ABSTRACT

ANDREEV SPECTROSCOPY MEASUREMENT OF GaMnAs SPIN POLARIZATION

by Diana Dahliah

Our aim is to reliably extract the spin polarization of GaMnAs from conductance measurements using the Andreev Reflection (AR) technique. The contact resistance at the superconductor/ferromagnet interface contains the information of the Andreev Reflection and was measured using Circular Transfer Line Method (CTLM) instead of the commonly used existing geometries. Our technique has the advantage of eliminating the spreading resistance, which is a problem in the existing geometries. It also produces better defined conductance curves closer to idealized theoretical models. Finally, our measurements suggest that some of the published work in the literature has been observing a Schottky barrier and mistakenly assuming the phenomenon to be due to Andreev Reflection. This lead to erroneous estimates of spin polarization and added unnecessary fitting parameters to the existing models that describe the AR effect.
ANDREEV SPECTROSCOPY MEASUREMENT OF GaMnAs SPIN POLARIZATION

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Chapter 1: INTRODUCTION

There is an array of applications and some basic science phenomena that depend on the spin polarization, injection, transport and detection in different materials. This makes it important to reliably measure the spin polarization of such materials. Several techniques have been proposed and utilized to estimate a material’s spin polarization, including tunneling magnetoresistance (TMR), light-emitting diodes (LEDs), and Andreev Reflection (AR). The Andreev Reflection technique has advantages over the other techniques due to the simplicity of the fabrication steps, having no restrictions on the sample geometry, and having the ability to perform the measurements without applying a magnetic field. Andreev Reflection is a universal technique applied to many materials such as transition metal elements, half metals, metallic alloys of transition metals and ferromagnetic semiconductors. Yet Andreev Reflection-based measurements of spin polarization of highly resistive ferromagnets, like ferromagnetic semiconductors, are problematic and have so far given conflicting results that are hard to understand properly. One major source of error is the large spreading resistance that results from the resistive material being characterized.

One material that has been of significant interest in studies using Andreev effect is GaMnAs, which is a diluted magnetic semiconductor (DMS), where the semiconductor GaAs is doped with the transition metal ion Mn$^{+2}$. Mn ions couple together to produce along range ferromagnetic ordering. GaMnAs is a P-type ferromagnet due to manganese atoms which act as acceptors. To get collective ferromagnetic behavior, at least 2% of Ga atoms have to be replaced by Mn ions during the molecular beam epitaxy growth of the crystal at about 250 C. The Mn$^{+2}$ ion acts as a shallow acceptor or as a deep level double donor depending on whether it is substitutional or interstitial. Substitutional Mn ions are the ones that replace Ga atoms and reside on their lattice site, while interstitial ions are the ones that settle in between lattice sites and do not replace a host Ga atom. The hole carrier concentration decreases due to interstitial Mn ions, which reduces the Curie temperature and electrical conductance. The effect of interstitial Mn ions can be reduced by annealing GaMnAs at low temperature, which helps to remove interstitial Mn from
the bulk of GaMnAs to the surface and increases the hole density and ferromagnetic transition temperature.\textsuperscript{7,8} GaMnAs is one of the promising candidates for spintronics technology applications that utilize both the charge and the spin of current-carrying electrons.\textsuperscript{10} It also has a strong spin-orbit coupling, which leads to a strong coupling between its electric and magnetic properties. Different magnetotransport effects have been seen in GaMnAs such as anisotropic magneto resistance, the anomalous hall effect, tunneling magnetoresistance, and current induced domain wall motion.\textsuperscript{9} Fig. 1.1 shows some of the magnetic and transport properties of GaMnAs. Fig. 1.1A) shows the strong relation between the hole concentration and the curie temperature of GaMnAs, while Fig. 1.1B) shows the effect of annealing on the Curie temperature and the behavior of magnetization and the resistivity curves for GaMnAs as function of temperature above and below curie temperature.\textsuperscript{8}

We studied the spin polarization of the resistive ferromagnet Ga$_{1-x}$Mn$_x$As using the Andreev Reflection effect. Andreev Reflection is a popular technique used to extract the spin polarization of ferromagnets. It occurs because electrons inside a superconductor can’t exist as quasiparticles.
(i.e. as an individual electron with spin up or spin down state). Rather, they should be in a form of a Cooper pair with net zero spin. At the interface between a non-superconductor and a superconductor an incident electron approaching the interface from the non-superconductor side will have no way to keep traveling (i.e. of being transmitted) into the superconductor unless it pairs with another electron with the opposite spin. This is usually achieved by the reflection of a hole in the opposite direction of that electron and thus, the creation of an electron pairing with the original one in order to proceed into the superconductor. These electron pairs are called Cooper pairs and the binding energy between them is the superconducting energy gap.

If the non-superconducting material is a ferromagnet the electrons can’t always find electrons of the opposite spin with which to pair, and so the current across the interface is suppressed. The reduction in conductance of the interface below the superconducting gap is directly dependent upon the imbalance between the density of conduction electrons with spin up and spin down (i.e. the degree of spin polarization) in the ferromagnetic material. From the correlation between the suppression in the interface conductance and the spin polarization of a ferromagnet, the spin polarization of any non-insulating ferromagnet can, in principle, be studied using Andreev reflection.

In chapter 2 of this thesis we introduce the theoretical basis of Andreev reflection at the interface between a ferromagnet and a superconductor and the theoretical model used to extract the spin polarization value from the measured Results. We also discuss the two common geometries that have been used to study the Andreev reflection at the interface between ferromagnets and superconductor and point to some of their non-idealities. In chapter 3 we talk about our proposed new geometry, which utilizes the circular transfer line method to eliminate the spreading resistance effect, and discuss the experimental setup and sample geometry. In chapters 4 and 5, results and analysis are discussed for two sets of samples: the first set was designed to be very similar to previous studies that used point contact Andreev Reflection geometry, while the second set studied Andreev reflection in highly transparent interfaces. Finally, the conclusions are given in chapter 7.
Chapter 2: Theory

The Andreev Reflection (AR) spectroscopy at a ferromagnet superconductor (F/S) interface has been used as a simple technique for measuring the spin polarization of ferromagnetic materials, spin-flip scattering at the interface, and the superconducting gap.\textsuperscript{11,12,13} AR is a novel process that occurs at the interface between a superconductor and a non-superconductor. Conduction electrons in a superconductor (super-current) traveling as pairs are known as Cooper pairs. Each pair has one electron with spin-up and one with spin-down. Assuming that there is no barrier at the interface no quasi-particle states (i.e. individual electron states with either spin up or down) are provided inside the superconductor. So an incident electron with energy less than the superconducting energy gap moving towards the interface can’t enter the superconductor as a quasiparticle, since it is a forbidden state. The only way for the incident electron to enter the superconductor is by forming a Cooper pair at the interface through pairing with an electron with the opposite spin from the Fermi sea of the normal conductor at the interface. As a result of Cooper pair formation, a hole with the opposite spin will form moving with the opposite group velocity and the inverse direction of the incident electron.\textsuperscript{13,14}

The AR probability depends on the spin polarization of the non-superconductor and falls between two extremes. The first is when we have a clean interface with a normal (i.e. a non-ferromagnetic) metal, where the current in the metal is un-polarized. When AR takes place at the normal metal/superconductor interface, the conductance doubles compared to its normal state because the current doubles due to the formation of the Cooper pairs. The other extreme is at half-metal/superconductor interfaces, where half metals are 100\% spin polarized materials.\textsuperscript{15} In this case the AR process cannot occur because the incident electrons at the interface cannot find a ‘companion’ electron with the opposite spin. As a result the conductance goes to zero (i.e. the resistance of the interface becomes infinite). Typical ferromagnets fall between these two extremes, where the imbalance in spin up and spin down electrons (i.e. spin polarization) suppresses the AR process\textsuperscript{16,12} so the probability of AR is limited by the minority carriers in the metal. For a perfect interface between a superconductor and a ferromagnet, the conductance at zero bias voltage is given by

\[ G(0) = 2G_0(0)(1-P) \]  \hspace{1cm} (2.1)
Where $G_n(0)$ is the conductance when the applied voltage is larger than the superconducting energy gap and $G(0)$ the conductance at zero bias voltage.\textsuperscript{12} The conductance gets suppressed depending on the abundance of minority spin electrons (i.e. polarization) as shown in Fig. 2.1. So we can use the correlation between the suppression of Andreev reflection at the interface and the conductance to find the polarization of ferromagnetic materials, like GaMnAs.\textsuperscript{17,18,19}

Fig. 2.1: Normalized conductance, $G$ for different values of the spin polarization. Non-magnetic metals have $G = 2$ at zero bias, while half-metals have $G = 0$. [ from Ref.20]

\section{2.1 Calculating the Spin Polarization from AR Measurements}

Spin polarization is usually defined as $\frac{n\uparrow-n\downarrow}{n\uparrow+n\downarrow}$ where $n\uparrow, n\downarrow$ are respectively the carrier densities of spin up and spin down electrons at the Fermi energy.\textsuperscript{11} The spin polarization found from AR is given by $\frac{i\uparrow-i\downarrow}{i\uparrow+i\downarrow}$, where $i\uparrow, i\downarrow$ is the current for spin up, spin down respectively, which is proportional to $\langle N\nu \rangle$ or $\langle n\nu^2 \rangle$ depending on whether the contact at the interface between the non-superconductor and superconductor is ballistic or diffusive. The conductance in the ballistic limit is given by $\textsuperscript{12}$:
\[ G = \left( \frac{e^2}{2\hbar} \right) (n\nu) A \] (2.2)

Where \( e \) is the electron charge, \( h \) is the plank’s constant, \( n \) is the density of electrons at the Fermi level, \( \nu \) is the electron velocity at the Fermi level, and \( A \) is the total area of the contact. From Equ. 2. 2, it is seen that \( G \) is proportional to \( \langle n\nu \rangle \), in the ballistic limit. In the diffusive limit the conductivity is proportional to the relaxation time \( \tau \) and the conductance is given by\(^{12}\):

\[ G = \frac{e^2}{h} \langle n\nu^2 \rangle \frac{A}{L} \] (2.3)

\( L \) is the length of the disordered region. Equ 2.3 shows that the conductance, and thus the polarization, is determined by \( \langle n\nu^2 \rangle \). So the polarization from AR doesn’t follow the common definition of \( P_n \), since the transport is not determined by the density of state (DOS) only, and the only case the two values of \( P \) will be equal is when the Fermi velocity for the two spin currents is the same.\(^{21,22}\)

### 2.2 The Modified Blonder-Tinkham Kalpwik Model (mBTK)

The AR technique is used to find the spin polarization of a ferromagnetic film by analyzing the conductance-versus-voltage curve of the ferromagnet/superconductor interface using the mBTK model.\(^{23}\) The (BTK) model was first used to analyze the conductance curve at the normal-metal/superconductor interface as a general case, where not all incident electrons within the superconducting energy gap will be Andreev reflected. A portion of them will be normally reflected due \( \delta \)-function potential barrier at the interface, which can arise due to an oxide layer or Schottky barrier. The strength of this barrier is usually given by a dimensionless parameter \( Z \).\(^{13,14}\) So the two possible options for an incident electron toward the interface with energy less than the superconducting energy gap is either to get normally reflected, with probability \( B \) proportional to \( Z \) or Andreev transmitted with probability given by \( C \), the BTK model finds the two possibilities \( C \) and \( B \) as function of \( Z \) and incident electron energy \( E \) as shown below, where \( u \) is the relative incident energy of electron to superconductor Fermi level.\(^{13}\)

\[ C(E) = \frac{\mu^2}{1 + Z^2 (1 - \mu^2)^2} \] (2.4)
The BTK model was modified to find the spin polarization for a ferromagnet by studying Andreev reflection at Ferromagnet/ superconducting interface, where AR is suppressed due to imbalance of spin up and spin down electrons at the Fermi level in the ferromagnet.\textsuperscript{12} The BTK model was generalized to include P (polarization) as an additional fitting parameter for the conductance curve. The modified BTK model divided the current into unpolarized and polarized current, where the unpolarized current will behave as normal metal/ superconductor interface, and the conductance given by BTK model is equal to\textsuperscript{24}

\[ G = \int_{-\infty}^{\infty} \frac{df(E-v,t)}{dv} \left[ 1 + C_n(E,z) - B_n(E,Z) \right] dE \]  

(2.6)

where \( C_n \) and \( B_n \) are the Andreev transmission and normal reflection probabilities for unpolarized current.\textsuperscript{24} The Z factor now includes the mismatch in velocity between the superconductor and ferromagnet Fermi levels. Since the electron concentration in a semiconductor is much lower than in the superconductor this leads to a potential step when the two Fermi levels align at the interface. So Z now has two components, the Z barrier which describes the reflections at the interface due to the tunneling barrier (other mechanisms might be included under this term) and Z from the mismatch of Fermi surfaces as shown in Equ (2.7) where \( r \) is the mismatch in the Fermi velocities between the superconductor and the ferromagnet.\textsuperscript{13,25}

\[ Z = Z_{\text{barrier}}^2 + \frac{(1-r)}{4r} \]  

(2.7)

For the polarized current it will behave as a half metal channel where for electron with energy less than superconducting energy gap, there is no AR allowed, and the conductance is given by\textsuperscript{24}

\[ G = \int_{-\infty}^{\infty} \frac{df(E-v,t)}{dv} \left[ 1 + C_p(E,z) - B_p(E,Z) \right] dE \]  

(2.8)

\( C_p \) and \( B_p \) are the probability for Andreev reflection, and normal reflection,\textsuperscript{24} so the conductance at the interface between a superconductor and ferromagnet is given by:

\[ G(v) = (1-p)G_n(v) + pG_p(v) \]  

(2.9)
The zero temperature conductance for unpolarized current and fully polarized for incident electron with energy less and larger than superconducting energy gap are given by table 2. An example is given in Fig. 2.2 showing the utilizing from the mBTK model to extract the spin polarization of GaMnAs by fitting the experimental conductance curve to theoretical one using the spin polarization energy gap and, temperature as adjustable parameters beside the elastic scattering Z parameter to fit the experimental conductance curve to get the polarization of GaMnAs.

Fig.2.2: the normalized conductance fits to the modified BTK model for Sn/ GaMnAs contact with $R_c=68 \ \Omega$ measured at $T=1.2$ K. [from Ref. 27]

Table 1: the total current at the interface in different regimes the notations which used below are $B = \frac{V}{\sqrt{V^2 - \Delta^2}}$ $F(x) = \text{Cosh}^{-1} \frac{2Z^2+x}{\sqrt{(2Z^2+x)^2-1}}$ [from Ref.26]

<table>
<thead>
<tr>
<th>Regime</th>
<th>$eV&lt;\Delta$</th>
<th>$eV&gt;\Delta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ballistic nonmagnetic</td>
<td>$\frac{2(1+B^2)}{B^2 + (1 + 2Z^2)^2}$</td>
<td>$\frac{2B}{1 + B + 2Z^2}$</td>
</tr>
<tr>
<td>Ballistic half-metal</td>
<td>0</td>
<td>$\frac{4B}{(1 + B)^2 + 4Z^2}$</td>
</tr>
<tr>
<td>Diffusive nonmagnetic</td>
<td>$\frac{1 + B^2}{2B} - Im[F(-iB) - F(iB)]$</td>
<td>$BF(B)$</td>
</tr>
<tr>
<td>Diffusive half-metallic</td>
<td>0</td>
<td>$BF[\frac{(1 + B)^2}{2} - 1]$</td>
</tr>
</tbody>
</table>
2.3 The Two geometries used to study AR in GaMnAs

The two geometries used to study the spin polarization of ferromagnets by AR technique are the planar geometry \(^{29,28}\), and point contact Andreev reflection (PCAR) \(^{11,12,19,23}\). Both techniques suffer from multiple problems, as shown below, when the ferromagnetic material has a high resistivity.

- **Planar geometry**:
  Computing the spin polarization of GaMnAs by AR was first studied using planar geometry \(^{28,29,30}\). This geometry had either two layers; ferromagnet/superconductor or a superconductor/insulator/ferromagnet trilayer. The measurements of conductance between a ferromagnet and a superconductor was carried out using four probe technique, where two contacts are attached to the back of the conducting substrate, and the two others at the top of superconductor as shown in **Fig. 2.3**. This geometry didn’t give reproducible results for the spin polarization of GaMnAs, for example S. H. Chun et.al (2003), \(^{29,30}\) were only able to get somewhat sensible AR spectra from one of several superconducting material types on GaMnAs. Also the results from this geometry suffered from difficulties in extracting the actual interface resistance from the measured one.

**Fig. 2.3**: A) A schematic of the (Ga,Mn)As heterostucture and the contact scheme. B) Normalized conductance spectrum of a GaMnAs/Ga junction. [From Ref.30]
- **Point contact Andreev reflection:**

Point Contact Andreev Reflection (PCAR)\textsuperscript{11, 12, 23, 27} is the most commonly used AR-based method to study the spin polarization of ferromagnets. The PCAR technique is the study of the conductance between a superconducting sharp tip and a ferromagnet surface (just like the scanning tunneling microscope), using a quazi-four probe technique to measure the AR contact resistance as a function of voltage at 4.2K, as shown in **Fig.2.4A**. The technique is quite simple and practical since the same tip can be used with different ferromagnetic films to measure the spin polarization of each film. On the other hand, the technique also suffers some drawbacks, such as the presence of a native oxide layer at the ferromagnet surface.\textsuperscript{25,31} The oxide might cause significant spin flipping because it is usually amorphous and complex in stoichiometry, and is Mn-rich. This is especially problematic since MnO is an antiferromagnet and causes too much spin flipping at that dirty interface. One significant problem is that the four probe geometry as shown in **Fig2.4B** is not ideal. The measured contact resistance includes an extra resistance (on top of the actual interface resistance) due to the spreading resistance on the film. This resistance can be much larger than the actual contact resistance at the ferromagnet/superconductor interface,\textsuperscript{11} and does not participate in the AR process.

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**Fig.2.4:** A) The setup for PCAR technique by Nadgornie et.al.[from Ref.32] B) The measured resistance by four probe technique contains extra spreading resistance.[ from ref.11]
Both geometries are suffering from the extra spreading resistance effect on their results. This extra resistance leads to a significant broadening of the conductance curve, where the superconducting gap appears to be much wider than the actual accepted values for the bulk superconductor. This broadening cannot be real, because the superconducting gap is smaller at the interface than it is in the bulk. Yet, this broadening occurs because a small fraction of the actual voltage is typically across the interface and the rest of that voltage drop is across the additional resistance, this spreading resistance effect can’t be eliminated experimentally or estimated theoretically. The conductance curve from this technique didn’t match with the theoretical fitting model mBTK directly. As a result a new fitting parameter has introduced which is the spreading resistance beside the multiple fitting parameters used in the mBTK model to fit the experimental conductance curve. The additional fitting parameter which used has a strong effect on the polarization result, since the superconducting energy gap is directly proportional to spreading resistance and by defining the spreading resistance incorrectly or not included this give a wrong value for the super conducting energy which is used as fitting parameter too which directly affect the value of P. Determining the value of the spreading resistance is a highly inaccurate process and many fitting parameters are necessary because of the inability to experimentally isolate the actual AR-contributing interfacial resistance.

To overcome these issues, we utilize the Circular Transfer Liner Method (CTLM) to measure the contact resistance. This method only needs one lithography step to be realized, and is a good way to provide information about contact resistance, which can include information about AR. Our technique eliminates the spreading resistance, and gives the exact value for the contact resistance.
Chapter 3: New Andreev Reflection Geometry & Sample Fabrication

3.1 Circular Transfer Line Method to Extract AR

The Circular Transfer Line Method (CTLM) was developed to extract the specific contact resistance at metal/semiconductor interfaces and the sheet resistivity of semiconductor films with high accuracy. The circular symmetry produces uniform current flow and eliminates the requirement for mesa isolation at the contact pattern, which reduces the patterning steps in preparation of the samples to only one,\textsuperscript{34,35} making the process much simpler. The CTLM method is used in this research to extract the contact resistance between GaMnAs and Nb which will provide us with the information about AR and the spin polarization of GaMnAs.

Our samples are Nb microstructures deposited on GaMnAs in the form of circular discs with diameters either 400 or 600 micrometers, separated from the surrounding Nb by circular gaps of bare GaMnAs of different widths varying from 4 to 80 micrometers as shown in Fig. 3.1. The resistance of each feature was measured by forcing a DC current between the inner discs and the outer Nb and measuring the voltage drop between the inner disc and the outer Nb using a DC nano voltmeter and utilizing the four probe technique. Measurements were mostly done at 4.2K, where Nb becomes a superconductor at temperatures below 9.2K. Other detailed measurements were also taken at room temperature of about 293 K to compare the results or at variable temperature to observe the behavior of the contact resistance with temperature. The main measurement in the Andreev reflection spectroscopy technique is to apply a small voltage to the superconductor/ferromagnet interface and measure the electrical current, or vice versa, then increment that voltage and measure the current repeatedly until the applied voltage is well above the superconducting energy gap per electron. The shape of the differential resistance will then depend on the spin polarization of the ferromagnetic material. From the measured voltage and the current, the total resistance of each feature can be calculated by using the definition of $R = \frac{dV}{dl}$ and by studying the behavior of the differential resistance of each feature at 4.2K as a function of voltage. When the voltage across the interface is greater than the superconducting energy gap ($\Delta$), no AR can occur and the total resistance will remain constant, independent of the applied voltage (assuming that there is no Schottky barrier at the interface). On the other hand,
for voltage across the interface less than $\Delta$, the contact resistance between GaMnAs and Nb will be suppressed (or enhanced) due to the spin polarization of GaMnAs, so the total measured resistance will vary as a function of voltage within $\Delta$. Our aim in this research is to eliminate the spreading resistance effect and isolate the contact resistance and its voltage from the measured resistance and measured voltage, which can be done by using the CTLM method. The contact resistance and the resistivity of GaMnAs can be extracted through a linear relation between measured resistance and the width of each gap, and by assuming that the measured resistance consists of two resistances in series: the bulk resistance of GaMnAs, which is independent of the applied voltage, and the contact resistance, which includes the information of AR. The bulk resistance and its voltage are eliminated using the linear fits from the CTLM to end with the voltage and the current of the contact resistance which we need to find the contact conductance as function of its voltage. This conductance of the interface is used to extract the spin of polarization of GaMnAs using the mBTK model.

![Cartoon Diagram](image)

**Fig.3.1:** a cartoon diagram shows our features, the red is Nb and the blue is GaMnAs.

### 3.2 Sample Fabrication

Our new geometry needs one lithography step to pattern the circular features on the GaMnAs. Different samples have been studied from the same wafer of GaMnAs but with different Curie temperatures. Also the surface properties have been taken into account by studying the effect of the existence of native oxide at the interface between Nb and GaMnAs. The native oxide layer was left in place when depositing Nb on some of the samples, while that oxide was stripped off
using an H$_2$SO$_4$ acid etchant just before Nb deposition. The steps of the fabrication process of the Nb/GaMnAs microstructures are listed below:

- We start with GaMnAs films of thickness 100nm and nominal Mn concentration 6%. These samples are grown using molecular beam epitaxy (MBE) by the group of Prof. Jacek Furdyna in the Physics Department at the University of Notre Dame.
- The GaMnAs sample is treated with water and acetone to remove any salt and organic compounds before the photolithography.
- Apply the lift off resist (LOR):

The GaMnAs wafer is placed in the center of the photoresist spinner, and secured by applying vacuum, then the lift off resist LOR is dispensed on the wafer using a dropper. After that the sample is spun in two steps: the first step lasts 10 seconds and runs at a speed of 500 rpm, and the second step 40 seconds at a speed of 3000 rpm. This lets the LOR spread over the whole surface uniformly forming a thin film. The sample is then baked on a hot plate for 10 min at 150°C in order to harden the LOR.

- Apply the photoresist (S1813):

The positive photoresist (S1813) is spun over the LOR layer using the photoresist spinner in two steps. The first one is for 10 seconds at a speed of 500 rpm, and the second lasts 40 seconds at a speed of 3000 rpm, then the sample baked at 105°C for 1 min.

- Light exposure:

The sample is transferred to a mask aligner and exposed to ultraviolet/ violet light through the mask for 4.6 seconds. The photoresist that gets exposed to light, becomes weaker and is removed by developing the samples using a chemical developer (CD26) for 40 seconds. The sample is then rinsed with water and dried with nitrogen gas.

- Nb deposition by sputtering:

Two samples are then attached to a small copper block and loaded into a metal deposition chamber. One of the samples has its surface treated with sulfuric acid to remove the native oxide
before Nb deposition process and the second has the native oxide on its surface. About 70nm of Nb are then deposited on the sample surface.

- Finally the metal was lifted off by dissolving the resist in acetone. This leaves the circular discs of Nb, which are separated from surrounding Nb by circular gaps as shown in Fig.3.2.

Fig.3.2: A) image of the circular features after the liftoff step by SEM, the black is GaMnAs and the gray is Nb. B) One feature of diameter of 400micrometers and width gap of 80 microns, an indium drop which is used to solder the wires is shown in the picture to compare its size to dimension of the features. C)image took by SEM Nb deposited on a sample which was cleaved and images tool for it from side to view its cross sectional of channels, as shown above you can see the bi layer resist of s1813 and lift off resist and Nb too. D) This image shows the undercut which arise from that the LOR dissolve faster than S1813 in the developer this give the undercut which is here about 157 nm which is enough for the acetone to reach the liftoff after deposition to remove the metal from unwanted areas.
Chapter 4: Measurements and Analysis of GaMnAs/native oxide/Nb

The Andreev Reflection at the interface between a ferromagnet and a superconductor is sensitive to the properties of the interface,\textsuperscript{30} so we start by studying the effect of the native oxide at the interface between GaMnAs and Nb on Andreev Reflection. Several effects have been studied at the interface between GaMnAs and Nb which are:

- The effect of Curie temperature on AR probability. The Andreev Reflection at GaMnAs/Nb with native oxide on the interface is studied for two wafers of GaMnAs with different Curie temperature (65K, 100K), since experimentally has been shown that the Curie temperature increases with increasing the Mn ions concentration, which provides the magnetic properties of GaMnAs\textsuperscript{8}.
- The effect of using the cold and hot soldering tip, to fabricate the four-probe connections, on the interface and AR probability.

4.1 The GaMnAs/native oxide/Nb [(Tc=65K)]

The first set of measurements was done on a GaMnAs film with a Curie temperature of about 65K. The native oxide on GaMnAs was kept on the surface to simulate the same characteristic of the surface in PCAR technique, where the surface is open to the ambient, and has a native oxide layer on it.\textsuperscript{26}

The GaMnAs wafer was patterned with CTLM and the gap in the Nb discs is either 4, 6, 8, 10, 15, 20, 40, or 80μm. The inner circle for all features has a diameter of either 600μm or 400μm. Our naming convention for each feature uses the inner diameter then the gap width. For example feature 600μm-8μm has an inner diameter of 600μm and a width of the circular gap in Nb of 8μm. The DC current was sent from the inner disc of Nb to the outer Nb structure. It travels from the inner Nb disc radially into GaMnAs gap then into the outer Nb structures. The four probe technique is used to measure the total resistance of each feature where two wires are connected to the inner Nb disc and two wires to outer Nb using cold tip pressing of indium solder as shown in Fig4.1. The purpose of pressing the indium solder at room temperature rather than use a hot tip is to avoid any inter-diffusion of Nb and GaMnAs at the interface. This keeps the interface as
sharp as possible. The measured resistance in the four probe technique is equal to the bulk resistance of GaMnAs and the contact resistance of the two interfaces through which current flows.

![Image of four probe technique]

**Fig.4.1:** A) Cartoon showing the four probe technique to measure the contact resistance. B) The two wires connected inside the circular feature with diameter 400µm, and a gap width of the gap 8µm using indium soldering.

We focus in this part on the effect of the presence of the native oxide layer between the GaMnAs and Nb on the measured ‘AR’ effect. Andreev Reflection is sensitive to the transparency of the interface, which depends on the treatment of the surface of GaMnAs prior to the deposition of the Nb layer. The first set of circular patterns was made on an as-grown GaMnAs wafer with Curie temperature 65K. The resistance of each feature was measured using the four probe technique at 4.2K, where the measurements were done with the samples inserted inside a liquid Helium Dewar. The differential resistance for each feature is drawn as a function of voltage as well as the characteristic curve of (I-V) as shown in Figs4.2 and 4.3. Figs4.2A), and 4.2C) are showing the current curve as function of voltage which its behavior is non-linear for the two circular features with circular disc diameter of 400µm and a gap width of 8µm, and 6µm. The Figs 4.2B), and 4.2D) are the differential resistance for the same features in Figs 4.2A), and 4.2D) respectively, the conductance almost is constant for large voltage above .5V. The differential resistance is enhanced around the zero voltage as expected from the AR effect. Fig4.3 is showing the behavior of the (I-V) and the resistance curve at 4.2K for another two circular features with diameter 600µm. the (I-V) curve is non-linear for the both features with gap width 20µm, and 80µm as shown in Figs 4.3A), and 4.3C) respectively. Also the differential
resistances for these features are showing the same behavior for the previous one which is almost constant at high voltage values then starts to enhance in low voltages below .5V. The resistance of all features that measured varies with small voltages and has a maximum peak at zero bias voltage which is consistent with the expected behavior of AR. But mBTK model expects the change in the resistance of the interface resistance should start at the theoretical position near the bulk superconducting energy gap which is for Nb around 1.4mV. Our results are showing a huge broadening in the conductance curve. This broadening in the resistance curve was explained by the two geometries that have been used to study Andreev Reflection (i.e. planar geometry and PCAR) due to the spreading resistance effect which comes from the resistive GaMnAs bulk. Since the measured resistance is not only the contact resistance, it includes a resistance from the ferromagnet.\textsuperscript{23} GaMnAs is a resistive ferromagnet, and its resistance can’t be ignored, which could be larger than the contact resistance. This will affect in resistance- voltage curve, which leads to broadening in the conductance curve as shown in Fig. 4.4.\textsuperscript{31} But if we examine the (I-V) curves carefully we see that the more likely cause of the change in resistance with voltage is the presence of a Schottky barrier at the interface, rather than due to the AR effect, since the I-V curves for the four features which are shown in Fig.4.2, and 4.3 are identical to the behavior of I-V curve for Schottky barrier, also the resistance for feature (400-6), and (600-20) as shown in Figs 4.2 D), and 4.3 B) their resistance is showing an enhancement by 300% by changing the voltage from .3V to zero volt, and this can’t be explained by AR effect since the maximum enhancement in the resistance is duplicate it’s value, when the ferromagnet is half metal. Also this broadening in our curve can’t be from the GaMnAs since if we compare the resistance for the four features in Figs4.3, and 4.2, its not increase linearly with increasing the gap of the bulk, which indicates this measured differential resistance is an interface resistance. To make sure whether we have a Schottky barrier at the interface or not, we studied the same features with same connections at room temperature, where Nb is not a superconductor anymore and AR effect can’t occur. If there is no Schottky barrier at the interface, we have to get a constant differential resistance curve as function of voltage.
Fig. 4.2: A) and B) the (I-V) curves for the two circular features with diameter 400µm, and gap width of 4µm, and 8µm respectively are non-linear as function of voltage. B) and D) are the differential resistance for the same features in figure A), and B) respectively. All the measurements were done at 4.2K for GaMnAs/oxide/Nb for as grown sample.
Fig. 4.3: A) & B) the (I-V) curves for the two circular features with diameter 600µm, and gap width of 20µm, and 80µm respectively are nonlinear as function of voltage. B) & D) are the differential resistance for the same features in figure A), and B) respectively. All the measurements were done at 4.2K for GaMnAs/oxide/Nb for as grown sample.

Fig. 4.4: PCAR spectrum of Pb/ GaMnAs at T=4.2 K, (black open circles) with spreading resistance fits, and the extracted PCAR spectra (red dashed line), after removing the contribution of spreading resistances. The fitting parameters are P=0.76, Z=0.00, Δ=1.09 meV, and × =4.831[from Ref.31]
The measured differential resistance and (I-V) curve for all features give the same results. Both the (I-V) and the differential resistance curves show that there is a strong Schottky barrier at the interface between Nb and GaMnAs. The effect of the Schottky barrier around zero voltage is weaker at room temperature compared to 4.2K since at low temperature the electrons don’t have enough thermal energy to cross the barrier. Fig. 4.5 Shows the (I-V) and the differential resistance curves for two features measured at room temperature. The two features have gaps 6 and 8µm and diameter of 400µm. Their differential resistance is less affected by Schottky barrier compared to 4.2K results, which is expected, where the differential resistance at zero voltage reduce from 1200Ω, and 600Ω at 4.2K to 250Ω at room temperature. The change in resistance for the features around zero voltage at 4.2K, is also most likely a result of the strong Schottky barrier. The effect of this barrier gets more pronounced at low temperature. So, most likely, there is no Andreev reflection effect observed even at 4.2K since the Schottky barrier acts as a high tunneling barrier which is strongly reducing the probability of AR. 27

Fig. 4.5: A), and C) the (I-V) curve for feature with circular gap of 6µm and 8µm at room temperature is not linear which indicates we have a Schottky barrier at the interface. B), and D) The differential resistance curve for the same features in A) and C) respectively, the differential resistance varies as function of voltage which indicates that we are having a Schottky barrier at the interface. These two features are with circular diameter 400µm for GaMnAs/oxide/Nb as grown sample the measurements done at room temperature.
Our results with native oxide between Nb and GaMnAs (which is similar to the interface used in the PCAR technique) show that the cause of the change in resistance in GaMnAs/superconductor interfaces is a Schottky not the AR effect. There are multiple indications that support our conclusion: First, the conductance curves for the F/S interface found by either the PCAR or planar geometry did not match with what expected ones according to the mBTK model. Also, the width of the curve is always significantly larger than that predicted by the mBTK model, which should equal twice the superconducting energy gap. For example in Fig.4.4 the measured resistance is 4.6KΩ while the contact resistance was estimated to be 1kΩ, and the rest of resistance is assumed to be a spreading resistance to explain the broadening in the curve. Another feature that is missing from the experimental results is the peaks near the superconducting gap. Also mBTK model predicts that the probability of AR at zero bias voltage to be constant as function of temperature below the critical temperature of the superconductor, but the results of PCAR shows that the peak of the resistance curve at zero voltage decreases as function of temperature, which is in contrast with the constant value which is predicted from mBTK. The experimental conductance curves found either by PCAR or planar geometry didn’t fit the theoretical conductance curve found by mBTK model properly and many empirical parameters have to be introduced to the model to fit the data such as: the effective temperature, the superconducting gap, the spreading resistance, inelastic and elastic scattering parameters, and the life time of Cooper pairs.

4.2 The Effect of Short Local Annealing on the AR and the Contact Resistance (GaMnAs/oxide/Nb)

In this section, we study the effect of the local short annealing (through the hot soldering tip) on the transparency of the interface and AR effect. A hot soldering tip of indium was used to make the connections of the four wires of the four-probe and to study its effects on the contact resistance. The short annealing was done at temperature of 560 °C for a few seconds for each connection. Its effects on an as grown sample with oxide are shown below in Fig. 4.6. We still get a Schottky behavior at room temperature and at 4.2K, but the strength of the barrier is reduced, and the value of the contact resistance decreased due to the short annealing. The Mn ions immigrate from inside GaAs to the surface, and/or Nb will diffuse to the GaAs substrate, due annealing, which can lead to more charge carriers at the interface. This causes the depletion
region between the GaMnAs and Nb to get thinner and the Schottky barrier height gets lowered. This in turn leads to a smaller variation in the interface resistance as a function of voltage and the peak of the differential resistance will get lowered at zero voltage compared to the resistance that we get by using the cold tip as shown in the previous measurements.

In Figs 4.6 A), and 4.6.C) is showing the differential resistance for the two features with circular disc 600µm and a circular gap 8µm and 80µm at 4.2K, the resistance is enhanced by 30%, and 25% at zero voltage compare to its value at high voltages, these results are so closed to the results in Ref.[11,31] where they used the point contact geometry, in their geometry they were not able to tell if this broadening in the gap is due the spreading resistance of GaMnAs since the non-ideality of four-probe technique, or its due the Schottky barrier, also in the two references mentioned above they didn’t give a characteristic curve for the (I-V), which we provide here which give a strong evidence that what has been measured is a Schottky effect not AR effect, also as showing in these two graphs i.e. in Figs 4.6A), and 4.6C) by varying the gap by 10times the total resistance changes only by 30% which means the hall contribution in the differential resistance is coming from the Schottky resistance at the interface. Figs 4.6.B), and 4.6.D) are showing the differential resistance at room temperature for the same features in Figs 4.6A, and 4.6C). the value of the resistance is lower than at4.2K which indicate that electrons are thermally tunneling through the interface, and by lowering the temperature to 4.2K the electrons don’t have enough energy to cross the barrier at low voltages which explain the enhancement in the resistance around zero in the cures at 4.2k. the resistance value is lowered compared to one in Fig.4.5, this due short annealing which either force the Nb to diffuse to interface or Mn ions to transfer to interface from the bulk which increase the charge carriers at the interface.
Fig 4.6: the differential resistance for two features of circular diameter 600µm and gap width of 8, and 80 µm at 4.2K as shown in A), and C), while B),D) the resistance measured at room temperature. The hot soldering used to study the effect of locally short annealing
4.3 GaMnAs/native oxide/Nb (Tc=100k)

The second set of circular features of diameter either 600 or 400µm and gap width between (4-80) µm were patterned on a GaMnAs wafer with Curie temperature 100K. The purpose of studying these higher transition-temperature samples is to study the transparency of the interface to spin polarization of the bulk, and to see if we can get any sign of AR with the presence of oxide at the interface between GaMnAs and Nb interface. The (I-V) curve and the resistance for each feature were measured at room temperature, where AR cannot occur, in order to find out whether our contact is ohmic or is rectifying, then at 4.2K where Nb is superconducting, in order to study the AR effect. The differential resistance curve as function of voltage shows that there’s a schottky barrier at the interface at room temperature and at 4.2K. But it’s strength is less than the one of the as-grown features. Furthermore, the differential resistance is less than that of the as-grown sample as well. The Schottky behavior gets stronger at 4.2K, as expected, and there’s no obvious indication of having AR. The resistance value decreases compared to the as-grown sample because interstitial Mn$^{2+}$ immigrate to the surface due annealing and the hole concentration at the interface is increased.$^{37,38}$

**Figure 4.7** is showing some results for the GaMnAs with Curie temperature 100K sample. The differential resistance for the three features with circular gap 10, 15, and 40µm measured at 4.2K as showing in **Figs 4.7A), 4.7C), and 4.7E)** respectively, the behavior of the resistance is the same for as grown sample with oxide on, the only difference is the value of the resistance is lowered and the strength of the Schottky barrier is get lowered since the depletion region at the interface reduced due to increase the hole concentrations at the interface. There is no clear sign for Andreev reflection. **Figs 4.7B), and 4.7E)** are the measured resistance for the previous three features(i.e.(10,15,40)) their resistance show that we have a Schottky barrier at the interface even at room temperature, which support our results that the enhancement in the resistance curves at low temperature is due Schottky barrier.

The effect of short annealing on the transparency of the interface is studied too, by using the indium hot soldering tip to fabricate the four-probe connections, the resistance at room temperature and at 4.2K is showing that we have schottky barrier, no AR was observed at 4.2K. The value of differential resistance has lowered by few ohms compare to the cold tip soldering
results, due either Nb diffuse into GaAs substrate or Mn\textsuperscript{+2} diffuse out to GaAs due short annealing. Annealing cause the depletion region at the interface to decrease, so the resistance value.

Fig. 4.7: the differential resistance as a function of voltage for several features using cold tip soldering at 4.2K as shown in A), C), and E), while B), D), and F) are done at room temperature for GaMnAs with Curie temperature 100K sample.
Chapter 5: Measurements and Analysis of GaMnAs/Nb

The oxide layer between Nb and GaMnAs was removed using H₂SO₄ etching before Nb deposition. The effect of having a clean Nb/GaMnAs interface on AR and the shape of the resistance curve is studied for two sets of samples with different Curie temperatures. The Curie temperature is used as an indicator of the Mn content inside the GaAs crystal and of the hole concentration inside GaMnAs. The change in AR effect at the interface due to changing the Curie temperature can indicate that Curie temperature is related to the spin polarization of the GaMnAs bulk or that the hole concentration has an effect on the Nb/GaMnAs interface, that modifies the AR curve. The effect of using the cold tip and the hot tip to make the connections of the four-probe technique investigated with its effects on the transparency of the interface, and its effect on the probability of AR, and on the contact resistance.

5.1 The GaMnAs/Nb (Tc=65K)

The second set of measurements is done for as-grown GaMnAs (i.e. not annealed GaMnAs that has the lowest Curie temperature) with Curie temperature 65K. The difference between this set of samples and the samples discussed in chapter 4, is that the GaMnAs native oxide film at the interface has been stripped off in these samples, while it was left intact in the samples discussed in chapter 4. The aim of these measurements is to find the effect of the native oxide film on the transparency of the interface, and the AR effect.

Since GaMnAs is a semiconductor, the contact between it and a metal could be ohmic, or Schottky (i.e. rectifying). This depends on the position of the Fermi level in the metal relative to that of the semiconductor. The bending of the energy bands creates a barrier at the interface, and the height of this barrier determines the behavior of the contact to be either ohmic or Schottky. So, we have to examine our contact resistance between GaMnAs and Nb whether it’s ohmic or not by measuring it at room temperature as a function of voltage.

The voltage and the current for each circular feature were measured using the four-probe technique at room temperature, and then the differential resistance was calculated as a function of voltage. The measurements of all features have shown linear (I-V) curves and decently horizontal lines of the resistance. This means that our contact is ohmic, or follows ohms law,
which states that current increases lineally with the applied voltage. **Fig.5.1** shows the (I-V) curves and resistance as functions of voltage at room temperature for the three features with different circular gap widths and disc diameters. **Figs. 5.1 A), 5.1 C), and 5.1 E)** show the behavior of voltage across each feature as a function of current, the voltage across the three features increases linearly with voltage, which clearly shows that we have an ohmic contact between Nb and GaMnAs. The resistance for the features (400-8), (600-6), and (600-10) at room temperature is given in the **Figs. 5.1 B), 5.1 D), and 5.1 F)** respectively as a function of voltage. The resistance for the three features is constant, which indicates the absence of a Schottky barrier. Comparing this with the voltage-dependent resistance of the samples with a native oxide film at the interface, it is clear that the Schottky behavior is due to the presence of the native oxide at the interface between Nb and GaMnAs. Furthermore, comparing the value of the resistance for the three features shows that, it changes only a little by changing the gap width, which indicates that the resistance of the bulk is small compared to the contact resistance at room temperature.

![Fig. 5.1: A), C), and E) The characteristic (I-V) curves as function of voltage for several features of GaMnAs/Nb as grown sample at room temperature. B), E), and F) are the resistance at room temperature the measurements are done at room temperature.](image-url)
The resistivity of GaMnAs and the contact resistance at the GaMnAs/Nb interface can be calculated using the CTLM. The calculated resistance of each feature is equal to the GaMnAs bulk resistance, which is proportional to the width of the gap, and the contact resistance of the interfaces through which current flows.\textsuperscript{40,41} The total resistance is given by\textsuperscript{42}:

\[
R = \left(\frac{\rho}{2\pi rt} W + 2R_c\right)C
\]  \hspace{1cm} (5.1)

where \(R\) is the measured resistance, \(\rho\) and \(t\) are the resistivity and thickness of the GaMnAs film, \(r\) is the radius of the inner Nb disc, \(w\) is the width of the gap, \(R_c\) is the contact resistance, and \(C\) is a geometrical correction factor given by: \(C = \frac{r}{w} \ln(1 + \frac{w}{r})\).\textsuperscript{41} Fitting the measured resistance for several features with different gap widths to Equ. (5.1), yields the value of the GaMnAs resistivity and contact resistance.\textsuperscript{43} Fig. 5.2 Shows two resistance measurement sets are taken at two different temperatures. Each of the sets is for several features as a function of gap width along with a straight line fit. From the fit at room temperature, we get a resistivity of GaMnAs equal to 9.42 m\(\Omega\).cm.

\[\text{Fig. 5.2: the total resistance as function of gap width at room temperature and 4.2K for as grown sample (GaMnAs/Nb) oxide removed.}\]
To study the effect of AR on the resistance curve, the resistance of each feature was studied as a function of voltage at 4.2K, where Nb is superconducting. The current-voltage curves for all features show a linear behavior, which indicates that our contact resistance at low temperature is ohmic too. The resistance for all features is constant for large values of voltage, which is expected since AR doesn’t occur when the applied voltage across the contact is larger than $\frac{\Delta}{e}$. Yet, the differential resistance has a dip around zero voltage. This dip occurs because the contact resistance of GaMnAs and Nb is suppressed due to AR which is dependent on the spin polarization of GaMnAs, when the voltage across the contact is less than $\frac{\Delta}{e}$. Fig5.3 shows the Ohmic behavior in one of the features with disc diameter 600µm, and a gap width of 6µm. The (I-V) curve is linear and its resistance is constant for large voltages. The resistance is reduced around zero voltage which indicates that the polarization of GaMnAs is less than 50%. Another feature of the curve is that around the apparent gap width there is a small peak, which could be due to spin flipping at the interface. Fig5.4 shows the (I-V) curve for the three features with circular disc diameter 600µm and a gap width 8, 10, and 15µm as well as the resistance of these features as a function of voltage. Both the (I-V), and the differential resistance curves for each feature show that we have an Ohmic behavior for the contact resistance at 4.2K. The reduction in resistance around zero voltage indicates that the samples have a low spin polarization. The differential resistance for the three features increased compared to its value at room temperature this is because the GaMnAs resistivity increased at 4.2K compared to room temperature as will be shown below. A first look at the data shows that the superconducting energy gap seems greater than its known actual value (1.4mV). This is the so-called broadening in the resistance curve and is due to the spreading resistance effect: The measured voltage in our technique is not only the voltage drop at the interface, which gives the information about AR, it also includes the voltage drop across the GaMnAs bulk. GaMnAs is a resistive material, so its resistance cannot be ignored. This extra resistance causes the broadening in the curves, or shifting the apparent $\Delta$ to higher values. Yet, our geometry allows us to eliminate this broadening resistance and extract the interfacial contribution alone.
Fig. 5.3: A) The (I-V) characteristic curve measured at 4.2K, B) (I-V) in small range to show the nonlinear behavior for the measured voltage due AR effect. C) The differential resistance as function of voltage at 4.2K. Both figures are for feature with circle diameter 600µm, and gap width of 6µm. for as GaMnAs with Tc=65K.

Fig. 5.4: A), C), and E) The (I-V) characteristic curve 4.2K, B), E), and F) the resistance as function of voltage at 4.2K. all features shown in this figure are for as GaMnAs grown sample.
To extract the contact resistance and its voltage on it and to eliminate the broadening effect, the CTLM method is used. The measured resistance consists of two resistances in series: the bulk resistance, which is proportional to the gap width and is voltage-independent, and the contact resistance, which varies as a function of voltage for \( V<\frac{\Delta}{e} \) and includes the information about AR. The overarching goal of this thesis research is to obtain this contact resistance and the portion of the applied voltage that appears across it.

The bulk resistance and its voltage are subtracted from the measured resistance to end up with the contact resistance and the portion of applied voltage that appears across it only. In this way we eliminate the spreading effect. To find the resistance of the GaMnAs bulk, the bulk resistivity is extracted from the slope of the differential resistance plotted as a function of gap width for several features at large voltage values where Andreev reflection doesn’t occur. This gives a linear relation as shown in Fig5.2. The resistivity of GaMnAs at 4.2K is extracted from the slope of the line, and found to be equal to 15.17 m\( \Omega \).cm. The bulk resistance of each gap was calculated, which is equal to \( \frac{\rho}{2\pi r} \), and its value was subtracted from the measured differential resistance. This gives the value of contact resistance without any contribution from the spreading resistance. Since both the bulk resistance and the contact resistance are in series, the voltage across the contact is easily extracted from measured voltage, thus eliminating the spreading resistance effect.

After eliminating the effect of broadening which comes from the bulk resistance, the contact conductance was drawn as function of contact voltage where the contact conductance is equal to \( G=\frac{1}{R} \), as shown in Fig.5.5. The flat part of the conductance starts to occur around .5mV which is less than the known energy gap value of Nb (1.4mV), as shown in Figs 5.5 B), 5.5C), and 5.5D). These figures show the conductance curves measured at 4.2K after eliminating the spreading resistance effect for three features with disc diameter 600\( \mu \)m, and a gap width of 15, 8, and 10 \( \mu \)m respectively. The decreasing of the superconductor energy gap at the interface is expected, since the Cooper pairs at the interface are weaker than inside the bulk. This reduction in the superconducting energy gap compared to the bulk Nb value comes from several reasons: The first one is the intermixing of ferromagnetic and superconductor atoms at the interface, which makes the Cooper pairs weaker than the Cooper pairs inside the bulk. The second reason
is the proximity effect, which rises from multiple Andreev reflections of particles at the interface. The conductance around zero voltage gets enhanced by (100, 40, 23, 72) % for the features of circle diameter of 600µm and with GaMnAs width gap 10, 15, 8 and 6 µm, respectively as shown in Figs 5.5A), 5.5B), 5.5C), and 5.5D). This indicates a very small spin polarization of the conduction electrons exiting GaMnAs into superconducting Nb. Our conductance curves do not show any small dip around the zero voltage. This shows that there is no tunneling barrier at the interface, whose effect is shown in Fig.5.6. The increase in the height of the barrier lowers the conductance around zero voltage and decreases the probability of AR around zero bias, in that model [Ref. 23]. The variation in the enhancement in the conductance curve can be explained due the spin flipping at the interface, which comes from the mismatch between velocities of Fermi level of GaMnAs and Nb, which can’t be eliminated, and the intermixing of between GaMnAs and Nb.23

![Graphs](image)

**Fig.5.5:** the conductance curve after eliminating the spreading resistance effect for as grown sample at 4.2K:A) (600-6) the conductance start to enhance around 1.5mV which is close enough for superconducting energy gap. B), C), and D) the differential resistance started to enhance around .5mV this because Cooper pairs are weaker at the interface than inside the bulk. The enhancement in the conductance indicates that the polarization is less than 50%.
Fig 5.6: Theoretical normalized conductance $G(V)/G_n$ versus $V$ curves at $T=1.5$ for different values of elastic scattering parameter $Z$. [from Ref. 23]

The conductance curves for the superconductor/ferromagnet interfaces produced by our technique are much closer to the theoretical model\textsuperscript{12} and need less fitting parameters to find the spin polarization. The conductance curves found by the PCAR technique do not fit the theoretical conductance curve of the mBTK model to get the spin polarization. PCAR approach suffers from the non-ideality of contact resistance due to many reasons such as uncontrolled oxides or chemical reactions at the interface between GaMnAs and the superconductor,\textsuperscript{5,31} the pressure from the superconducting tip to the sample, which change the electronic properties of the material.\textsuperscript{5} Also the measured resistance in this technique is not the contact resistance only, it includes an extra resistance from the sheet of ferromagnet\textsuperscript{9,31} and that resistance cannot be eliminated experimentally since the variation of spreading resistance, coming from the ferromagnet film, is not linear as a function of distance from the contact point as shown in Fig.5.7A).\textsuperscript{11} Multiple empirical fitting parameters have to be introduced in order to fit the experimental results, mainly due to this spreading resistance effect. The effect of the spreading resistance on the conductance curve is shown in Fig.5.7B).\textsuperscript{31} A second parameter usually introduced to fit the experimental results is the broadening effect arising from the inelastic and
elastic scattering parameters at the interface, which reduce the life time of quasi particles.\textsuperscript{5,27} As the quasi particle life time decreases, the broadening effect $\tau$ increases, which smears out the conductance curve. A third parameter used for fits is the effective temperature, which suggests that the broadening in the curve is due to local heating effects that increase the $\Delta$ value and explain the broadening in the curve, even though the measurements are done at a known constant temperature.\textsuperscript{5,31} Beside all these broadening parameters, there are factors which cause opposing effects on the conductance curves and increase the uncertainty in choosing the value of the parameters to fit the experimental conductance curves and find the spin polarization value. The mBTK model used three fitting parameters $Z$, $p$ and either $\Delta$ or $T$ to fit the experimental curves. But due to the non-ideality of PCAR technique, other parameters are added to the mBTK model such as the effective temperature, the quasi life time, the inelastic and elastic scattering parameters, and the spreading resistance parameter. All these factors reduce the uniqueness of the fit, besides the high uncertainty in determining the value of these factors. For example Fig5.8\textsuperscript{5} shows the effect of spreading resistance on the spin polarization values, where the spin polarization was extracted for several theoretical curves with same spin polarization value for the ferromagnet and the superconducting energy gap but with different spreading resistance values. The curve shows how the value of spin and energy gap vary as there’s spreading resistance,\textsuperscript{5} which the incorrect value of spreading resistance affect the value of $\Delta$ and the spin polarization where the plot shows 1% error in energy gap correspond to 1% error in spin polarization.

![Fig5.7](image.png)

Fig5.7: A) Schematics of a point contact and a current lead with radius of 30 nm and 127um and a separation of 2 mm on a thin-film plane, and resistance between A and a voltage lead along the dashed line.[from ref.11]. B) is Spreading resistance model with zero or slight interface scattering at 4.2 K. By varying the relative ratio $X$ of the spreading resistance to
the point contact resistance, the original features around the band gaps in the mBTK curve were smeared out and the spectra broadened. [from Ref. 31]

Fig. 5.8: Polarization versus superconducting energy gap for one point-contact spectrum of Sn/LSMO. The inset serves to illustrate that, in each extracted value of Pc obtained from the models, a quality fit was achieved. The limits for Rs in the datasets (inset) for values of Δ between 0.5–0.62 meV are approximately 11.8–9.3Ω, respectively. [ from Ref.5]

5.2 The effect of local annealing on the AR and the contact resistance (GaMnAs/Nb)

The effect of a short annealing on the AR probability is investigated by using hot soldering tip to form the four-probe connections. In order to study the short annealing effect on the contact resistance, we picked two features from the previous measurement (done with the cold pressing of the wires) and re-wired them using the hot tip. Both of them show AR around zero bias at 4.2K, but one of them has ohmic behavior, and the other a schottky behavior. The effect of hot soldering used to see if has any effect on the behavior of the contact and its value, and the probability of AR. In Fig.5.9A, and 5.9B) two features, the first feature is with circle diameter of 600um and gap width of 6um, its resistance is ohmic while the second sample is a feature of gap width of 40um its differential resistance show Schottky behavior.
Fig. 5.9: two features with circular diameter 600µm their differential resistance measured at 4.2K. A) With gap width 6µm it resistance is ohmic and have a dip around zero voltage due AR effect. B) With gap width of 40µm it’s contact resistance show a Schottky behavior at 4.2K also its showing a dip around zero voltage due AR effect. Cold tip used to fabricate the connections. C), and D) : The differential resistance for the same features at 4.2K, but here the hot soldering is used to fabricate the connections. C) the behavior of the differential resistance as function of voltage is showing a little Schottky barrier for feature with circle disc 600µm, and a gap width of 6µm, D) the Schottky barrier is reduced compare to cold tip measurement, there is an obvious AR at the interface for the feature with circle disc 600µm, and a gap width of 40µm.

The short annealing was done at temperature 560 °C for a few seconds for each connection. It affects the total resistance for both features as shown in Figs.5.9C), and 5.9D), where the resistance is decreased compared to its value when the cold tip is used. This decreasing might be due to interdiffusion between the Nb and GaMnAs films that increases the local carrier density and reduces the contact resistance.
After subtracting the contribution of the bulk, the contact conductance of both features was calculated and shown as a function of contact voltage in Fig.5.10. The value of the conductance increased as expected due to the short annealing. Both samples show the same behavior; their conductance curve started to enhance around 1.5mv which is close enough to the theoretical energy gap of Nb, the conductance is enhanced by 22%, which indicates that we have low spin polarization of GaMnAs.

![Fig. 5.10: the contact conductance for the two features in figure 5.10, after subtraction the spreading resistance is shown in A), and B) The differential for both features enhanced by 22%, the superconducting energy gap seems to be 1.5mV.](image)

5.3 GaMnAs/Nb (Tc=100K)

The AR effect is studied for another set of features that are patterned on a piece of the same GaMnAs wafer as before, but the GaMnAs was annealed at temperature 180 °C for 96 hours before depositing the Nb, which changes the Curie temperature from 65K to 100K. This helps us investigate the effect of changing the Curie temperature on the spin polarization of GaMnAs bulk, and to see if the AR at the interface is sensitive to the change in the spin polarization of the bulk or not.

The results of differential resistance for different features are shown in Fig.5.11. The resistance for most features shows ohmic behavior as in Figs.5.11A), 5.11B), and 5.12D), where it is decently horizontal (i.e. independent of voltage). The resistance also increases with gap width, but some features show a low Schottky behavior as shown in Fig5.11.C). The Schottky behavior
could be due to the formation of a thin oxide layer at the interface during the time the chamber need to pump down to reach $10^{-7}$ mbar. The total differential resistance is reduced compared to as grown features because the resistivity of GaMnAs decreases with increasing the Curie temperature, causing the total resistance to decrease.

![Graphs showing differential resistance for several features of GaMnAs with Tc=100k wafer, measured at room temperature. A), B), and D) have ohmic differential resistance, while C) has Schottky contact resistance.]

**Figure 5.11:** the differential resistance for several features for GaMnAs with Tc=100k wafer, the measurements done at room temperature. A), B), and D) their differential resistance is ohmic, while the contact resistance for C) is Schottky.

The resistivity of GaMnAs was calculated by drawing the resistance for several features as a function of gap width as shown in Fig 5.12, which will give a straight line. From its slope the resistivity of GaMnAs was calculated to be 6.39 mΩ.cm. This is significantly lower than the resistivity of the as-grown GaMnAs because the annealing forces the interstitial Mn ions to
transfer to the surface of the bulk, and increase the hole concentration inside the bulk which reduces the resistivity of GaMnAs.

Fig 5.12: the differential resistance for several features as function of gap width, for GaMnAs wafer with Curie temperature 100K, at room temperature.

Fig5.13 shows the behavior of resistance for several features at 4.2k. Their resistance is constant at large voltage, which indicates that their contact resistance at the interface is ohmic. The total resistance is reduced by a few ohms compared to room temperature, which indicates that the electrons cross the barrier at the interface by tunneling, since the resistance doesn’t increase by lowering the temperature. In Fig5.13A), 5.13B), 5.13C), and 5.13D) the resistance is constant at large voltage, and its value is linearly increased by increasing the gap width, which is consistent with CTLM. In Fig 5.13B) there is a big peaks around the dip, this due the spin flipping at the interface. Fig 5.14 is showing another set of measurements carried at 4.2K for the same sample but different features. The behavior of the resistance is similar to the previous measurements. It is constant for large voltages and has a dip around zero voltage. But in Fig5.14.B) the resistance shows a shallow Schottky behavior that might be due to the presence of a thin oxide layer at the
interface. In **Fig5.14A)**, and **Fig5.14.B)** the resistance is again enhanced around zero voltage which indicates there is a low tunneling barrier at the interface which suppresses the probability of AR effect. In **Fig5.14.D)** the conductance has another minor small dip around zero, this small minor dip indicates that there’s no elastic scattering at the interface.⁶

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**Fig5.13:** the differential resistance for several features at 4.2K, for GaMnAs Tc=100K, using the cold tip to fabricate the connections of four-probe technique. **A),B),C), and D)** are for the features (600-15), (600-20), (400-80), and (400-40). The dip around zero voltage is due AR effect.
Fig. 5.14: the differential resistance for several features at 4.2k, for GaMnAs Tc=100K, using the cold tip to fabricate the connections of four-probe technique. A), B), C), and D) are for the features (400-20), (400-15), (400-8), and (400-10). The dip around zero voltage is due AR effect.

Fig. 5.15 shows how the CTLM is used to find the contact resistance and to eliminate the spreading resistance effect. The resistivity of the bulk was calculated from the slope, then the
resistance of GaMnAs gap was subtracted from the measured resistance, and then the contact resistance was calculated as a function of its voltage. Finally the contact conductance was drawn as a function of contact voltage after eliminating the spreading resistance effect, as shown in Fig.5.17. Fig.5.17 shows an enhancement in the conductance with (20, 33, 20, 6, 6, 44) % for the features ((600-15), (600-20),(400-40), (400-80), (400-15), and (400-8)) respectively. The experimental superconducting gap is close to the theoretical one as shown in Figs 5.17.A), 5.17B), 5.17D), and 5.17F). While in Fig5.17C), 5.17E) the superconducting energy gap is almost 2mV and is larger than its theoretical value which shows that our results do not have 100% consistency. The conductance of feature (400-15) shows a decrease in the conductance around the zero voltage due decreasing the probability of AR to the presence of tunneling barrier at the interface.

![Graph](image.png)

Fig. 5.16: the differential resistance as function of width gap at 4.2K for GaMnAs sample with Curie temperature of 100K, the resistivity of the GaMnAs was extracted from the slope which is equal to 5.3mΩ.cm.
Fig. 5.17: the contact conductance for several features for GaMnAs with Curie temperature 100K, after eliminating the spreading resistance effect. A), B), C) and D) are for the features (400-40), (400-80), (400-15), and (400-10) respectively.
**Fig. 5.18** shows the effect of using a hot tip (a short annealing) to connect the samples. The resistance is reduced compared to that measured using the cold tip. The total differential resistance is clearly reduced due to annealing. After that the effect of spreading resistance is eliminated and the contact conductance curves are calculated as functions of voltage. Two of the features show 100% enhancement in the conductance curves which indicates that the surface of GaMnAs acts as a non-magnetic i.e. it’s a normal material, as shown in Figs 5.19B), and 5.19D) for the features (600-20), and (400-8) respectively. This is in contrast to the results with the cold tip, where when using the cold tip, the conductance of these two features increased only by 30%. Two of the curves in Fig.15.19A), and Fig.15.19C) show enhancement of 5% and 16% which indicate that the GaMnAs has a spin polarization around 30%. Their conductance was enhanced by 6% and 20% respectively when using the cold tip. So, the short annealing does not seem to have a large effect in the AR effect observed in these samples. The superconducting gap is around 0.7 mv for most features, which is smaller than the theoretical one due to the proximity effect.

![Fig5.18: the differential resistance for several circular features at 4.2k, using the hot soldering to fabricate the four-probe connections. The differential resistance for (400,40), (600-20), (400-80), and (400-8) are shown in A), B), C), and D).](image-url)
Fig. 5.19: the effect of short annealing on the contact conductance and AR at 4.2K. A), B), C), D) are the contact conductance for the same features in fig 5.18A), B), C), and D) respectively after eliminating the spreading resistance effect.
Chapter 6: Contact resistance and GaMnAs resistivity at different temperatures

The total resistance for each feature was studied as a function of voltage at different temperatures beginning from room temperature towards 10K. This is in order to make sure that our resistance is ohmic at low temperature and the dip around zero voltage in resistance curve is due to AR effect, when Nb is a superconductor. Also the contact resistance and the resistivity of GaMnAs were studied as a function of temperature.

Several micro-structures from the two sets of samples with Curie temperatures of 65K and 100K had their resistance studied as a function of temperature. The resistance of two of these features is shown in Fig.6.1; a feature with Tc=65K is shown in Fig6.1A), and a feature with Tc=100K is shown in Fig6.1B). Both features’ resistance behavior is identical to the typical behavior of GaMnAs resistance as a function of temperature, where it has a peak around the Curie temperature. This gives a sign that the most significant part of the resistance of each feature comes from the resistance of the GaMnAs gap.

Fig. 6.1: A) The voltage for an as-grown sample as a function of temperature. B) The voltage for a feature from GaMnAs wafer with Curie temperature 100K as function of temperature.
The temperature was stabilized, then the resistance of each feature was measured, as a function of voltage to check whether the behavior of the contact resistance is ohmic or not. All features show a linear ohmic behavior at all temperatures between (10K, 300K). **Fig6.2** Shows the ohmic behavior of the resistance of (600-6) feature at several temperatures where Nb is not superconducting and Andreev Reflection can’t occur. All features show a constant resistance that is independent of the current (i.e. the voltage) at all temperatures above the critical temperature of Nb. This is evidence that the dip around zero voltage at 4.2K in the resistance curve is due to AR.

![Graph showing ohmic behavior of resistance](image_url)

**Fig6.2**: the differential resistance for the feature with circular diameter 600µm, and gap width of 6µm, as function of current at different temperatures.

The resistivity of GaMnAs and the contact resistance was extracted by plotting the total measured resistance as a function of gap width. The resistivity and the contact resistance are then calculated from the slope and the intercept of the line, respectively, at each fixed temperature, as shown in **Fig6.3**. The calculated resistivity as function of temperature has a
peak around 65K As shown in Fig. 6.4 A), which indicates that the curie temperature is 65K. This is typical in ferromagnets to have a peak in their resistance at the Curie temperature due to correlated spin fluctuations in the paramagnetic and uncorrelated spin fluctuations in the ferromagnetic state.\textsuperscript{42} The peak for the annealed sample occurs at 100K, as shown in Fig6.4B. The resistivity of the as-grown sample at low temperatures is higher than resistivity at room temperature. Annealing forces interstitial Mn ions to move to the surface and reduces electron scattering at low temperatures. Removing Mn ions, which acts as double donors, from inside the bulk to the surface will increase the hole concentration inside the bulk and cause the resistivity of annealed samples to decrease. Fig. 6.4 shows that the resistivity behavior as a function of temperature for both samples and is showing that at each temperature the as grown resistivity is larger than the resistivity of annealed sample. Also due to annealing, more charge carriers will be at the interface and the depletion zone will be thinner, causing the contact resistance for the annealed sample to be less than the as grown ones. The contact resistance remains almost constant at different temperatures between (10K -295K), which indicates that the mechanism of electron transfer from Nb to GaMnAs is by tunneling, which is independent of temperature, due to the thin tunneling barrier at the interface that results from the high doping concentration of GaMnAs. Fig.6.5 Show the contact resistance between Nb and GaMnAs as function of temperature for both samples.
Fig. 6.3: the differential resistance as function of gap width at different temperatures.

Fig. 6.4: A) the resistivity of GaMnAs (as grown sample) as function of temperature. B) The resistivity of GaMnAs (TC=100K) as function of temperature.
Fig. 6.5: A) the contact resistance for as grown sample as function of temperature. B) The contact resistance for GaMnAs (Tc=100K) as function of temperature.

The effect of short annealing on the GaMnAs and the contact resistance of the interface has been studied too. The short annealing doesn’t affect the resistivity of GaMnAs. Fig. 6.6 A) shows that the resistivity at all temperatures is the same value whether the measurements were done after using a cold tip or a hot tip to wire the samples. On the other hand, the contact resistance at the interface has reduced after the short annealing, as shown in Fig. 6.6B). Short annealing probably caused intermixing between Nb and GaMnAs, which caused a change in the properties of the surface and the number of charge carriers.
Fig. 6.6: A) the effect of short annealing on the resistivity of GaMnAs at different temperature values. B) The effect of short annealing on the contact resistance between Nb and GaMnAs at different temperatures.
Chapter 7: Conclusions

We proposed a new geometry to study AR at the interface between resistive ferromagnets and superconductors. This geometry utilizes the circular transfer line method to eliminate the spreading resistance that comes from the resistive ferromagnet. Our new geometry overcomes some non-idealities with the planar and PCAR geometries, which only work for low-resistivity ferromagnets. In these two geometries, it has been assumed that the largest voltage drop occurs at the interface, and the contribution from the ferromagnet film resistance is small. This assumption doesn’t work for resistive materials such as GaMnAs, since the effect of spreading resistance causes conductance curve to broaden, and makes it misleading to extract the spin polarization accurately.

The fabrication of the interface between the ferromagnet and the superconductor is well controlled in the circular transfer line method, which enables us to prepare the interface in different ways, and study the effect of interface surface properties on AR probability.

In my thesis I criticized the results of PCAR technique by simulating the characteristic surface of the interface, by keeping the native oxide at the interface, where the surface of GaMnAs is open to the ambient, and has a native oxide layer on it. Our results showed that there is no AR detected and that the enhancement in the differential resistance curve is due to a Schottky barrier. The Schottky barrier reduces the probability of AR at the interface significantly. Our results indicate that what has been measured by PCAR is a schottky behavior not AR effect, which is proven by the mismatching between the experimental results and the mBTK model.

We studied AR at well-cleaned GaMnAs/Nb interface, where native oxide at the interface surface was etched using H$_2$SO$_4$. We have shown how we utilized from CTLM to eliminate the effect of spreading resistance. Our results show after eliminating the resistance of the bulk from the measured resistance, the spreading energy gap is smaller than the theoretical one. The reduction in the superconducting energy gap is expected, due to the intermixing between GaMnAs and Nb at the interface, which makes the Cooper Pairs weaker at the interface compared inside the bulk. Our results show an enhancement in the conductance curves, which
indicates our polarization, is low. The low polarization is due the treating the surface with H$_2$SO$_4$, which makes the surface heavily depleted with Mn ions. So the surface acts like normal metal/superconductor interface. Our previous research on GaMnAs/Cu showed suppression in magnetization near interface. We tried to prepare the surface with different way, by using HCL instead of H$_2$SO$_4$, but due to high humidity we were not able to get Nb superconductor. The enhancement in the conductance curve is not the same for all features, this is due to the intermixing of Nb and GaMnAs at the interface, which cause a large spin flipping at the interface. Also our resistance is in diffusive limit, where the Cooper pair breaks several times at the interface in its way to inside Nb bulk. These factors affect the conductance curve. To extract the actual value of spin polarization of GaMnAs, the previous effects should be included to mBTK model.
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