

INTERSHIP REPORT

FABRICATION AND MEASUREMENTS  
OF NIS JUNCTIONS TO  
CHARACTERIZE PLASMA ETCHING

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With the collaboration of

GRENOBLE INP PHELMA

AALTO SCHOOL OF SCIENCE



Within the PICO GROUP



Under Professor Jukka PEKOLA's supervision

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## The PICO Group

The PICO Group is a research team which work under the responsibility of the Aalto University School of Science. Its facilities lies in the Micronova building, situated on the Aalto Campus of Otaniemi, in Espoo. Otaniemi is the centre of all scientific activites of the Aalto University and the Micronova building holds the major part of research about nano and microphysics in Finland. The team consists of nineteen members for the moment, including, of course the professor Jukka Pekola, who leads the group and doctor Matthias Meschke who is in charge of the laboratory. Then, the team is divided between five post-doc, seven PhD students and five summer students.

The Group's researches are varied but especially lies on superconductors properties. The main topics are linked with quantum thermodynamics and single electron transport through nano and microscaled devices. Among the group there are theoretical physicists who focus on the theoretical aspect of research : statistics that rule heat transfert between two nanoscaled resistors[1], for example, whereas some other are focused on practical research : realization of NIS thermometers, electron counting through devices... Moreover, the Group work actively with the Centre for Metrology and Accreditation because one of their main goal is to redefine the ampere, the unit used to measure electrical current[2].

All theses topics of research can be reached thanks to the devices the Group and the building hold. The building owns a clean room, widely used by the members of the Group to make structures. The devices they use are mostly the Electron Beam Lithographier(EBL), evaporators LISA and MASA, and the Scanning Electron Microscope(SEM). The clean room also have devices for making semiconductors-based structures and the Atomic Layer Deposition device was invented here. Then, the Group have a dedicated room in which there are many machines and especially several dilution cryostats. Indeed, since most of the work done within the group has a link with superconductors, they need to reach low temperature to have access to this particular state of matter[3]. There are three regular dilution cryostats that can reach temperature as low as 20mK, and a BlueFors cryostats that works differently but can reach even lower temperatures. In other rooms, there are also devices to characterize and measure : probestation, Atomic Force Microscope (AFM), and SEM, bounders...

The University and these devices provide the Group the possibility to make research efficiently as we can see the numerous publications published every year in famous journals.

# Introduction

Nanostructures are more and more complicated. Researcher in this field always tend to try to make sophisticated devices with many functionalities. This is possible thanks to clean room devices that provide the researchers a way to make more and more different processes. Yet, some of functionnalities in the devices are not well characterized since researchers tend to focus on their actual structures rather than on the characterization of the devices, especially when devices are quite new. However, it is important to have access to all the possibilities that a device offers, this is why it is important to characterize them.

The evaporator LISA is quite a new device in Micronova's clean room, so that all its functionalities were not characterized, for example it allows plasma etching, which is an *in situ* etching that can be used to get rid of a layer of matter that is not wanted on the structure. But, in order to make this etching reliable, its effect on samples need to be characterized : how does samples react to etching, does etching damage the samples, is the plasma uniform ? All these questions that need answers in order to be able to use the plasma properly.

My role among the group is to make and measure simple structures : Normal Metal-Insulator-Superconductor junction (NIS junction) to determine characterize the plasma etching in an evaporator.

First of all, I will need to understand the theory behind such structures since it is not part of my course, with a literature study (Chapter 1).

Then, since I will fabricate some structures, I need to know how to do it. I will have trainings to use several devices in the clean room, but I need to understand how they really work, not just knowing on what button to push. Why do we use this device with this particular way to proceed, what are the parameters than have to be taken into account (Chapter 2).

Once I know how to the devices work, I need to set some parameters to make consistent measurements and characterize the other parameters with changing only one at a time : I need to build an experimental procedure. I also need to put in place a set up to make measurements : what type of measurements should be done, and how can we measure it (Chapter 3).

Then, the measurements will give me some datas, which I will need to exploit to turn them into results which I can interpret to understand what happens (Chapter 4).

# Chapter 1

## Theory

The first thing to do before starting anything is to understand the goal of the project. For that, it is important to study the behaviour of the structures we want to realize and the physics and quantum phenomenon that can occur. In this chapter we will sum up the litterature study that we have done to understand the theory behind the experiments.

### 1.1 Superconductivity

During the research project that Grenoble INP PHELMA gave me the opportunity to realize during the first year of my master degree, I have studied superconductors with my group which make this study a bit easier for me since I am familiar with some of the concept mentionned here. Let's start with the basics.

Superconductivity is a state of matter which occur mostly at low temperature for several materials. It is a state where the material have an absolute zero resistance, so that current can run without energy losses. It is also a state where the material totally excludes magnetic field and becomes perfectly diamagnetic[4]. These two major properties have a quantum explanation which is the Bardeen-Cooper-Schrieffer(BCS) Theory[5]. The qualitative aspect of this theory is quite simple whereas the quantitative involves the second quantization and advanced quantum theories.

The electron within the matter can pair in so-called Cooper pairs which come from an interaction between electrons and the ion lattice. At low temperature, electrons are slow and they tend to attract ions. These ions have a relaxation time to come back to their initial state, but during the time they are in a non-equilibrium state, they create a local positive charge that can attract another electron. This electron is then paired with the previous one. It has the same impulsion but an opposite spin according to the BCS Theory. The Cooper pairs, even if formed by two electrons are no longer fermions but bosons so that they follow the Bose-Einstein statistics and form a condensate within the matter.

Then, the electrons that are not coupled follow a different density of states where there are two pikes at the distance of  $\Delta$ , around the energy of the condensate  $E_F$  : This is the so-called superconductor gap.

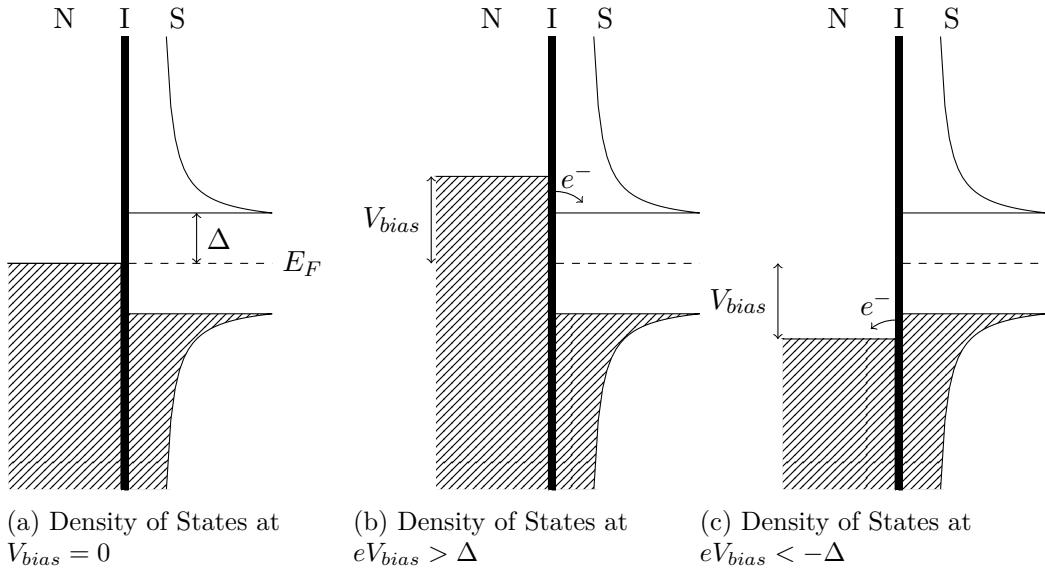


Figure 1.1: Density of states for different  $V_{bias}$  for a NIS junction

## 1.2 Normal Metal-Insulator-Superconductor Junction

In the case of a Normal Metal-Insulator-Superconductor (NIS) Junction, we make a contact between different density of electronic states, as shown in Fig. 1.1a. From this Figure, we can understand what happens with these junctions when we apply a voltage. Applying a voltage will translate these densities of states. While  $|eV| < \Delta$ , the electron cannot tunnel through the insulator, but as soon as  $eV = \Delta$ , they can start to tunnel in one direction or the other as shown in Fig. 1.1b and Fig.1.1c.

## 1.3 Leakage current

The previous explanation was of course the ideal one, at 0K, with a totally perfect sample and so on. In practice, we cannot work at absolute 0K, so thermal agitation will perturbe the whole system : the occupations of the densities of states is not ideal when the temperature goes up. So, there always have some electrons above the Fermi level for the normal metal and above the gap for the superconductor so that there can be some tunneling even when  $|eV| < \Delta$ . Moreover, the densities of states described before come from a calculus with approximations.

## 1.4 Dilution refrigerator

# Chapter 2

## Experimental methods

In order to make the NIS structures, we need to learn more about the different methods available in clean room. Then, we will be able to realize the structures which we be useful to characterize to help the process for the nanowires. I will describe here only the useful devices and methods for our process. In the last part, I talk about the dilution cryostat which I used to cool down some samples.

### 2.1 Resists

The resists are the starting point of the fabrication, without them we would not be able to design the pattern we want and without pattern, no structure.

#### 2.1.1 From theory...

The resists we use consists of polymer materials : Polymethyl Methacrilate (PMMA) and Methyl Methacrilate (MMA) (Fig. 2.1). PMMA is a polymer made out of MMA.

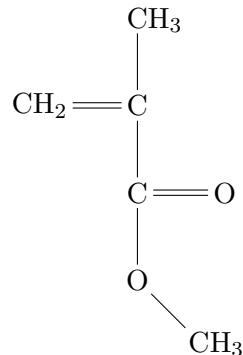


Figure 2.1: Chemical structure of MMA, PMMA is a polymer made of this monomer

The resists are liquid, we depose them on the top of the wafer and then a spinner rotate the wafer and make a thin and uniform layer of resist.

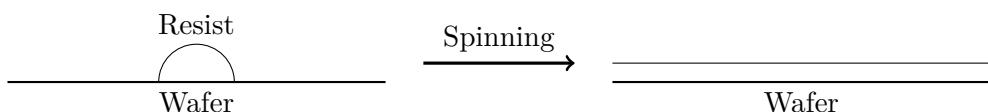


Figure 2.2: Spinner

The resists we use are sensitive to electrons, what we will do next is to send high energy electrons onto them (See 2.2.1) which will imply structural modifications. The electron break the polymer into smaller pieces, which make the exposed resist more soluble in Methyl IsoButyl Ketone (MIBK) (See 2.2.3).

Other type of resists exists, especially some resists damaged by light (photoresists), which are mostly used for semiconductors-based structures.

### 2.1.2 ...To practical

Practically, we use these two types of resists because they do not react the same way to exposure and development. First, we want a quite thick layer of MMA. Since the spinner create a fixed thickness, we repeat the proces until having the thickness we want, which means four times. Then we want a quite thin layer of PMMA, for this, only one spinning is good. When we have to bake the resists with a hot plate to make them solid instead of liquid. The resists have a data sheet where all necessary information is provided including optimal temperatures and baking durations. We finally obtain this (Fig. 2.3) kind of cross-section view.

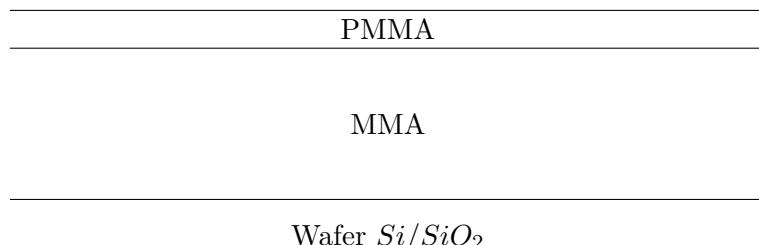


Figure 2.3: Cross-section view after resist deposit, spinning and baking

## 2.2 Electron Beam Lithography and development

Important part of the process, it determines either the structures will be as we expect or not, since it really impacts the resist.

### 2.2.1 Electron Beam Lithography

The Electron Beam Lithographier (EBL) is a device which is used to design the patterns we want to have for our structures within the resist[6]. It sends a electron beam onto the resist to damage the bonds within it. The functionnal diagramm (Fig. 2.4) shows the ways the electrons are focused int the EBL. This diagram reminds some optical systems, since the goal is to focus eletrons on relatively small areas. Indeed, the size of the structures is quite small and we want to be the most accurate possible. This is why it is possible to adjust the resolution on the EBL, the accuracy of the beam. The higher the resolution is, the more time it takes to expose the resist, so the best thing is to find a compromise between resolution and time. For example, the only part that requires an good accuracy is the junction, so we have to use a good resolution there, but for the leads it does not matters much, so we choose to go faster. Then, the other adjustable parameter is the exposure dose, which means the amount of electron we send through the resist. The higher the exposure dose is, the more it damages the resist. As for the resolution, we have to find a compromise between enough electrons to make sure the bonds are destroyed within the resist

and avoid completely burning it. The EBL is designed to send electron onto the resist, yet, these electrons have a too high energy to break the bonds of polymers. Actually, when they encounter matter, they generate other electrons from atoms, named secondary electrons. These are these electrons who have the right energy to break the bonds of polymers. The goal of the EBL is to generate the larger amount of secondary electrons possible and this is achieved by using high energy incident electrons that can penetrate deeply within the resist layer. The deeper they go, the more secondary electrons they generate and more bond are broken.

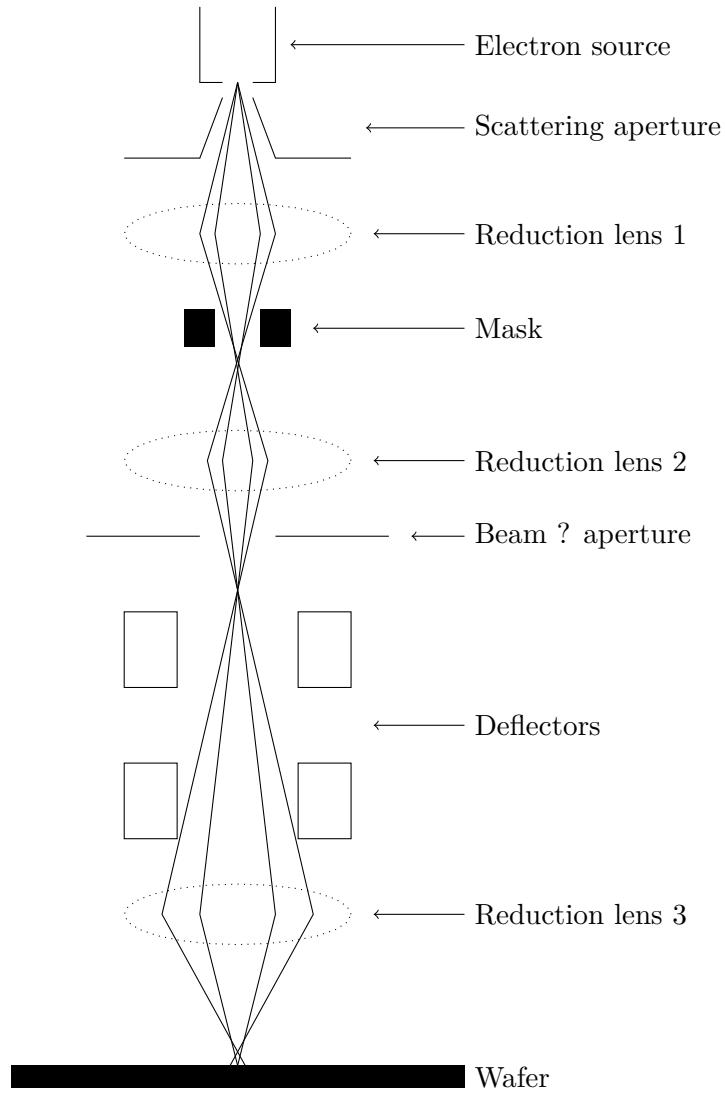


Figure 2.4: Simplified EBL functional diagram



Figure 2.5: Electron Beam Lithographier Vistec of Micronova clean room

### 2.2.2 Pattern design

The beam is controled by computers, we can create all the designs we want in a software and import them to make the EBL expose the resist. The beam breaks the bonds in the selected zones, as shown in Fig. 2.6.

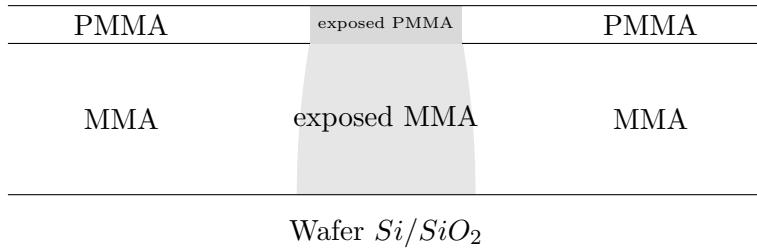


Figure 2.6: Cross-section view after EBL

### 2.2.3 Development

Development consists in the withdrawal of the exposed resist. It is realized by a succession of chemical reactions. First of all, we have to pierce the PMMA to access the MMA. It is the role of MIBK (Fig. 2.7) to dissolve the exposed PMMA and MMA. The MIBK pierces a strait hole in the resist (Fig. 2.8), but it will not be enough for the structures we want to do.

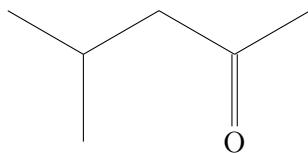


Figure 2.7: Chemical structure of MethylIsoButylKetone (MIBK)

Our goal is to make small junction between Al and Cu, if we depose Al or Cu on this wafer, they will depose everywhere and we will just have Aluminium covered by Copper, which is not what we want. To manage to make the structure, you have to find a way to not depose metal everywhere. This is the role of the undercuts ans evaporation angles (See 2.3.3). We add a step to the development : we dive the chip in MethyGlycol. MethyGlycol can dissolve MMA even if this one have not been

exposed to the electron beam, but do not damage PMMA. This allows us to create a dome, a larger empty area below the PMMA (Fig. 2.9) named undercut. There, we can see that if we deposit metal along a certain angle, they will not necessarily be in contact, this is what we are looking for. The final step of development is to stop the reaction. We dive the sample in Isopropanol which is neutral with our two resist and stops the reaction with MethylGlycol.

Of course, the reactions follow some kind of kinetics and the dissolution will depend on the time we dive the samples into the chemicals. Indeed, electron exposure only ease the dissolution of the resists, this means that we cannot just let the dissolution go and come back when it is over, we have to determine the accurate duration for our application. If it is less important for MIBK (because the difference between dissolution coefficients of exposed and non-exposed resist is large enough), it is very important for MethylGlycol. We have to make sure the undercuts are large enough for our structures but ensure that PMMA does not collapse.

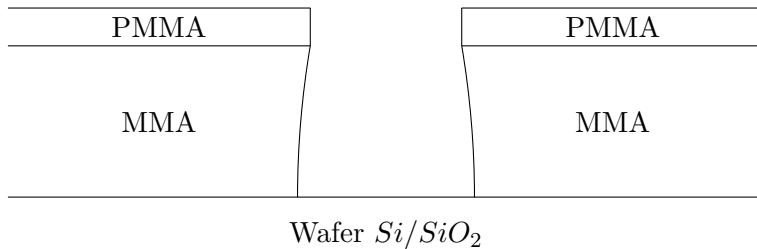


Figure 2.8: Cross-section view after MIBK

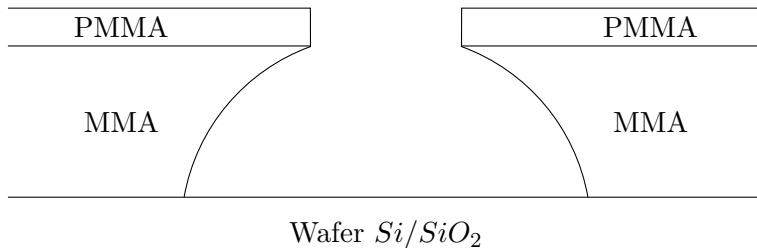


Figure 2.9: Cross-section after full development

## 2.3 Evaporator

The main part of the process, where we actually make the structures by evaporating metal.

### 2.3.1 Functioning of evaporator

The evaporator is a tool that allows to deposit a uniform, thin layer of metal inside the undercuts newly developed. A filament is submitted to a huge tension and current, it emits photons that will melt the metal and tear atoms from it. These atoms can move freely in the very low pressure chamber ( $P \sim 10^{-7} \text{ mbar}$ ) : the mean free path is long enough to allow atoms to go everywhere in the chamber especially in our undercuts. The deposition of the atoms is very uniform and there are sensors to measure the thickness of the layer. So, we can set the thickness we want and the

device will automatically stop when the thickness reaches this value. There is a valve for Oxygen, so that we can oxidize. It is particularly helpful to realize insulators *in situ*,  $\text{Al}_2\text{Ox}_3$  is an insulator and it creates an energy barrier, quantically speaking.

### 2.3.2 The Plasma gun

There is also a Plasma Gun, with Argon valve. The plasma is mostly used to ease the lift-off (See 2.4.1) by weakening the resist. But with the accurate parameters it can also tear atoms from samples, this is a technique called plasma etching, and we will use it quite a lot. One of the goals of this project is to characterize this technique in the evaporator here : characterize the plasma and determine its effect on samples (See 3). We need to know better about plasma etching because the nanowires will come from Copenhaguen and the Aluminium layer will be strongly oxidized, thing that we do not want. We will need to get rid of it one way or another, this is why we try plasma etching. We will realize different junctions with different plasma parameters to know better how it behaves.



Figure 2.10: Photographie de l'évaporateur LISA utilisé pour réaliser les structures

### 2.3.3 Evaporator in action

The Fig. 2.11 shows what happens in the evaporation chamber. First, we deposit Aluminium with a previously determined angle, then the evaporate Copper, with another angle. Thus, we can see that even if they are in the same undercut, they do not touch each other. To make the junction, we have to have two different undercut that overlap. Like this it is the metal from one undercut which will be in contact with the other metal for the other undercut so that they are in contact in a tiny area and not in the whole pattern. Of course, we can add other steps to this process like oxidation and plasma.

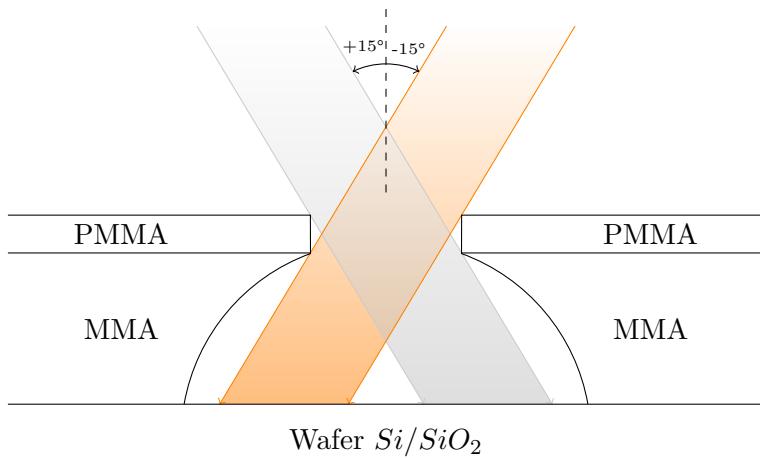


Figure 2.11: Cross-section during evaporation

## 2.4 Lift-off and Scanning Electron Microscope

This is the final part of the process where we complete the structures and check if everything was fine.

### 2.4.1 Lift-off : resist withdrawal

Metals evaporated everywhere on the chip but, they mainly are upon the resist. The structures are protected inside the undercuts. So, we will just dissolve the resist with acetone. It will take the metal off the wafer and let only the structures.

### 2.4.2 Scanning Electron Microscope functioning

The functioning of Scanning Electron Microscope (SEM) is very similar to the EBL one, since actually, SEM can be used as EBL, except that this time we do not want to weaken the matter but to observe the scattering of electrons within it. Each material does not scatter electron the same way as its neighbour, particularly if they are very different electronically, such as metal and silicon for example. We can detect the electrons and observe contrast discrepancies between metal and silicon and determine if the sample seems good or not. Moreover, the SEM has a "secondary electron" mode that can detect the secondary electrons emitted by the matter while the primary electrons hit. There, each material reacts differently, so we can see a contrast difference between Aluminium and Copper, but the brightness is very weak on this mode since secondary electrons are very outnumbered by primary electrons. We can use the first mode to check that the metals evaporate well, but if we see some strange shapes, we can switch mode to see if the shapes are still here. If they are, it means it is metal, which can be problematic, but if they disappear, it might just be some resist remains or defaults within the wafer.



Figure 2.12: Scanning Electron Microscope used to observe the structures

### 2.4.3 Observation of the samples

The SEM requires some adjustments in order to give us good images. The settings look very like optical settings : focus, stigmatism, aperture... To set them, we first choose a place where there is no samples, in order to avoid to charge them since we send electron within the matter. Of course, if there is nothing at all, it will be very difficult to set anything, so we choose a place with metal but which does not belong to a structure. Once the location chosen, we have to set the different parameters to get the best image possible. We align both focus and stigmation together by adjusting, and zooming when we have the optimal settings. The more we zoom in, the more tricky it becomes to get a good image. Then, while we consider that the image is good, which means precise enough and clear, we can start to observe the samples (Fig. 2.13). We still have to pay attention not to charge them, so we try to observe fastly to avoid any problem. We do not need to observe each sample, we just take some pictures to check if the sample seems good in average. It is quite easy to detect if there has been a huge problem but the images only give an indication about the samples. Some samples seems bad and while we measure them, there are totally accurate whereas sometimes they seem quite good but the measurements show odd results.

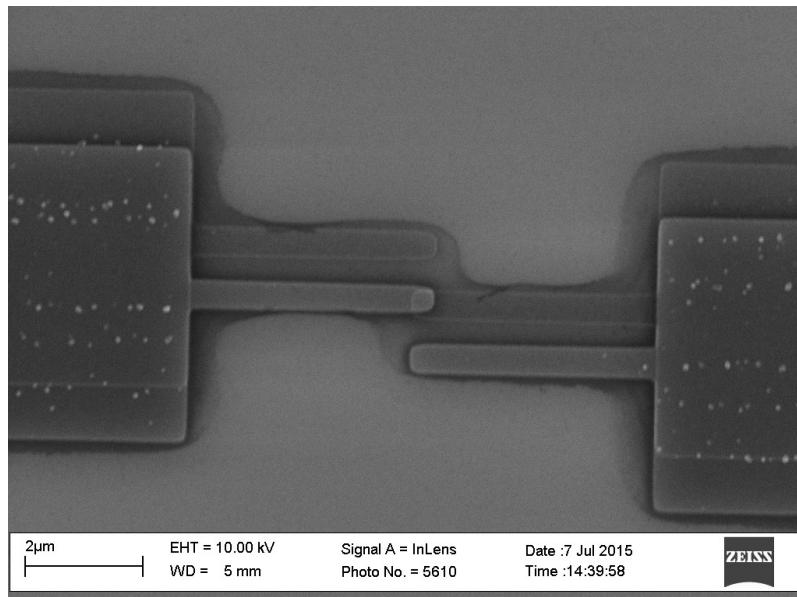


Figure 2.13: SEM image of a sample

## 2.5 Dilution cryostat

The cryostat is not a clean room device, though it is very useful for a low temperature laboratory. Since, I used it to cool down some samples we can consider it is part of the process to get some results and it is definitely an experimental device. First, I will talk about some theory to set the fridge up, then I will talk about the cooling down procedure we follow to reach 50mK.

### 2.5.1 Theory about Helium

### 2.5.2 Preparation of the cooling

The first thing to do with low temperature measurements is to prepare the sample to get in the fridge. There are sample stage that we use to make measurements. They consist in metallics pads linked to 12 pins where we can bound the pads of our samples. There is a bounder which allows us to link our sample to the pads by bounding a thin Al wire between them (Fig. ??). Once the bounds are done, we can check if the sample is alive with a multimeter. If everything is allright, we can screw the sample stage at the right place on the fridge (Fig. ??). Then we put the vacuum chamber (IVC) in place, with a indium wire and hexagonal screws to seal it (Fig. ??).

### 2.5.3 Cooling down

The cooling down process in itself is quite long even if there is not much to do but waiting most of the time. The first thing to do, once the sample stage is in place is to pump the IVC and the 1K pot (a small tank which be be thermalized at 1K with pumping over a Helium bath). Then we flush the 1K pot with Helium, to make sure there is no more air inside it. Once the pressure in the IVC is low, we can add some helium which will be the exchange gas. Then, we can put the IVC in liquid nitrogen for 30 minutes to start the cool down (Fig. ??). Then, we rapidly switch

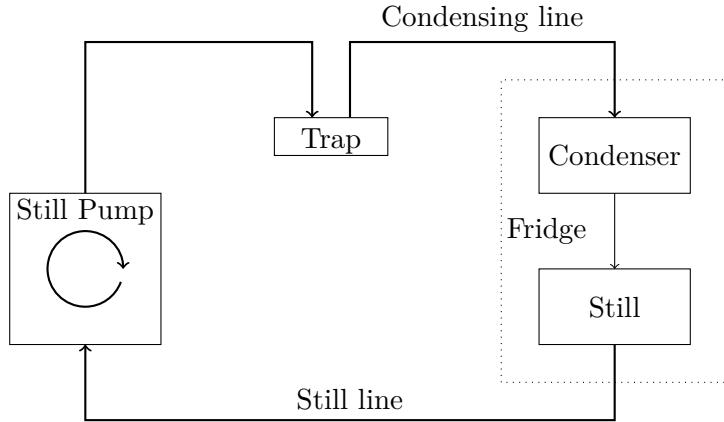


Figure 2.14: Circulation diagram

the nitrogen tank to put the fridge in the helium dewar after having depressurized it before. We set up the condensing and the still lines and pump them to avoid air, because air it would freeze. We also pump the trap, which is a part used to clean gases by nitrogen and carbon, with helium several times to remove air from it. Then, we just have to pump IVC and wait that the fridge thermalize at 4K, which is quite long (Fig. ??). Finally, we have to pump the IVC for a long time. We take advantage of this to also pump the condensing and still lines.

After having waited enough, we stop pumping the IVC and we will start to condense. The pressure in the IVC is very low, we stop pumping it and remove the line. We open the 1K pot valves to put some Helium in and pump over it to cool it down to 1K. Since Helium is superfluid at this temperature, we would notice it there was any leak, because it could go everywhere. We put the trap in liquid nitrogen, so that we can flush the condensing and the still lines with mixture. The mixture goes from the pump to the lines through the trap and back to the pump, so it cleans the gas inside the lines : if there was still some air for example. We flush three times to divide the amount of impurities by  $10^8$  ( $10^2$  each time). Once the lines are clean, we start to send the mixture to the cryostat through the still line. The still pump contains some mixture, but the tank contains a much larger amount, so we have to open the tank valves to take the mixture from here too. Then we wait until the tank is empty. Once it is empty, and that the temperature is low, we can start to circulate : we close the tank valve and create a loop for the mixture (See Fig.2.14), so that it is always moving and cools down the fridge.

#### 2.5.4 Warming up

Warming up the fridge is quite easy, first we have to stop the circulation of the mixture by closing the trap in and start to pump it from both still and condensing lines to the tank, so we open the tank and still valves. The mixture is liquid so it is difficult to pump it in this state. To ease the pumping, we will heat up to evaporate the mixture. For this, there are two lines where we can apply some voltage, which will go through thermoresistor and heat the area and then the mixture. Since the mixture is pump with the still pump, we have to be careful that the pressure does not exceed 1 mbar, or it could damage the pump. To avoid huge augmentation of the pressure we increase slowly the applied voltage, especially around 1.3K when the mixture starts to boil. Of course, we can control the temperature through Matlab,

like we did for the cool down. The pressure increases while the mixture is evaporated. It can take one hour until the mixture is totally pumped out.

Once it is the case, the temperature increase rapidly and the pressure goes down. Here, we wait five minutes to be sure all the mixture went out. Then, we close all the valves of the condensing line and the still line, we keep pumping the mixture to the tank, finally, we close the tank and remove the fridge from the dewar. Here, the fridge just needs to thermalize at the room temperature, we let it like that for a while before removing the sample and getting the fridge ready for the next cooling.

# Chapter 3

## Experimental protocol

In this part I will focus on the relevant tests made, results of the first tests will be found in Appendix A.1.

### 3.1 Parameters

The following table describes the different devices used for the realization of the samples and shows the parameters that have to be taken into account during the fabrication. The parameters in bold are the most influent in the results we will obtain, so they are important and the settings needs to be determined precisely.

| STEP                | DEVICE        | PARAMETERS                           |
|---------------------|---------------|--------------------------------------|
| Resist deposition   | Spinner       | Rotation Speed                       |
| Resist baking       | Hot plate     | Temperature                          |
| Pattern design      | EBL           | <b>Dose, Shape(area), Resolution</b> |
| Development         | MIBK, MG, IPA | <b>Duration</b>                      |
| Deposition of metal | Evaporator    | <b>Angle</b>                         |
| Oxidation           | Evaporator    | <b>Pressure, Duration</b>            |
| Plasma Etching      | Plasma gun    | <b>Duration, Position</b>            |
| Lift-off            | Aceton        | $\emptyset$                          |

The chip we realize consists in twenty samples, with four different surface areas ( $0.5, 1, 1.5 \& 2 \mu m^2$ ) and five different electron doses (from 2000 to 3000 by  $250 e/\mu m^2$ ) in the EBL. This gives us some statistics : we do not stick to one result but we have several ones to make sure the datas we get are relevant and not due to any problem. Moreover, some little troubles can always occur on one of the samples (EBL default, speck of dust...) and fabricating 20 of them avoids to loose a whole attempt. Mathieu already defined some settings such as the development time and he made the patterns and the EBL writing.

### 3.2 Experimental procedure

In order to have coherent results I have always followed the same process to realize the samples. Based on 4 layers of MMA and one layer of PMMA and EBL dose from 2000 to  $3000 \mu m^2$ , according to the pattern in Fig. 3.1, I develop 20s in MIBK, 20s in MG and IPA. Then I evaporate 20nm of Al. Depending on the sample I want

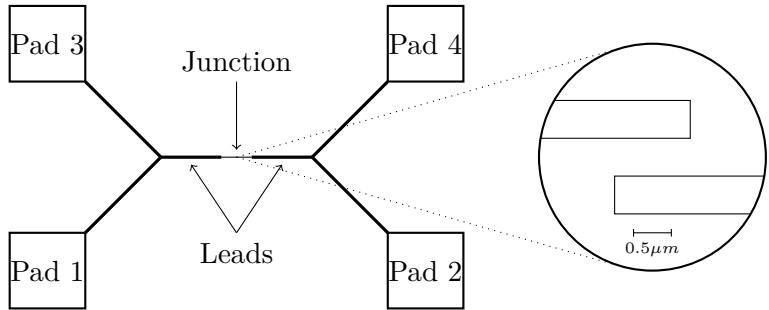


Figure 3.1: Pattern of the junctions

to make, I can strongly oxidize with a pressure of 200mbar of  $O_2$  during 10 min. Then, I can use the plasma to etch, the parameters are a pressure of  $4.10^{-4} mbar$  of Argon, a power of 40mA, an extraction voltage of -0.8kV (lowered down to -250V in July, due to problems that high voltage caused) and a Ion Energy of 1.5kV. I can also oxidize with a pressure of 2mbar during 2min. Finally, I evaporate 25nm of Cu before letting the sample in lift-off for more than an hour..

I have realized samples with strong oxidation and plasma etching to see how much oxide was etched, then I have realized samples with strong oxidation, plasma etching and regular oxidation and regular oxidation reference samples in order to cool them down to see the differences and so the effect of the plasma on the quality of the NIS junction. You can find an exhaustive table of the parameters used for each test in Appendix C

### 3.3 Problems with the devices

When I was here, there have been some problems with the evaporator LISA which I used to make the structures. The plasma gun seemed to have a problem in the begining of July, since it didn't work properly. We could see that nothing happened when we set the power current. It was cleaned and repaired and the main user advised to limit the Extraction to -250V to avoid future breakdown. After this, the plasma seemed to act differently, it seemed more powerful according to the results (See 4). Then, just before I left, everyone seemed to have troubles with the junctions made in the evaporator. I tried to

### 3.4 Measurement setup

#### 3.4.1 Room temperature setup

We first used a method to measure the samples but it was not rigorous enough, I talk about it in Appendix A.2.

Let's focus on the good measurements. I have made four-probe resistance measurements on the samples I have fabricated with a probe station. The four-probe measurements make sense as it is the only way to measure the real resistance of the device, without parasite resistances. The probestation make a slope of voltage from -100 mV to 100 mV, measure the voltage and the current between two electrodes and trace an I-V curve. It export the data obtained in .DAT files with the current and voltage tables. I have then realized a Matlab program to exploit them efficiently and be able to trace charts showing the more parameter dependances possible.

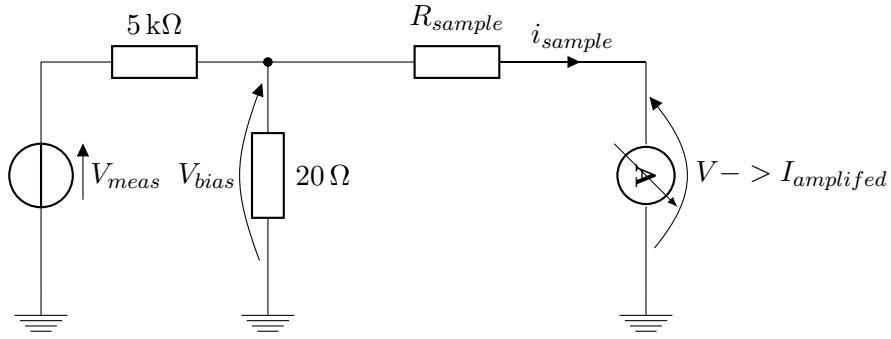


Figure 3.2: Schematics of the set up for low temperature measurements

### 3.4.2 Low temperature setup

Since the samples I make are superconductors-based structures, it is worth measuring them at low temperature to have the superconductivity state of Al. The critical temperature of Al is 1.2K, so we have to cool the sample lower than this temperature to obtain results. We use a dilution cryostat as the ones described earlier. Once the sample is cold, we can start the measurements.

There are devices that can be connected to the cryostat lines which are lined to the samples. Everything is controlled through Matlab programs. I have adapted an existing program to make it fit better with the measurements I wanted to do and the way I wanted to exploit the datas. First, I have to set the parameters, then the program is linked with the devices, it controls the generator to make a voltage slope and in the meanwhile it asks the multiplexer to get the bias voltage and the voltage which comes from the current amplifier (See Fig.3.2). These are rough values that are not the real ones. I can draw IV curves to check that everything is correct : good  $V_{bias}$  range, accurate current amplification... Then the program saves the datas that I will exploit with a better program later. The program I wrote allows to draw the IV curves, and determine easily the resistance and the leakage of the junctions.

# Chapter 4

## Results of the measurements and interpretations

In this part, I will present the results of the measurements I have made, both room temperature and low temperature measurements, and I will get the informations that we can get from these results.

### 4.1 Room Temperature Results

#### 4.1.1 Reference Samples

Before starting to use the Plasma, I need to have some references to compare to the results with Plasma. I have made several references samples : a clean contact, a strong oxidation and a regular oxidation.

I first made a clean contact sample, with only Al and Cu as a reference (See Fig.4.1). Since the contact is clean, the resistance of the junction between the two metals is close to zero, then the resistance we measure belongs to the leads which are following the following law :

$$R = \sum_{Al,Cu} \frac{\rho L}{S}$$

Since all the leads are the same, the resistance does not depends on the surface area of the junction, as you can see in Fig. 4.2 the results of four probes measurements. The theoretical calculus from this law with the parameters used gives  $78\Omega$ , which is close to the value we find. Of course, there are some uncertainties : the thickness sensor in the evaporator is not very accurate, since we evaporate with an angle, the dimension are a bit different on the actual junctions, from the pattern dimension. The point is that the order of magnitude is correct, so our reference for the leads resistance will be around  $70\Omega$ .

After the clean contact, I made some reference samples for a strong oxidation, I oxidized freshly evaporated Al under a pressure of 200mbar during 10 minutes before evaporating Cu. The SEM images can be seen on Figure 4.3.

For the NIS junctions, it is more relevant to draw conductance in function of the surface area of the junction so that we obtain a linear curve (See Fig. 4.4), and we can determine the RS factor.

$$RS = 7.18k\Omega.\mu m^2$$

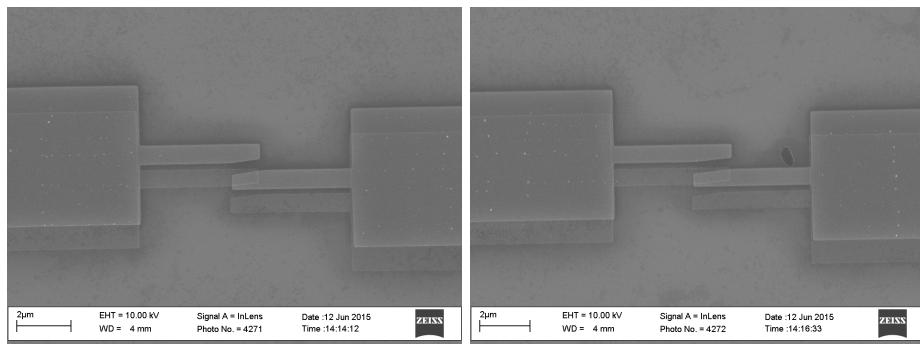


Figure 4.1: SEM images of the clean contact samples

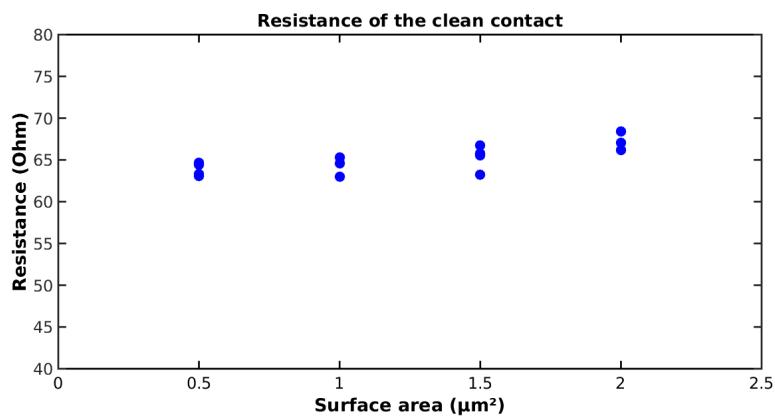


Figure 4.2: Resistance of clean contact in function of the surface area

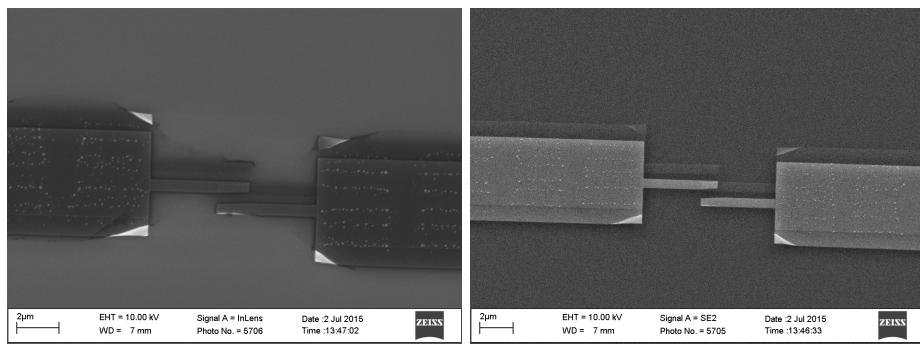


Figure 4.3: SEM images of the Strong Oxidation samples

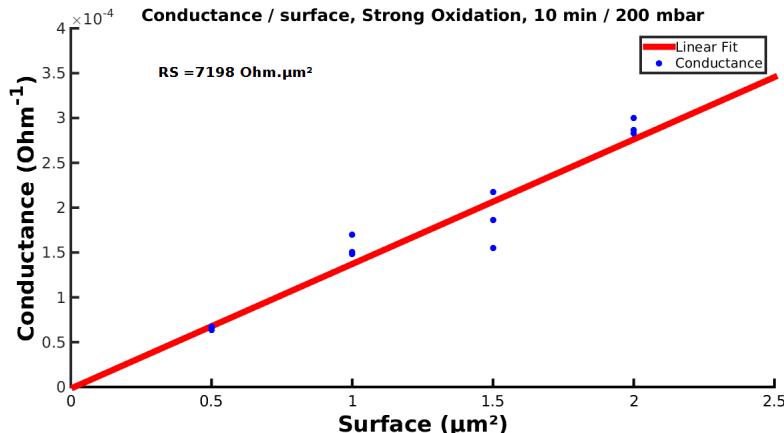


Figure 4.4: Conductance in function of surface area for a strong oxidized sample

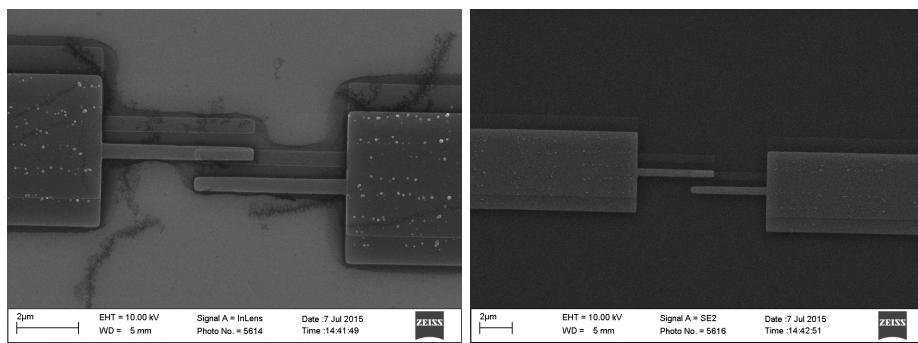


Figure 4.5: SEM images of the Regular Oxidation samples

This factor is relevant because :

$$R = \frac{\rho l}{S} \implies l = \frac{RS}{\rho}$$

In the case of the oxide,  $l$  is the thickness of the oxide layer, so that  $RS$  is proportional to the thickness.

In order to cover a large range of resistance, I have also made a regular oxidation reference sample, with the evaporation of Al placed under 2mbar for 2 minutes before evaporating Cu.

Again, it is more relevant to draw the conductance (See Fig. 4.6) to determine the  $RS$  factor.

$$RS = 7\Omega.\mu\text{m}^2$$

$RS$  is ten times less important than for the strong oxidation which means that the layer of oxide is ten times thinner. These three references give us a base to evaluate the quantity of oxide left when we will etch it. For example, if we find a resistance around  $1k\Omega$ , we can assume that the thickness of oxide is close to the thickness of the regular oxidation.

#### 4.1.2 Plasma Etching : Position of the sample

Then, I wanted to check if the plasma is uniform. We do not know if it affects equally all the areas of the sample stage or not. This is why I've made several tests where

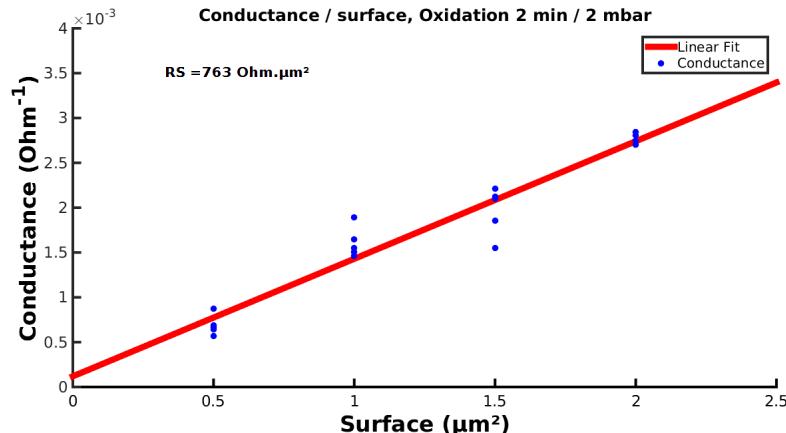


Figure 4.6: Conductance in function of surface area for a regular oxidized sample

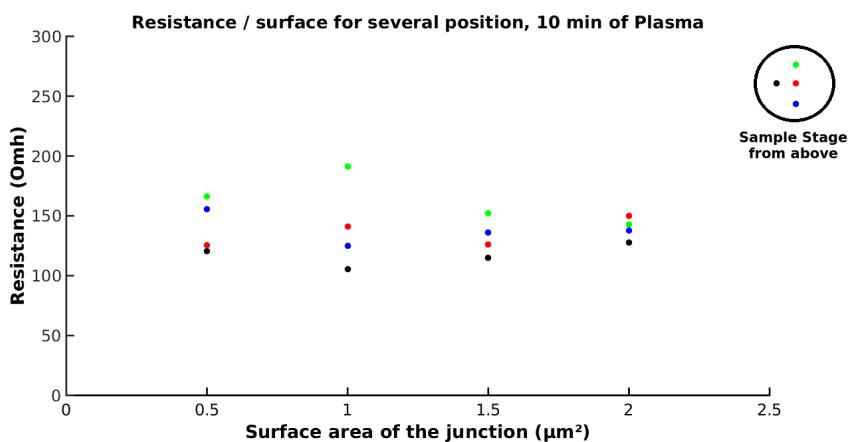


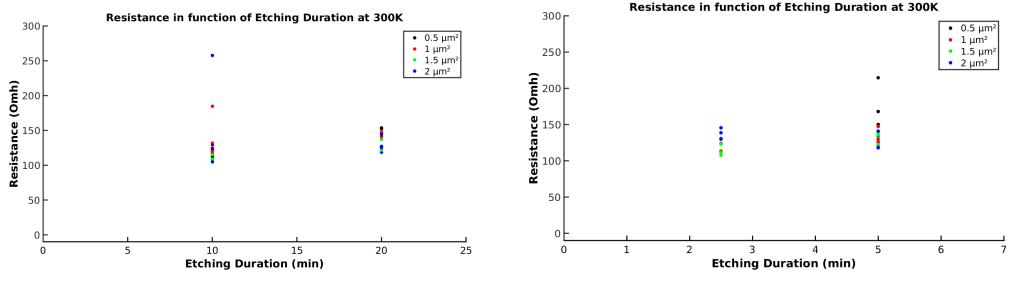
Figure 4.7: Resistance in function of surface for different positions of the samples on the LISA's sample stage

I placed 4 samples on the sample stage and did the evaporation. The Figure 4.7 shows the results of these tests. We can see that the position does not affect much the resistance of the sample so we can assume that the etching is quite uniform. And another conclusion of this graph is that 10 min of plasma are enough to etch all the Al oxide : there is no surface dependance like for the oxidized samples and the resistance is about the same order of magnitude than the clean contact junction.

#### 4.1.3 Time of plasma

The time during which the sample is exposed to the plasma is important and determine how much oxide will be etched and implicitly the resistance of the sample. I followed the same procedure all along and only changed the etching duration. In Figure ?? there are some good samples made with plasma etching. Some samples also turned out to become failures, there are SEM images with explanation in Appendix B.1.

The Figure 4.8a shows the resistance compared to plasma etching duration before the cleaning of the plasma gun. We can see that there is not a lot of differences between 10 and 20 minutes but the more relevant result is what we can see on the



(a) Resistance in function of Plasma Etching duration before the cleaning      (b) Resistance in function of Plasma Etching duration after the cleaning

Figure 4.8b, with samples made after the cleaning, it seems that less than 5 minutes is enough to etch all the oxide, since the results are very similar, yet for totally different duration times. It means that the cleaning had an effect on the plasma settings.

## 4.2 Low temperature measurements

### 4.2.1 NIS Junction

Thanks to the dilution cryostat I was able to cool down some samples down to 50mK. Of course the pads on the sample stage are in a limited number so that I had to choose the best samples to bond and to cool down.

### 4.2.2 Plasma etched samples

## 4.3 Summary of the results

# Conclusion

# **Appendix A**

## **First measurement method**

### **A.1 Bad pattern**

We first used a two pad pattern, which is not very practical to make 4 probe measurements, since I either had to put two needles of the probestation on one pad or bound samples stage pad to one another. We finished the samples that were already written with EBL and the we switched to 4 pads patterns.

### **A.2 Bad way of measuring**

At the beginning, with the 2 pads samples and with the first 4 pads samples, I bounded the samples to a sample stage to measure them with a complicated Matlab assisted setup.

# **Appendix B**

## **Failed Samples**

### **B.1 Failure due to plasma**

The plasma sometimes had some troubles and did not work properly. In the report there were the good samples but the plasma often burned the resist so that the copper evaporation could not be done properly.

### **B.2 Failure due to EBL**

The first EBL writing we made with the 4 pad pattern failed, there was a height problem. The wafer was not complete so it could not be clamped properly with the regular clamps, we had to have another one, which we forgot. The writing was really not usable as we saw with the first evaporation. Of course, we made another one to check that the problem really came from the EBL writing and not from the LISA.

## Appendix C

# Parameters table of the samples

| TEST N°   | STRONG OXIDATION | PLASMA    | REGULAR OXIDATION | COMMENT                    |
|-----------|------------------|-----------|-------------------|----------------------------|
| Test 10   |                  |           |                   | Failed EBL                 |
| Test 11   |                  |           |                   | Failed EBL                 |
| Test 12   | No               | No        | No                | Clean contact reference    |
| Test 13.i | Yes              | 10 min    | No                | Several positions          |
| Test 14.i | Yes              | 20 min    | No                | Several positions          |
| Test 15   | Yes              | No        | No                | Strong Oxidation reference |
| Test 16   | No               | No        | Yes               | Reference Sample Test 17   |
| Test 17   | Yes              | 10 min    | Yes               | Resist burned              |
| Test 18   | Yes              | 10 min    | Yes               | Resist burned              |
| Test 19   | No               | No        | Yes               | Reference Sample Test 18   |
| Test 20   | Yes              | 10 min    | Yes               | Resist burned              |
| Test 21   | No               | No        | Yes               | Reference Sample Test 20   |
| Test 22   | Yes              | 5 min     | No                |                            |
| Test 23   | Yes              | 2 min 30s | No                |                            |
| Test 24   | Yes              | 10 min    | Yes               | Etched the wafer           |
| Test 25   | No               | No        | Yes               | Reference Sample Test 24   |
| Test 26   | Yes              | 2 min     | Yes               |                            |
| Test 27   | No               | No        | Yes               | Reference sample Test 26   |

Parameters :

- Strong Oxidation = 10 min under a pressure of 200 mbar of O<sub>2</sub>
- Regular Oxidation = 2 min under a pressure of 2 mbar of O<sub>2</sub>
- Plasma = Pressure of Ar of 4.10<sup>-4</sup> mbar, Power=40mA, Extraction=-0.8kV<sup>1</sup>, Ion Energy=1.5kV

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<sup>1</sup>Lowered to -0.25kV starting from Test 22

# **Appendix D**

## **Compléments de résultats**

### **D.1 Images SEM**

### **D.2 Graphes supplémentaires**

# Bibliography

- [1] D. Gobulev and J.P. Pekola. Statistics of heat exchange between two resistors. 2015.
- [2] J.P. Pekola, O.P. Saira, V.F. Maisi, A. Kemppinen, and M. Möttönen. Single-electron current sources: Toward a refined definition of the ampere. *Review of Modern Physics* 85, 2013.
- [3] F. Giazotto, Tero T. Heikkilä, A. Luukanen, A. M. Savin, and J. P. Pekola. Opportunities for mesoscopics in thermometry and refrigeration: Physics and applications. *Rev. Mod. Phys.*, 2006.
- [4] M. Tinkham. *Introduction to Superconductivity*. McGraw Hill, 1996.
- [5] J. BardeenN, L. N. Coopeer, and J. R. Schieffer. Theory of Superconductivity. *Phys. Rev.* 108-5, 1957.
- [6] M.A. Mohammad, M. Muhammad, S.K. Dew, and M. Stepanova. Fundamentals of Electron Beam Exposure and Development. *Springer*, 2012.