5–13	+-	5.6
BL3-23	12–5, 13–8	+-	1.55
BL3-24	4–12, 5–13	+-	0.955

The sweep direction in table 6.2 signifies how the DC sweep was done. A sweep starting at a positive voltage going to a negative one is denoted as +-. Conversely -+ denote a sweep starting at the negative voltage. In addition, the sweep direction determines the polarity of injected quasiparticles. The sweep direction is also important if there is some element of hysteresis.

#### 6.2.1 Data processing

The I-V data saved by the LabVIEW program was loaded in a MATLAB program. The LabVIEW program saves the voltage  $V_B$  across the magnetic induction line (or chosen junctions) and the  $V_{out}$  from the multimeters. The magnetic current  $I_B$  was calculated as  $I_B = \frac{V_B}{R_{Magnet}}$ , where  $R_{Magnet}$  is the bias resistance for the magnetic induction line (or the chosen junctions).

The  $V_{out}$  voltage includes a factor 20 from the amplifier in the lock-in amplifier and an additional 1000 times from the amplifier. The voltage is given in nanoamperes. The LabVIEW program also scales the voltage using a resistance factor, which should be equal to the bias resistance. This means that the actual voltage over the sample,  $V_{sample}$ ,

is given by (6.1). In certain cases the program has used the incorrect scaling factor. In those cases  $R_{Bias}$  has been replaced with the correct scaling factor.

$$V_{sample} = \frac{V_{out}}{R_{Bias}} \cdot \frac{10^9}{20 \cdot 1000} \tag{6.1}$$

The differential resistance  $R_{diff}$  is found by dividing the voltage over the sample,  $V_{sample}$  by the bias current on the chip. The bias current equals the AC modulation voltage divided by the bias resistance  $R_{Bias}$ . The phase difference between the superconductors was computed using (2.43). The normal resistance  $R_N$  was calculated in an IV measurement.

$$R_{diff} = \frac{V_{out} \cdot R_{bias}}{V_{ac}} = \frac{V_{out} \cdot 10^9}{V_{ac} \cdot 20 \cdot 1000} = \frac{V_{sample}}{V_{ac}} \cdot 5 \cdot 10^4$$
(6.2)

#### 6.2.2 The Andreev interferometer



**Figure 6.7** – Modulated resistance in the Andreev interferometer for a sweep from +10 to -10 V. The phase shift, shown in red, is calculated from the resistance using equation (2.43).

The interferometer resistance modulation was tested using the setup described in section 5.2.1 on page 51. A voltage was established over the magnetic induction line and associated bias resistances, creating a current and thus a magnetic field. An AC voltage of 1 V and 135 Hz was applied over junctions 4 and 12, while the bias resistances were connected to junctions 5 and 13. This created a current through the primary loop and the interferometer.

The resistance modulation is shown in figure 6.7. The resistance is considerably higher than the normal resistance, which is consistent with equation(2.43). Within the range of periodic behaviour the blue curve has a difference between highest and lowest resistance of 1.992 k $\Omega$ . Compared to the normal resistance for the Andreev interferometer and junctions 5–13 this corresponds to a modulation of 35%.

The behaviour around  $I_B = 0 \,\mu A$  was inspected more closely. By using a magnetic sweep from  $+10 - 10 \,\text{V}$  and a magnetic bias resistance  $R_B = 1 \,\text{M}\Omega$  the current steps are
shorter. Figure 6.8 shows the differential resistance for this current sweep and applied AC voltages from 2V to 250 mV. The behaviour is fairly smooth and matches that in figure 6.7.



Figure 6.8 – The resistance of the Andreev interferometer for high AC bias voltages.

When the AC voltage gets smaller in amplitude the resistance looks more discrete and quantized. Figure 6.9 shows the differential resistance for AC voltages from 62 mV down to 4 mV. For comparison, the Sharvin resistance, given in equation (2.46), is approximately  $26 \text{ k}\Omega$ , close to the highest curve of the graph.



**Figure 6.9** – Quantization of resistance in the Andreev interferometer for different low AC bias voltages.

At sufficiently high modulation voltages the current through the chip becomes so high that it overheats the bimetallic loop. This results in a supercurrent with opposite trend in the differential resistance. A case of this is shown in figure 6.10. The limit is in the interval 3.5 - 3.626 V.

The magnitude of the modulation in the interferometer's resistance is unexpected. Other experiments have not been in the  $k\Omega$  range but rather in the  $\Omega$  regime. The maximum modulation in the experiment conducted by Petrashov, Antonov, Delsing and Claeson was 15% [8], compared to 35.6% in this experiment.



**Figure 6.10** – At sufficiently high bias voltages the current through the chip is so high that it overheats the bimetallic loop. This results in a trend change in the differential resistance.

#### 6.2.3 The thermoelectric effect in the bimetallic loop

The thermoelectric effect is achieved by heating the topside of the primary loop. The design, with the five *SIS'* junctions connected to the loop, provides the possibility to heat one part of the loop and measure the temperature change. Not all junctions were operational on the BL3-53 chip. Because of this, a part of the experiment had to be abandoned. The loop could be heated but the temperature gradient ( $\nabla T$ ) could not be measured.

Two junctions are needed to create a current in the loop. A symmetric current is created if the junctions are equal in size as in the case of junctions 7 and 8. If two junctions of different size are selected, like 3 and 7, the injected current is non-symmetric shape.

Contacts 7 and 8 are connected to junctions with the same size, while junction 3 is smaller.

Figure 6.11 shows how the sweep of the heating current induces a change in the resistance of the interferometer. This modulated resistance means that the heat has affected the phase of the superconductor and there is some, still undetermined amount of thermoflux induced. It should be observed that there was no voltage over the magnetic induction line in contacts 10 and 11 for this experiment.



**Figure 6.11** – The resistance of the Andreev interferometer as function of current injected in different junctions. Junctions 7 and 8 are both large, so the blue curve corresponds to symmetrical quasiparticle excitation. Junction 3 is small, so the red curve corresponds to asymmetrical excitation.

The malfunctioning small junctions on the BL3-53 chip make it impossible to measure the induced temperature gradient. To compare the results with other studies the induced thermoflux and its dependence on temperature must be known. A sample with more working junctions would be desirable for future progress. The results must also be repeatable with different polarities of the induced quasiparticle current.

#### 6.2.4 Loop interaction experiments

These measurements have not been repeated since the BL3-53 sample malfunctioned during measurements. Instead of injecting a quasiparticle current in the primary loop, the secondary loop in figure 6.3 can be used to induce another thermomagnetic field. The secondary loop is heated in this interaction experiment to investigate potential readout in the interferometer.

The thermomagnetic field which is caused by the heating on the secondary loop affects the primary loop. The magnitude of this effect is unknown, as it is unclear how the coupling between the loops occur. This field affects the phase of the superconductor in the primary loop and thus the interferometer resistance is changed.



**Figure 6.12** – The interferometer resistance in loop interaction experiments. The current is injected through different junctions in the secondary loop. Junctions 1 and 16 are large, while 2 and 15 are small. The arrows point to repeated curve features and a seeming periodicity.

To test the interaction effect a voltage is applied between two junctions. The large junctions 1 and 16, and the small junctions 2 and 15, were considered. Two symmetric (1–16 and 2–15) cases were tested as well as one asymmetric (15–16) and a combination of the two symmetric ones (2–15,1–16). The resulting measured resistances are shown in figure 6.12. Unfortunately it was not possible to measure the temperature gradient in the secondary loop either.

The asymmetric case (15–16) gives a higher resistance than the symmetric case with two small junctions (2–15). Both cases seemingly exhibit periodicity. The arrows in figure 6.12 point to repeated curve features. The combination case (2–15, 1–16) showcases a nearly flat behaviour, as if the two currents cancel out the periodicity. It also gives a lower magnetoresistance than the previous case, which further points to some cancelling.

It must be stressed that this is far from a clearcut result. Work remains to ascertain that there is no extraneous field affecting the measurements. This is especially important given that the modulation amplitude is so smaller for the interaction experiments.

### **Chapter 7**

# Discussion

The fabrication results have provided feedback to the process and the next wafer will have some changes in design. The geometry of the BL3 wafer will change so larger contact surfaces between high contact pads and the smaller lower structures is discussed in under the geometry. The material issues have given some good indications of silver and titanium, but the problems with pure silver are still unresolved, as discussed in materials.

The results to be discussed in this chapter are the thermoelectric effect in the bimetallic loop, the modulation of resistance in the interferometer, the degrees of charge imbalance in the loop interaction experiment. Quantization of resistances from a base resistance of 22 k $\Omega$  have also been explained in this section.

#### 7.1 Fabrication

Every sample fabricated has apart from the probabilistic chance of working, a large number of parameters which have controlled the machines in the process. In table 6.1, only the geometry, materials and their thicknesses are considered. Samples evaporated together, with the same material and thickness, are sometimes etched different lengths of time in order to see which amount of etching gives the best defined structures and most reliable result.

The fabrication leads to very different room temperature resistances. Two samples, as discussed in the previous chapter can have the exact same fabrication parameters and still yield very different junction resistances. Though the resistance of the magnetic induction line is similar for similar parameters.

The junctions meanwhile have a much smaller effective area and the resistance depends

on local statistical variations. The resistance of a junction is highly dependent on the uniformity of oxidation of the superconducting aluminium layer. If certain areas are less oxidized they may open conducting channels through the junction, thus lowering the resistance. Many channels would drastically change the resistance, which is also the case in many of the fabricated samples. Resistances in around the expected, thus  $1 \, \mathrm{k}\Omega$  for small junctions and  $4 \, \mathrm{k}\Omega$  for large are good. As soon as they start to differ by an order of magnitude or more, the junction would be regarded as bad.

#### 7.1.1 Developments in the geometry

The design change is visible where two separate materials need contact and overlap. The height difference of the contact pads, connection lines and the smaller structures needs to be considered. This is because a thin layer evaporated on top of a thick one, could break if it is lifted over the surface of the wafer, see the left part of figure 7.1 as an example. The thick layer does not have a straight edge, therefore the evaporation of the thin layer will not reach the edge of the thick layer.



**Figure 7.1** – To the left, the contact between the thinner aluminium layer and the larger gold layer is broken. The small contact can be seen from above in the upper left part of the figure. A new design with a larger contact area was developed and can be seen from above in the upper right part. The new design proposes a smaller difference in height between the contact pads and structures and a larger interaction surface. The intended result is shown in the lower right of the figure.

The breaks of thin lines caused many bad contacts, and broken structures in the early design. Increasing the area of the thin layer on top of the high layer strengthens the

structure and the thin layer is not as prone to break. The new structure between the large and the thin layer is illustrated in the right part of figure 7.1. The thin layer is also evaporated around the larger layer, which creates a greater contact between the layers.

The differences between the layouts have been discussed in the design comparison section 4.2.2 on page 45. BL3 samples have mostly been fabricated with the old design. Given functional samples, the new design has been shown in some scanning electron microscope pictures (figure 6.5) and is one of the next designs proposed in further studies.

The problems with small junctions, mainly 14 and 2 could be related to the CAD design. Increasing the junction area would give more reliable fabrication results, but increase the critical current. A small junction can suppress the critical current hysteresis.

#### 7.1.2 Materials used and their influence

The BL2 design, using silver as normal metal has at room temperature either had low, shunting resistances or too high resistances. The BL3 had few functional junctions which had pure silver as normal metal. Considering the BL3 with pure titanium, junctions look good, but many interferometers were broken. Some samples were however measured in the cryostat and gave modulated resistances when injecting current into the magnetic induction loop. BL3-23 was measured without a magnetic shield. Thus the interference from the earth's magnetic field make the results good for fabrication, but not conclusive to the rest of the experiment.

When the cryostat was reopened, two new samples, BL3-24 and BL3-53 were measured, this time with the magnetic shield. Coincidentally, the BL3-24 did not work as well as hoped. BL3-24 had a slightly thicker *S*-layer, but the same amount of titanium. The BL3-53, meanwhile with the titanium and silver as normal metal, gave as discussed better results.

The mix of silver and titanium seems to have a positive effect. The resistance of the sample indicates that the titanium conducts most of the current, and the silver stops it from superconducting. Thus the silver saves phase coherence and ensures normal metal behaviour. This behaviour could be verified by more experiments, as discussed in section 7.4.

The use of silver and aluminium on the samples has given the Bolometer group unexplained results. There seems to be a degradation of the resistance in the interface between the metals. An electrochemical reaction starts after the sample has been measured changing the resistance of the junction. Samples which at first give good results, can have infinite resistances a day or two later. The group has had little input from others with similar problems, and have not found a good explanation of the process.

# 7.2 The Andreev interferometer and its quantized resistance

The modulation is considerably higher than in previous experiments. Petrashov, Antonov, Delsing and Claeson reached a modulation of 15 % [8] in a cross interferometer, and in Stoof and Nazarov's model the theoretical limit is 9.7 % [9]. Stoof and Nazarov considered a different geometry. The result of 35% in this experiment is therefore quite unexpected.

As described in the introduction chapter, there are to our knowledge no earlier Andreev interferometer experiments in k $\Omega$  regime [76]. The earlier experiments have been in the  $\Omega$  range. This opens up a new area of mesoscopic physics, and may be important for the understanding of the unexpectedly high resistance modulation.

During the measurements Andreev interferometer's resistance an unexpected quantization was found. It is clear in figure 6.9 that there are small jumps in the resistance indicating that the resistance can only assume discrete values. The highest resistance is close to  $22 k\Omega$ , with a jump down to  $15 k\Omega$  for the first increase in the AC bias voltage. The falling trend holds for all curves in the figure.

The highest resistance is close to the resistance quantum  $\frac{h}{e^2} = 25.8 \text{ k}\Omega$  from the Sharvin resistance equation (2.46). That resistance quantum is reached for one conducting channel. The observed resistances could be explained as a number of open conducting channels, in some configuration of parallel and series channels. A higher voltage opens more channels.

The resistance quantum should give the basic resistance series given in equation (7.1) [44]. The observed resistances do however not follow the series. This can be attributed to the complicated interferometer geometry, which consists of two parallel branches and the lower part in series [11].

$$R_{AI} \sim \frac{1}{n} \frac{h}{e^2} \tag{7.1}$$

The trend of falling resistance with increased AC bias voltage continues for higher voltages in figure 6.8. There is however one exception at  $V_{AC} = 2 \text{ V}$ , where the resistance is higher than the other curves. This cannot be attributed to the number of conductive channels.

Stoof and Nazarov [9] consider the temperature dependence of the resistance in Andreev interferometers, as discussed in section 2.4.3 on page 25. They provide figure 7.2, showing how the normalized resistance varies with temperature, which is proportional to  $\left(\frac{L}{\xi}\right)^2$ . The length *L* is comparable to the interferometer arm length. The coherence length  $\xi$  is a function of temperature according to equation (2.34), where  $\alpha$  is a function of temperature as described in section 2.1.2 on page 11.

The AC bias voltage creates a current, which causes an increase in temperature. The bias voltage can thus be seen as a temperature. For a given current  $I_B$ , the observed resistances can therefore be placed along the curve in figure 7.2. The minimum in



**Figure 7.2** – Temperature dependence of the normalized resistance  $\frac{R}{R_N}$  for an Andreev interferometer. The temperature is proportional to  $\left(\frac{L}{\xi}\right)^2$ . The figure is from Stoof and Nazarov [9].

observed resistance, for  $V_{AC} = 1 \text{ V}$ , coincides with the minimum of the normalized resistance in figure 7.2. The lower bias voltages are placed to the left of the minimum, while the resistance for  $V_{AC} = 2 \text{ V}$  is to the right of the minimum.

This provides a qualitative understanding of the trend. However, it should be noted again that Stoof and Nazarov used their model to predict a maximum modulation of 9.7 % [9]. This should be compared to the 35.6% modulation found in this experiment. It appears that a new model is needed to explain the modulation in the new resistance regime. However, the new model is likely to share aspects with Stoof's and Nazarov's model, in order to achieve a similar qualitative agreement.

The behaviour is therefore explained in terms of both conduction channels and a temperature dependence. For low bias voltages (or temperatures) there are few conductive channels, which dominate the resistance. The number of channels increase rapidly with the bias voltage. At a sufficiently high voltage there are enough channels already open, that a change in temperature does not provide remarkable steps. Instead a temperature dependence, such as the one described by Stoof and Nazarov [9], begins to dominate the change in base resistance.

#### 7.3 The bimetallic loops

The two different experiments on heated bimetallic loops and thermoelectric effects are discussed here. The experiment on the thermoelectric effect in the primary bimetallic loop is discussed in the following section. The loop interaction experiments discussion is placed in section 7.3.2 on page 73.

#### 7.3.1 The thermoelectric effect in the bimetallic loop

It was mentioned in the results chapter on page 64 that the temperature gradient could not be measured. The loop could be heated, but the remaining *SIS'* junctions could not be used for temperature measurements in the loop. This poses a difficult situation in comparing the results with other studies concerning thermoflux in a loop coupled with an Andreev interferometer.



**Figure 7.3** – There are three different regions in the magnetoresistance. The first region is increasing in resistance, the second is decreasing and the third is increasing again. Note that in the second region the resistance is decreasing despite the increasing current.

In figure 7.3, there are three regions which need to be explained. The first regime is from -20 nA to the maximum at -2.95 nA. This region has an increasing amount of quasiparticle charge imbalance. As temperature increases from the quasiparticle injection into the top of the loop, resistance increases. A larger amount of quasiparticles travel into the superconductor before reflecting out into the normal metal again. In this region most quasiparticles have energies higher than the energy gap,  $E_k > \Delta(T)$  [54].

In the second region from -2.95 nA to the minimum at 10.2 nA the resistance is decreasing with increasing quasiparticle current. The interaction between quasiparticles and particles in this region is non-trivial. This can be seen in the discrepancy between to Ohm's law and the results in figure 7.4.

For a constant AC voltage we have  $V = I \cdot Z$ . The circuit is purely resistive, giving Z = R. The observed results are clearly contrary to Ohm's law. This means that there must be another interaction that is responsible for the observed behaviour. The theory used for the following explanations is from section 2.4.3 on page 25.

The decrease in resistance is because of quasiparticle relaxation mechanisms and quasiparticle populations converging to equilibrium. The degree of charge imbalance is decreasing from an excited state towards a mixed state, where excitations and relaxation of the quasiparticles are coexisting. A fraction of the quasiparticles above the Fermi level will be reflected into the far side of the superconductor as a quasiparticle below the Fermi level, causing interference. The energy of the quasiparticles in this region is more uncertain, but large fractions have  $E_k < \Delta(T)$  [53].



**Figure 7.4** – The results from the experiment on the thermoelectric effect in the primary bimetallic loop are shown in the red and blue curves. The blue curve corresponds to symmetrical heating and the red to asymmetrical heating. The black dashed line shows the expected behaviour according to Ohm's law  $V = I \cdot R$  for a constant voltage.

The resistance increases in the third region of interest to the far right in figure 7.3. The re-entrant resistance is caused by the proximity effect: the existing Cooper pairs are smeared out into the normal metal in this interval [58]. This could be an indication of a fluctuating order parameter in this region. A larger fraction of quasiparticles have en energy higher than the energy gap, increasing the resistance [53].

#### 7.3.2 Loop interaction experiments

Since the loop interaction experiments, as yet have not been verified with similar parameters, these conclusions, based on section 2.4.3, must be treated very cautiously. Until these experiments are confirmed by another measurement, the effects described below can be of a number of reasons: parasitic magnetic fields inside the cryostat, magnetic fields from voltage bias cables or any external magnetic source not of interest for the experiment. The authors hope these effects are not misleading, but real results!

Like in the previous experiment, the temperature was not known because of a difficult measurement situation with broken *SIS'* junctions. This makes it problematic to compare the results with other studies. In addition, no previous studies on these effects have been conducted to the authors' knowledge.

The combination of junctions 1 and 16 does not exhibit any periodic behaviour with an applied quasiparticle current. However, the asymmetric cases junctions 15, 16 and 2,15 with 1,16 exhibit seemingly periodic or nonvarying results, respectively. 15 and 16 are one small and one large junction. This measurement exhibits a seemingly periodic behaviour in the right hand side of the graph in figure 6.12 on page 66. The oscillation is modulated by a difference of  $0.17 \,\mathrm{k\Omega}$ . The two observed modulations are probably because of the asymmetric current flowing into the junctions.

On the other hand, focusing on the measurement with only small junctions, the

modulation gets much larger. The secondary loop is heated through junctions 2 and 15. Instead of only two modulations, the graph in the previously mentioned figure exhibits three repetitions of similar curve features.

The modulation of resistance is of the same order as the previous measurement, however, it is a factor of one and a half as large at  $0.26 \text{ k}\Omega$ . With only small junctions which are near each other, the current is slowed down due to the high resistance. This can be contrasted to the 1-16 measurement, where the quasiparticles have a higher speed. The modulation depends on the constructive interference of the quasiparticles.

A more unexpected result is when junctions 2-15 and 1-16 are heated. Then only a small modulation in the resistance can be seen. The graph is almost flat compared to for instance the previous result from junctions 2 and 15. There must be some destructive interference from the quasiparticles in all the four junctions.

#### 7.4 Suggestions for future work

There are mainly three directions for the future work. One, the data should be examined and explained in theoretical models. Two, more samples need to be fabricated in order to repeat the results. Three, the new samples should be improved so that more useful information can be obtained from the  $k\Omega$  regime.

If a sample with fully functioning junctions could be fabricated, then the eperiment on the thermoelectric effect could be conducted in full. The induced temperature gradient should be measured. To do this the development of the fabrication process should continue. The aim should be 100 % working structures. We suggest that the combination of titanium and silver is the most suitable interferometer material found so far, and recommend a variation of the material thicknesses.

The size of the small junctions could be increased a little, to give medium junctions. As shown in table 6.1 they are more unreliable than the large junctions. The change would lead to a decrease in resistance and the junctions would be more resilient to burn out due to heat dissipation. It should also lead to a higher number of operational samples.

The resistance modulation of the Andreev interferometer is high. In further experiments the temperature dependence should be measured. Also the sweep should be increased to see how many modulation periods can be completed. Experiments can be made with varying thickness of the magnetic shield, to see if additional effects can be measured.

For the loop interaction experiments the coupling should be studied. It is also important to make sure that no extraneous magnetic field affects the measurements. Most of all the experiment needs to be repeated.

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# Appendix A

# **Fabrication recipes**

This appendix has the fabrication recipe for the samples. It include a step by step description of creation of contact pads. The electron beam lithography, shadow evaporation and liftoff process is also described. An overview of the fabrication process is shown in figure A.1.



**Figure A.1** – Overview of the fabrication processes. Sections A-E describe preparations for large structures, F-I creation of large structures, J-O preparations for small structures and P-S creation of small structures.

#### A.1 Contact pads

The different steps are shown in figure A.1. Steps A-I cover the contact pads

- 1. Take a 2" wafer of Si coated with oxide. Step A in figure A.1
- 2. Turn on two hotplates program, one to 180  $^\circ$  and 5 min and the other to 110  $^\circ$  and 2 min.
- 3. Clean the chuck (wafer mount) thoroughly with acetone.
- 4. Take a large tissue, cut a hole in the center and place it around the spinner's axle. Place the chuck on the axle mount.
- 5. Select program for spinner (machine #219): 3000 RPM for 1 min.
- 6. Blow the pipettes dry with nitrogen to clean them.
- 7. Place the wafer on the chuck.
- 8. Draw some LOR3A resist from the bottle and place on the wafer until 60-70% is covered.
- 9. With the lid closed, start the spin by pressing 'E' twice for automatic start.
- 10. Move wafer to  $180^{\circ}$  hot plate and bake for programmed amount of time.
- 11. Let the wafer cool, and clean the spinner's chuck. The cross section will now look like step B in figure A.1.
- 12. Reprogram the spinner to 6000 RPM for 1 min.
- 13. Put the sample on spinner. Place the S1813 resist in the center until 60-70% is covered. Close the lid and start the spin.
- 14. Bake on 110° hot plate. The wafer has now reached step C in figure A.1.
- 15. Place the chromium-glass photomask "Bolo BM Loop" in Mask aligner tool # 222.
- 16. Align the wafer with resist side up and slide the wafer in. Adjust rotation and position with the knobs on the front.
- 17. Raise the wafer to the mask to get good contact. Program machine for correct exposure (11 s) and expose.
- 18. Remove the wafer and develop the resist in a beaker of MF319 for 1 min. Stop development with water. The cross section should now look like step D in figure A.1.
- 19. Ash the sample in Batchtop # 419 using oxygen plasma for 30 s at a power of 50 W. This results in step E, figure A.1.
- 20. Place the wafer in Balzers evaporator # 514 and program for materials, rates and thicknesses from table A.1. The deposition of materials are shown in steps F through H in figure A.1.

Tal	ole	A.1	– I	Parameters a	nd mat	erials	used	in	Bal	zers
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	Ti	Au	Pd
Rate	.2 nm/s	.5 nm/s	.2 nm/s
Thickness	10 nm	130 nm	10 nm

- 21. Move the samples to a wet bench and prepare a beaker of Shipley 1165 remover. Place the samples in a bath and use ultrasound to speed up the process. 100% ultrasound power.
- 22. When all excess gold has been removed, rinse in isopropanol and water. The contact pads are now formed on the wafer as in step I in figure A.1.

#### A.2 Electron beam lithography

- 1. Use spinning as described in the previous section to apply a 360 nm layer of copolymer on the wafer. Bake. The contact pads are covered as shown in step J in figure A.1.
- 2. 40 nm of germanium is evaporated to form a hard mask, see step K in figure A.1.
- 3. Spin a 200 nm layer of electron beam resist PMMA onto the wafer, to prepare for electron beam exposure. See step L in figure A.1.
- 4. Scribe the wafer with a diamond to make it possible to split the wafer into individual samples.
- 5. Place the wafer in an electron beam lithography machine to have chosen areas of the wafer exposed.

#### A.3 Shadow evaporated structures

Steps L to S are covered in figure A.1.

- 1. After electron beam exposure (also shown as step L in figure A.1), develop the PMMA in Toulene:Isopropanol 1:3 for 40 s. Stop in isopropanol and dry with nitrogen gas, giving step M in figure A.1.
- 2. Prepare tool # 404 for germanium etch. Select program "Sumedh Ge Etch" and set time to 85 s. Place sample in load lock and run program. After etching the cross section would be similar to step N in figure A.1.
- 3. Move the sample to tool # 419 for copolymer etching. Program oxygen etching 100 W, 100 mT for 160 s. Figure A.1, step O shows the undercut etched in the copolymer. The undercut should be about 250 nm.
- 4. When the etching is completed, take the sample to Edwards evaporator (tool # 425)
- 5. Close the external gate valve of the fine pump (so that the aluminium ring is visible)
- 6. Press vent and unlock the door. When venting is completed, seal the rough pump.
- 7. Clean chamber with vacuum-cleaner and/or IPA. Check shutter function.
- 8. Mount appropriate boats for the materials used and put enough metal in the boats for a complete evaporation. Note which boat has which material
- 9. Mount the samples in rotational holder. Be careful considering directions and rotation axis, since the evaporation angles must be correct. Take care not to scratch samples when tightening screws.
- 10. When everything is in place, wipe the door edges and close the door. Cycle the roughing pump and check that the oxygen valve is closed.
- 11. When the roughing is complete, the controller shows "pump down". Open the gate valve to the fine pump by pressing the red button.
- 12. Wait a few hours for pressure to drop to around  $10^{-6}$  mbar.

- 13. Program the automatic shutter control for the materials used and the required thicknesses. Check that the tooling parameter is set to .57 for the rotational sample mount.
- 14. Select the correct source and programmed layer. Check that the shutter is closed. Turn the voltage to "LT" and slowly increase the voltage.
- 15. Pre-evaporate a few nm, to ensure any dirt or impurities are removed, and to attain a stable evaporation rate (.2 nm/s. Open the shutter, and make certain the rate is stable during the process. Let the shutter automatically close.
- 16. Turn off the voltage and switch "LT" to "0". Wait for 5 min. The sample should now a a thin layer of metal, as shown in step P in figure A.1.
- 17. To oxidize the previous layer, seal the chamber. Leave the gate valve open, for a continuous flow. Open the oxidation valve completely and adjust the pressure to the required level. Start timer and oxidize for required time.
- 18. When the oxidation is complete, turn off flow of oxygen.
- 19. For other metal layers, rotate the sample holder to the desired evaporation angle and evaporate as described. Aluminium evaporated with oxygen is done through letting a flow of oxygen into the chamber and evaporating simultaneously. Wait 5 min between each material. For evaporation angles and materials in our recipe, see table A.2.

Table A.2 - Parameters and materials used in Edwards

	Ti	Ag	Al	Al
Rate	.1 nm/s	.2 nm/s	.2 nm/s	.2 nm/s
Thickness	3 nm	42 nm	35 nm	80 nm
Angle	$-43^{\circ}$	$-44^{\circ}$	0 °	45 °

- 20. When all metals have been deposited, close the gate valve, and vent the chamber. Make certain that the lock on the door is opened. Step R in figure A.1 shows three layers of deposited material.
- 21. Remove samples and tidy up.

#### A.3.1 Liftoff

- 1. Place the samples in a beaker of 1165 remover covered with a glass lid. Mark with a paper and leave a few hours. If there is no silver on the chip, acetone can be used instead of 1165, and is a lot faster.
- 2. Clean the samples in water and blow dry. The finished sample is shown in step *S*, figure A.1.

## Appendix B

# Samples - details and parameters

A more detailed description of the samples fabricated is presented in this appendix. In table B.1 the thicknesses of the different metals for the small structures are shown for each fabricated chip. The thicknesses are given in nm.

The information about what was done with the fabricated chips is also shown in the table. If they are put in the cryostat, the chips were measured in room temperature first. The chips labeled with SEM were analysed with the scanning electron microscope.

Sample	Material	j14	j15	J16	J1	j2	j3	J4	J5	j6	<b>J</b> 7	<b>J</b> 8	j9	MIL	AI
BL2-45	Ag 56, Al 45	В	В	В	В	В	В	В	В	В	В	В	В	311	Ν
BL2-46	Ag 56, Al 45	В	В	В	В	В	17	17	В	В	В	В	В	414	В
BL2-53	Ag 42, Al 35	1.3 k	205	240	6	В	Ν	N	N	Ν	Ν	Ν	203	416	Ν
BL3-25	Ag 30, Al 40	Ν	Ν	Ν	Ν	В	Ν	В	В	В	В	Ν	В	Ν	В
BL3-24	Ti 30, Al 30	В	В	Ν	Ν	Ν	137 k	1.8 k	1.8 k	В	1.1 k	3 k	В	Ν	1 k
BL3-53	Ti 3 + Ag 42, Al 35	N	Ν	N	Ν	Ν	N	10 k	10 k	В	8.5 k	8.8 k	311	Ν	5.6 k

**Figure B.1** – Fabricated samples with subkelvin resistances of individual junctions, the magnetic induction loop and the Andreev interferometer. Unmeasured junctions are marked with "N" and infinite resistances, bad contacts and short circuits "B". Large junctions are marked with a capital "J" in boldface, while the smaller junctions are marked with a lowercase "j". All resistances are in  $\Omega$  or k $\Omega$ , as marked in figure.

The chips that have been measured in the cryostat are shown in figure B.1, which gives an

Sample	N-S-S'	Thickness (nm)	What happened
BL2-45	Ag-Al-Al(O)	56-45-60	Cryostat
BL2-46	Ag-Al-Al(O)	56-45-60	Cryostat
BL2-53	Ag-Al-Al(O)	42-35-60	Cryostat
BL2-54	Ag-Al-Al(O)	42-35-60	Room temperature
BL3-12	Ti-Al-Al(O)	42-35-84	SEM
BL3-13	Ti-Al-Al(O)	42-25-60	Evaporation aborted
BL3-14	Ti-Al-Al(O)	42-25-60	Evaporation aborted
BL3-15	Ag-Al-Al(O)	30-40-62	Room temperature
BL3-21	Ti-Al-Al(O)	42-35-84	None
BL3-22	Ti-Al-Al(O)	30-25-60	Room temperature
BL3-23	Ti-Al-Al(O)	30-25-60	Cryostat
BL3-24	Ti-Al-Al(O)	30-30-60	Cryostat
BL3-25	Ag-Al-Al(O)	30-40-62	Cryostat
BL3-31	Ag-Al-Al(O)	42-35-60	Room temperature
BL3-32	Ag-Al-Al(O)	42-35-60	Room temperature
BL3-33	Ti-Al-Al(O)	42-35-84	SEM
BL3-34	Ag-Al-Al(O)	42-35-60	Room temperature, SEM
BL3-35	Ag-Al-Al(O)	42-35-60	None
BL3-41	Ti-Al-Al(O)	30-30-60	SEM
BL3-42	Ti-Al-Al(O)	30-30-60	Room temperature
BL3-45	Ag-Al-Al(O)	42-35-60	None
BL3-52	Ag-Al-Al(O)	42-35-84	Cryostat
BL3-53	Ti+Ag-Al-Al(O)	3+42-35-84	Cryostat
BL3-55	Ag-Al-Al(O)	42-35-60	None
BL3-62	Ti+Ag-Al-Al(O)	3+42-35-84	Room temperature
BL3-64	Ti-Al-Al(O)	42-35-84	SEM

Table B.1 – An overview of fabricated samples

overview of the results and the metals used for small structures. The material column has the normal metal, its thickness in nm followed by the strong superconducting material and its thickness. Samples BL3-53 and BL3-62 have two layers of normal metal, both Ti (3 nm) and Ag (42 nm).

BL2-45 and BL2-46 were evaporated with silver and had many bad junctions. The bad junctions are either infinite resistances, bad contacts or short circuits. The measured chips from the BL3 wafer showed better results.

## Appendix C

# Measurement setup

This appendix has the circuit diagrams for the different measurement setups together with a brief description of how a measurement is conducted. The setup and electric schematic for the I-V curves is described in section C.1. The setup for the measurements with an applied magnetic field is described in section C.2.

#### C.1 I-V setup

A component's I-V curve is measured in both room temperature and at subkelvin temperatures. It is conducted by sweeping a voltage through a bias resistance  $R_{Bias}$ , producing a current through the component. The voltage drop, caused by the junction or component is then amplified and measured. The I-V curve is constructed from the amplitude of the sweep and the output voltage.

#### C.2 Magnetic induction setup

The magnetic field is induced by running a current through the magnetic induction loop. This current is generated by a voltage sweep, where the voltage is measured with a LabVIEW controlled voltmeter, and passed through a bias resistance  $R_B$ . The resistance of the Andreev interferometer on the chip is measured using an AC-modulated signal from a lock-in amplifier. The signal is passed through a resistance  $R_{Bias}$ , thus providing a modulated current through the resistance. The voltage drop is measured over the resistance, amplified by an instrumentation amplifier and then passed back to the lock-in.



**Figure C.1** – The sample's resistance is measured using two voltmeters connected to LabVIEW. A sweep is generated, measured by the first voltmeter and passed through a bias resistance  $R_{Bias}$ , the current through the sample's resistance  $R_{Sample}$  gives rise to a voltage. This voltage is amplified by an instrumentation amplifier and measured by the second voltmeter.

The lock-in amplifier uses the AC-modulated signal to filter out noise from the measured signal. This is done in a phase-locked loop by minimizing the phase difference of the two signals. A time constant dictates how long to minimize and the filtered signal is then read to LabVIEW using a voltmeter.



**Figure C.2** – A voltage sweep, measured by the first voltmeter, is passed through bias resistance  $R_B$ . The current is then run through the magnetic induction line on the chip. The resistance of the sample,  $R_{Sample}$  is measured with a lock-in amplifier which generates an AC-voltage passed through the bias resistance  $R_{Bias}$ . The voltage drop over  $R_{Sample}$  is amplified and passed back into the lock-in where it is mixed in a phase-locked loop to remove unwanted signal noise.

## Appendix D

# Machines

The electron beam lithography machine is described in this appendix. The appendix also describes the cryostats used during subkelvin temperature measurements.

#### D.1 Electron beam lithography machine: JBX-9300FS

The JBX-9300FS electron beam lithography machine is designed for writing patterns in the nanometer to sub-micrometer regime. The desirable pattern is designed using a CAD program. The data is converted, where operations like scaling, mirroring, rotation, reversing of tone and resizing can be done and sent to the computers which manage the electron beam lithography.

The electron beam lithography is operated in vacuum, which is evacuated with four ion pumps. The electron-optical system is shown in figure D.1 and utilises an electron gun, four lenses and a detector. The electrons are emitted and accelerated from the anode before they pass the first lens, which adjusts the flow of the electrons. The lenses consist of magnetic fields, which interact with the electrons and thereby function as optical lenses. The second and the third lenses operate as zoom-lenses and interact with each other.

Before the fourth lens the electrons pass through an electromagnetic stigmator. This apparatus is used for astigmatism correction of the beam at the center of the optical axis and the correction of the field curvature. Finally the electrons pass through the fourth lens, the most important for focus, and hits the wafer. The backscattered electrons are detected by a solid-state detector, below the fourth lens [69].



**Figure D.1** – The electron beam lithography machine. The figure is inspired by references [83] and [69].

Many controls have to be used before exposure, including control of displacements and height. The accuracy of workpiece displacement is measured utilising a laser-interferometer with resolution of approximately 0.62 nm [69]. The technique is to compute the difference between the workpiece position and the designated position, subsequently forwarding the error to the electron beam for correction of its position.

Height changes are discovered by detecting optical-slit images. The images are reflected on the workpiece and detected by the position-sensing detectors. Since the detectors are positioned opposite each other, accurate measurements are possible even if the workplace is tilted. The measurements are forwarded for correction of the lens focusing, deflection amplitude and rotations of the deflection systems [69].

A mark of heavy metal is built into the workstage for detection. While scanning across the mark, backscattered electrons are detected with peaks and the position

is consequently determined [69]. The errors from drifting are calibrated from these marks [62].

On the stage there are two electron-absorbing detectors consisting of a mesh mark and a solid-state detector under the mark. As for the backscattering electron detection, the mesh is detected and then correlated. This enables measurement of the beam size and therefore automatic adjustment of the focus can be performed [69].

#### D.2 Cryostats

Cryostats are vessels constructed to provide cryogenic temperatures for a prolonged time, between hours to days or months. This requires the ability to cool the inside of the vessel and its contents to temperatures close to absolute zero using either fluid coolants such as liquid helium and liquid nitrogen or with mechanical refrigeration methods such as vacuum pumping. Both cryostats used in the experiments, Triton<sup>TM</sup>DR and Heliox<sup>TM</sup>AC-V are closed-cycle cryostats, which utilise pulse tube refrigeration technology.

The pulse tube refrigerator (PTR) was first described by Gifford and Longsworth in 1959 and uses as the Stirling and Gifford–McMahon cryocoolers a regenerative cycle [84]. In contrast to Stirling and Gifford–McMahon cycles, the pulse tube refrigeration cycle must be considered for each element in the gas. Each molecule can follow a different sequence of transitions resulting in a cooling effect throughout the chamber [84].

A pulse tube refrigerator consists of a compressor, regenerator, pulse tube, orifice and a reservoir [84]. The compressor creates a pressure wave, with gas forced through the regenerator. The regenerator's gas-wave causes liquidation of the gas in the helium cup, absorbing heat from the cold part.

This heated, compressed gas passes through the pulse tube and passes through the orifice to the reservoir. Since the temperature of the gas-wave is higher than that of the reservoir, heat is transferred through the warm heat exchanger at the end of the pulse tube. Flow stops when the pressure in the pulse tube is reduced to the average pressure. The piston then moves up, expanding gas in the pulse tube. This draws gas from the reservoir through the orifice and the gas flows through the cold end heat exchanger, cooling the sample holder [84].

Cryostats enable experiments to be conducted at ultra low temperatures. The physics of the system can be modelled approximately with temperature  $T \approx 0$  K. The thermal energy is nearly negligible , which is useful for solid state physics, but heating from the sample must be accounted for.

#### D.2.1 Triton<sup>TM</sup>DR

The Triton <sup>TM</sup> consists of two cooling technologies together. Two pulse tube refrigerators; one to cool the outer chamber to 50 K, and inside that, one to cool the next inner chamber to 4 K. This next chamber, the second stage, holds the second cooling system the reciprocating dual absorption pumps for the dilution refrigerator [81].

Dilution refrigeration is a technology first proposed by H. London in 1951, see p. 35 in [82], and has in later years been developed and understood further. The fridge uses a mixture of the Helium isotopes <sup>3</sup>He and <sup>4</sup>He which is cooled to below a critical temperature, causing it to separates into two phases. Both phases have a temperature dependent concentration of <sup>3</sup>He and <sup>4</sup>He. The enthalpy of <sup>3</sup>He in the two phases is different, which can be used for a cooling effect, see p. 35 in [82]. When <sup>3</sup>He is moved from the "diluted phase", rich in <sup>4</sup>He to the "concentrated phase", rich in <sup>3</sup>He.

By pumping the <sup>3</sup>He away from the liquid surface in the still, osmotic pressure moves liquid <sup>3</sup>He from the mixing chamber. On the way from the mixing chamber the liquid <sup>3</sup>He passes through a series of heat exchangers. The two absorption pumps inside the second stage take in turn to pump the still to a low pressure and a special collector controls the flow of <sup>3</sup>He back into the dilution unit, see p. 36 in [82].

Cooling is continuous, since the two sorbs work together. The lower <sup>3</sup>He stage is held at 400 mK [81] continuously and acts as a cryopump for the selfcontained system of <sup>3</sup>He and <sup>4</sup>He, connected with heat exchangers to the mixing chamber where the samples are mounted. The two major stages in the Triton (400 mK and the 3 K) have cooling powers of 200  $\mu$ W and 10 mW respectively [81].

#### D.2.2 Heliox<sup>TM</sup>AC-V

The single vacuum chamber of the Heliox is initially pumped to a pressure of  $10^{-6}$  mbar and the first stage pulse tube refrigerator starts cooling. When stage one has reached 3 K the cooling cycle is started. The heat switch is opened, connecting the adsorption pump to the second stage, the adsorption pump is heated to about 30 K and <sup>3</sup>He expands. The pulse tube refrigerator cools the gas to around 3 K [80].

The helium can then be dumped into an expansion chamber, which provides more cooling. When the heat switch is closed, the adsorption pump is cooled, which starts the pumping of helium from the pot [80]. Around the base temperature of 300 mK, the cooling power of the cryostat is important for heat budgeting. At 350 mK, the cooling power is  $100 \,\mu$ W [80], assuming that radiation shields are mounted.

## Appendix E

# The SIN junction

The superconductor-insulator-normal metal junction provides the possibility of a tunnelling current. For an electron to tunnel through the barrier, there must be an empty hole in the density of states on the other side. The tunnelling probability does thus depend on the electron densities both sides of the insulator. All possible electron energies must be considered, so the product of electron densities is integrated over all energies, as shown in equation (E.1), see p. 75 in [16]. The electron energies can be shifted using a voltage bias, V, which encourages the current in a certain direction.

$$I_{SIN}(V,T) = \frac{1}{e R_{SIN}} \int_{-\infty}^{\infty} n_s(\epsilon) \left[ f(\epsilon,T) - f(\epsilon + e V,T) \right] d\epsilon$$
(E.1)

The normalisation, with the electron charge and resistance  $R_{SIN}$  is determined when the superconductor is in its normal state. This gives a normal metal–insulator–normal metal relation from classical circuit theory. For a given temperature, the voltage bias can raise the energy of the superconductor's electrons above the energy gap. This breaks the superconductivity and the superconductor turns into a normal metal. However, the resistance of the insulator remains and motivates the  $R_{SIN}$ -factor as part of the transmission coefficient [85].

Golubev, Kuzmin, and Willander presented a more detailed model for equation (E.1) [85], taking into account the different temperatures of the normal metal and the superconductor. By applying a voltage bias to the normal metal, the I-V characteristics depend on the temperature of the normal metal. This provides the possibility to build super sensitive thermometers with *SIN*-junctions.



**Figure E.1** – The I-V characteristics of the *SIN*-junction for four temperatures below the critical temperature  $T_c$  and the normal resistance, above the critical temperature