

INTERSHIP REPORT

CHARACTERIZATION OF
INAS NANOWIRES
BY ELECTRON PUMPING

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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[2], 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[5].

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. 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

Nanowire... Quite a famous word within the research field nowadays. Yet, nanowires are still misunderstood. A nanowire is a nano or microscaled structure and shaped as a cylinder and at least a prisme. The properties that come from this particular shape and size mostly find their origins in the quantum theory and this is the main reason why these devices interests so much. They allow to understand better some aspects of the quantum theory that are still misunderstood and maybe they can lead to use some quantum properties in practical applications. Research about nanowire will eventually find practical applications in years or decades (according to past the observation of semiconductors theory for example), but to achieve that, it needs to make progress. This is why we propose to study the properties of induced superconductivity within a semiconductor (InAs) nanowire.

Peter Krogstrup, a researcher in the University of Copenhaguen put in place a process to growth InAs nanowires epitaxially to Al[3]. The InAs is a semiconductor whereas Aluminium is superconductor at low temperatures, induced superconductivity will appear within the nanowire who will become superconductor. It is this phenomenon we would like to characterize but to achieve that, we will need to study and understand the theories behind it (Chapter 1).

The goal we aim is to integrate the nanowire in a Normal metal-Insulator-Superconductor-Insulator-Normal metal (NISIN) junction to study the Andreev bound States that appear in the insulator barrier. The Copenhaguen team made structures with the nanowires but, we saw that there were troubles with the gates. We would like to integrate the structures here, and for this we need what techniques are available in the clean room to make structures (Chapter 2).

Once we know what we can do, we need to define some parameters and attribute values to them. This is the role of the several tests we have made : understand the effect of each parameter on the structure. For this, we did not use the nanowires but some very simple structures were we can easily understand what happen if we see any problem (Chapter 3) Moreover, these tests will teach us to deal with the clean room devices and get used to use them.

Then, we can start to try to integrate the nanowires to a viable structure. According to the tests, the process seems accurate but there will be some adjustments to do, obviously since the conditions are different(Chapter 4).

Finally, the project does not stop here, there is still a lot to do. First of all, it is important to confront the results with the theory : it is possible to go further and open the project to other perspectives. For example the study of Majorana's fermions within the Semiconductor-Superconductor interface. Research never stops, there is and will always a further point to reach, that what makes it so beautiful (Chapter 5).

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 phenomemon 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. These two major properties have a quantum explanation which is the Bardeen-Cooper-Schrieffer(BCS) Theory. Even of this theory does not make unanimity among physicist, since it does not explain everything, it is the most famous at the moment. The qualitative aspect of this theory is quite simple whereas the quantitative involves the second quantization and advanced quantum theories. The eletron within the matter can pair in so-called Cooper pairs which come from an interaction between eletrons an the ion lattice. At low temperature, electron 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 an 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 implusion 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 are 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 Δ , around the energy of the condensate E_0 : This is the so-called superconductor gap. Theoretically, the pikes are infinite for a temperature of 0K.

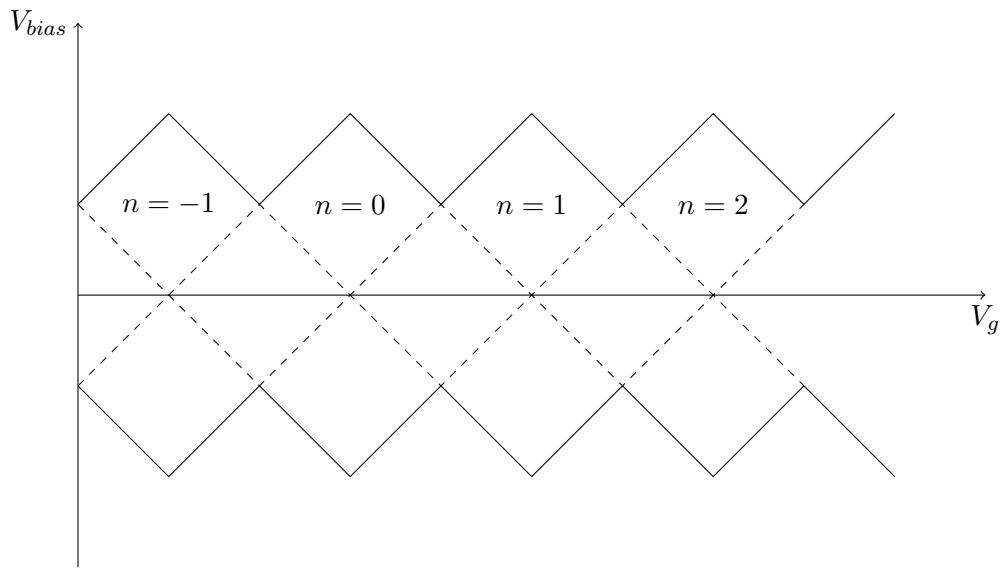


Figure 1.1: Blocage de coulomb dans un supraconducteur et principe du pompage d'électrons

1.2 NIS Junctions

[1]

1.3 Dilution cryostat

Chapter 2

Clean Room methods

In order to make the structure with the nanowires, we need to learn more about the different methods available in clean room. Moreover, we will make tests before starting with the nanowires that will help us to master the devices. I will describe here only the useful devices and methods for our process.

2.1 Resist

The resists are the central 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) (Fig. 2.1) and Methyl Methacrilate (MMA).

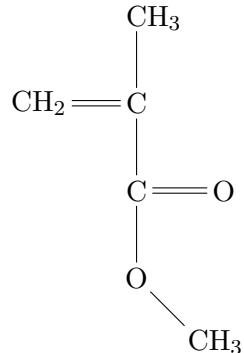


Figure 2.1: Chemical structure of MMA, PMMA is a polymer made of this monomer

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.

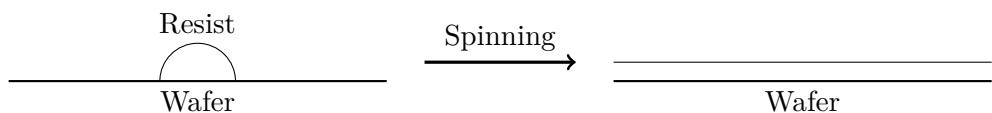


Figure 2.2: Spinner

The resists we use are sensitive to electrons, what we will do next is to send high energy electron 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 this 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.

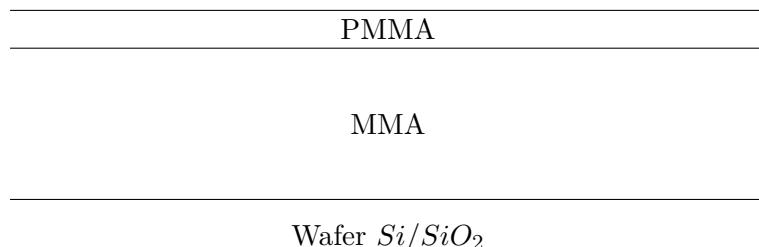


Figure 2.3: Cross-section view after resist deposit, spinning and baking

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 impact 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[4]. 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.

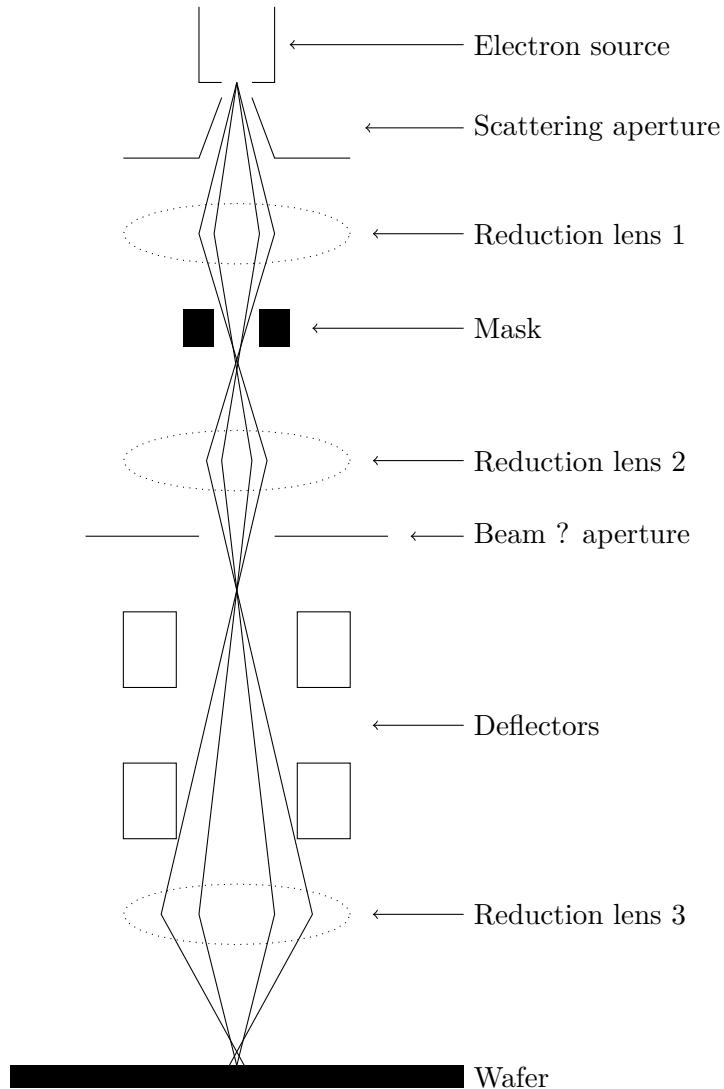


Figure 2.4: Simplified EBL functional diagram



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 show the Fig. 2.6.

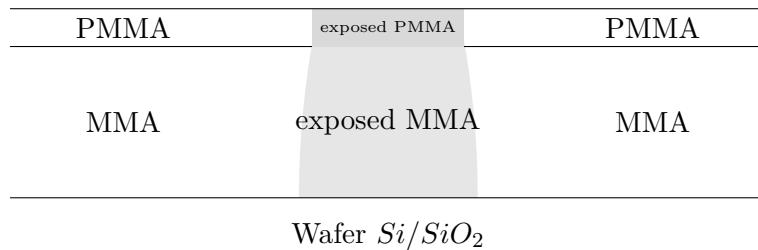


Figure 2.6: Cross-section view after EBL

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. Our goal is to make small junction between Al and Cu, if we deposite Al or Cu on this wafer,

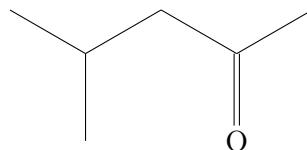


Figure 2.7: Chemical structure of MethylIsoButylKetone (MIBK)

Our goal is to make small junction between Al and Cu, if we deposite Al or Cu on this wafer, they will deposite 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 deposite metal everywhere. This is the role of the undercuts ans evaporation angles (See 2.3.2). We add a step to the development : we dive the chip

in MethylGlycol. MethylGlycol 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.

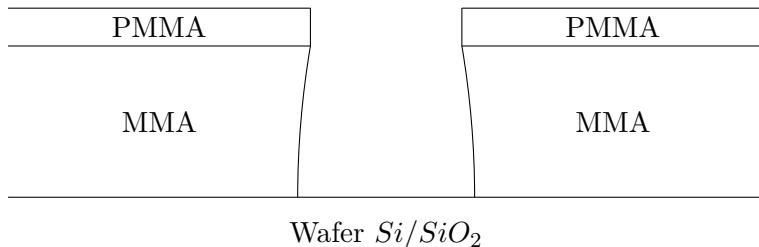


Figure 2.8: Cross-section view after MIBK

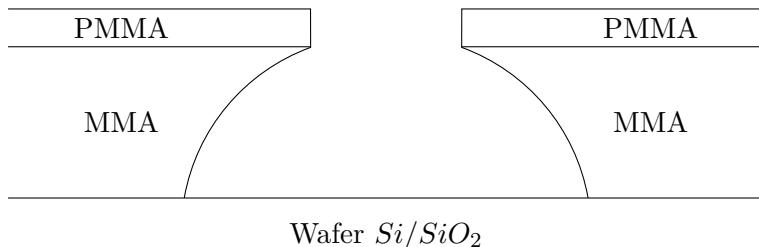


Figure 2.9: Cross-section after full development

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*, Al_2O_3 is an insulator and it creates an energy barrier, quantitatively speaking. 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. The second goal 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.



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

2.3.2 Evaporator in action

The Fig. 2.11 shows what happens in the evaporation chamber. First, we deposit Aluminium with a previously determined angle, then we deposit 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.

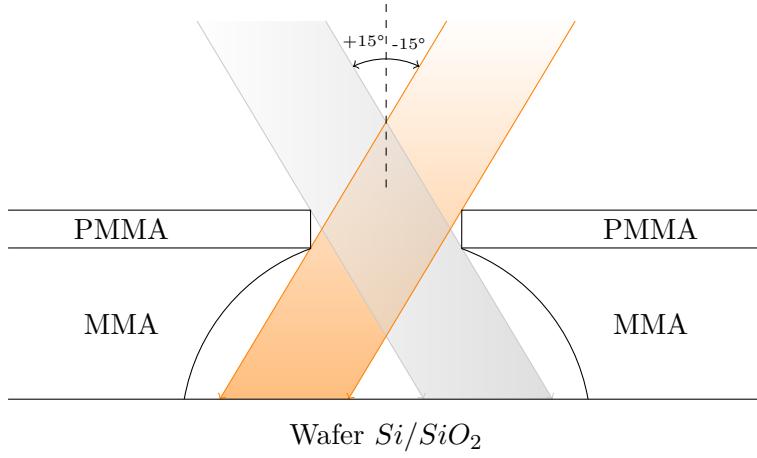


Figure 2.11: Cross-section during evaporation

2.4 Lift-off and Scanning Electron Microscope

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 aceton. 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.

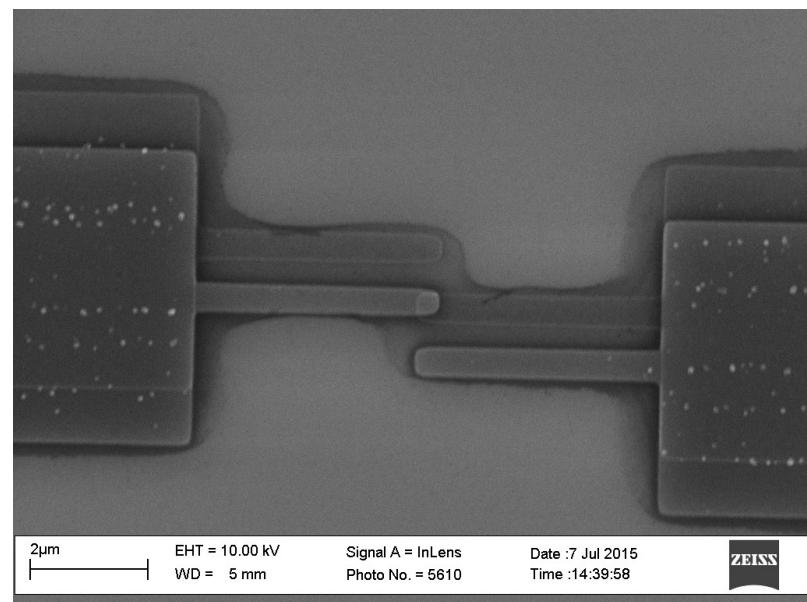


Figure 2.13: SEM image of a sample

Chapter 3

Realization of tests

The process we want to put in place is quite heavy, this is why we have to determine some parameters even before starting to think about the nanowires. In this part I will focus on the relevant tests made, results of other tests will be found in Appendix B.1.

3.1 Fabrication process and influent parameters

3.1.1 Fabrication process

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

3.1.2 Influent parameters

In the table above, you can see the influent parameters in bold : these are the parameters that have the biggest impact on the samples and the on we will focus on for the tests.

The chip we realized 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 $250 c/\mu m^2$) in the EBL. This gives us some statistics : we do not stick to one results but we have several one to make sure the value measures is not due to any problem.

Another goal of these tests was to characterize the plasma in the evaporator. This is why we made tests with the plasma with different settings.

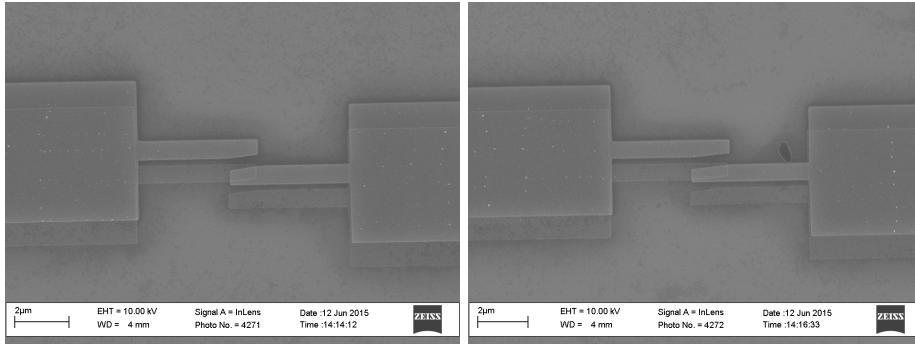


Figure 3.1: SEM images of the clean contact samples

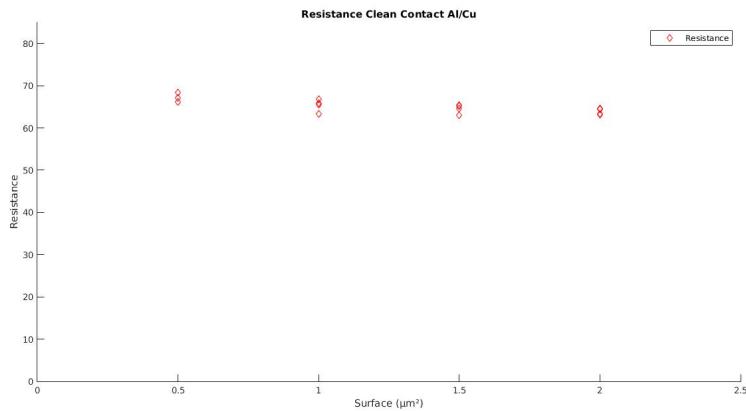


Figure 3.2: Resistance of clean contact in function of the surface area

3.2 Measurement setup

We first used a method to measures the samples but it was not rigorous enough, I talk about it in Appendix B.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 interferences. The probestation make a slope of voltage and trace an I-V curve. The data are .DAT files with the current and voltage tables. I have realized a Matlab program to exploit them efficiently and be able to trace many charts showing the more parameter dependances possible.

3.3 Room Temperature Results

3.3.1 Clean Contact

We first made a clean contact sample, with only Al and Cu as a reference. Since the contact is clean, the resistance of the junction is close to zero, then the resistance we measure belongs to the leads.

The Figure 3.2 shows the resistances obtained with four probes measurements with a clean contact. These results seems relevant according to previous measurements done.

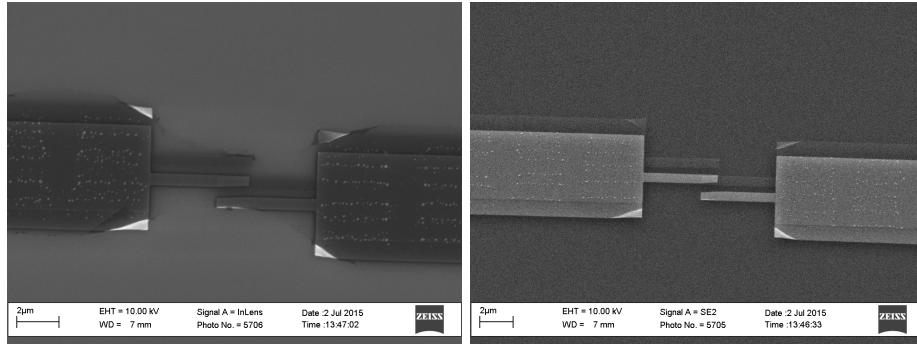


Figure 3.3: SEM images of the Strong Oxidation samples

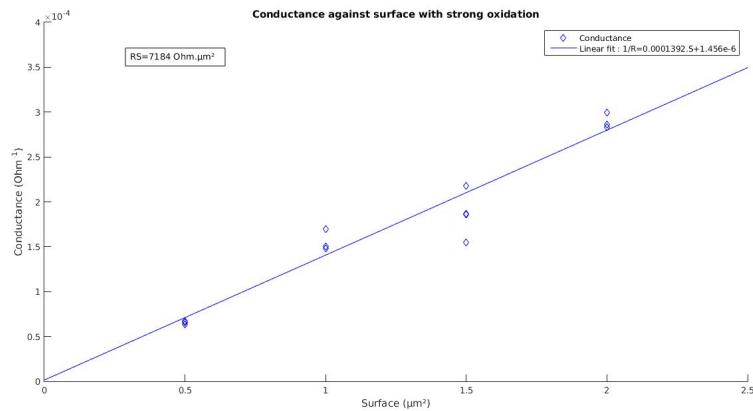


Figure 3.4: Conductance in function of surface area for a strong oxidized sample

3.3.2 Strong Oxidation

After the clean contact, I realized 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 3.3.

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. 3.4), and we can determine the RS factor.

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

3.4 Regular Oxidation

Then I have realized some samples with a regular oxidation to compare to the samples with the plasma. Here, I oxidize Al under 2mbar for 2 minutes before evaporating Cu.

Again, it is more relevant to draw the conductance (See Fig. 3.6) to determine the RS factor.

$$RS = 1.64k\Omega.\mu\text{m}^2$$

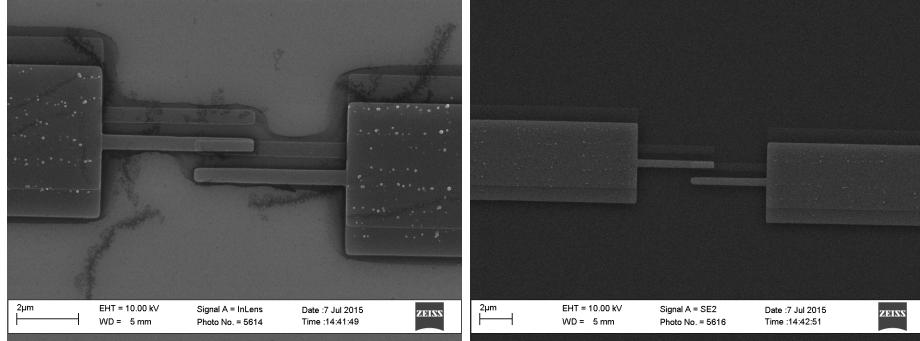


Figure 3.5: SEM images of the Regular Oxidation samples

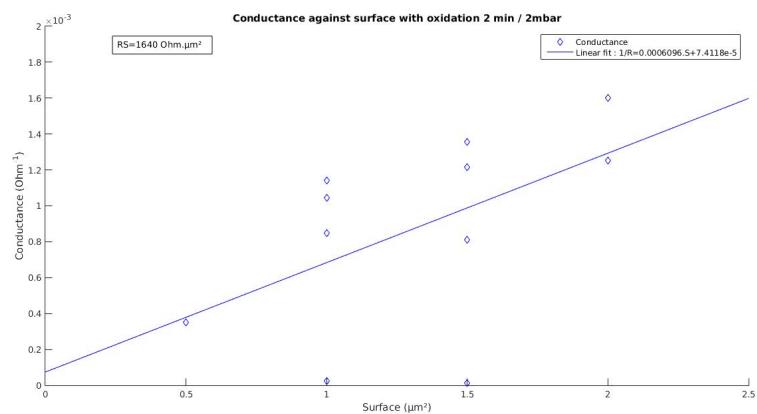


Figure 3.6: Conductance in function of surface area for a regular oxidized sample

These junction are less resistive than the strong oxidation junction, which is normal because the thickness of the oxide is lower, so more electrons can go through the oxide.

3.4.1 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. 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 C.1.

3.5 Summary of the results

Chapter 4

InAs Nanowires with InGaAs barrier

With all the tests realized, we know better about the clean room process and the plasma etching, so we can start the real process and measurements.

4.1 Clean room process

The main question we have to ask is, starting from the methods seen in Chapter 2, how can we integrate the nanowires to realize a real measureable structure.

4.1.1 Observation and EBL

First of all, we receive the nanowires as they were made which means half-covered by Al and randomly dispatched upon the wafer's surface (Fig. 4.1). The goal is to realize NISIN structures with Cu leads as normal metal, InGaAs barriers as insulators and the nanowire as superconductor thanks to Al and induced superconductivity. First, we will try to realize only NIS, by covering one of the barriers with Al, to see if the process can work out. The first thing we have to do is to observe the chip to find some wire that seems good enough to be exploited, get their coordinates on the surface and design a pattern to draw with the EBL.

4.1.2 Aluminium Etching

Then, we do not want to have Aluminium over the whole nanowire, so we have to etch it which we will do chemically.

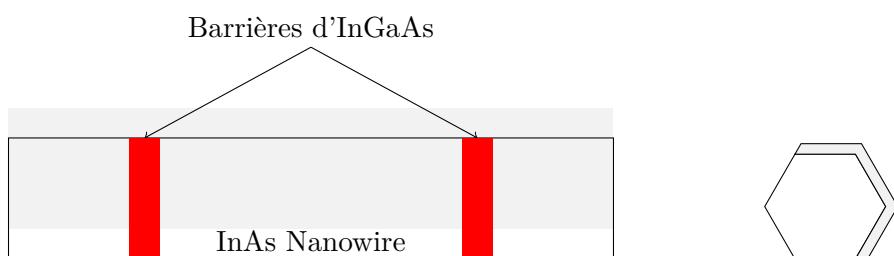


Figure 4.1: Nanowires as received from Copenhaguen

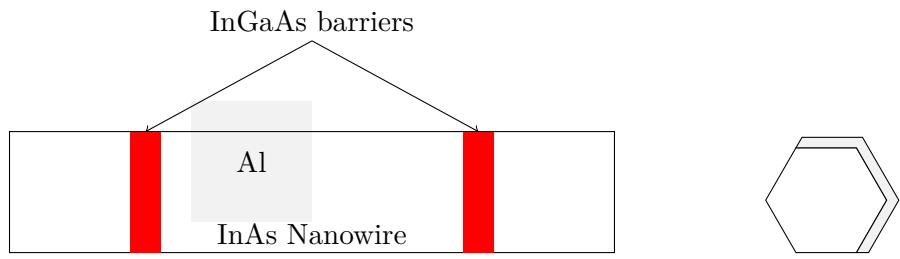


Figure 4.2: Nanowires after Al etching

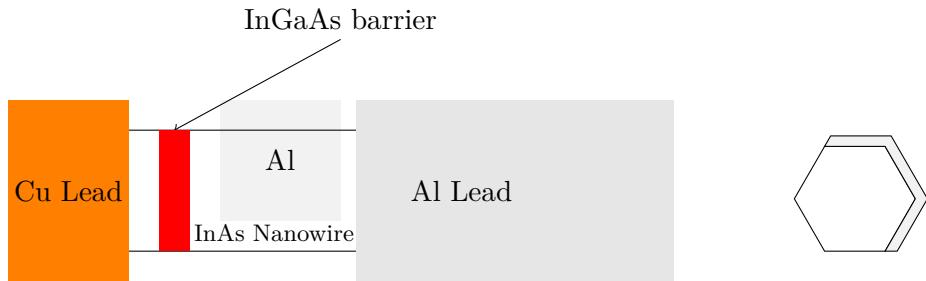


Figure 4.3: Nanowires after evaporation

4.1.3 Leads evaporation

Finally, we will evaporate the leads on the nanowire, with the evaporator.

4.2 Caractérisation

4.3 Interpretaisons

Chapter 5

Bilan

Le projet ne s'arrete pas avec mon départ et il reste encore beaucoup à faire.

5.1 Autres tests à réaliser

5.2 Etude théorique poussée

5.3 Ouverture vers d'autres structures

Conclusion

Appendix A

Structures de Copenhague

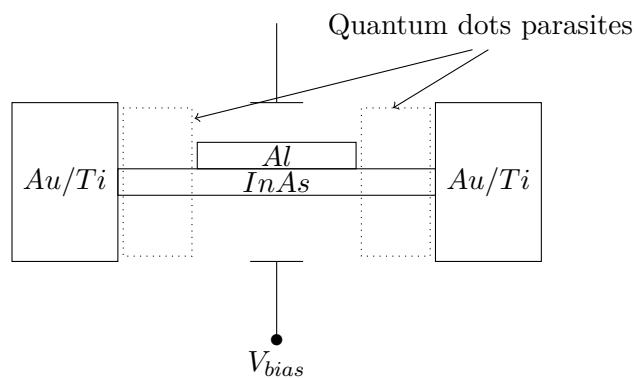


Figure A.1: Schéma en coupe représentant la structure réalisée à Copenhague

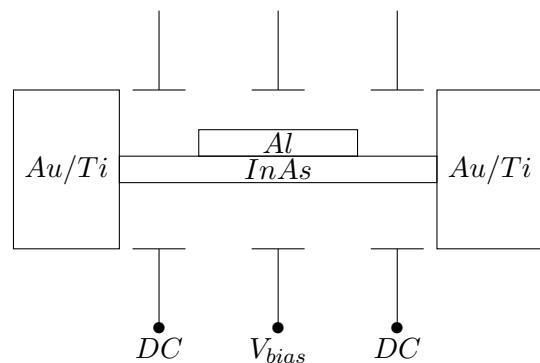


Figure A.2: Schéma en coupe représentant la structure réalisée à Copenhague après ajout de capacités de polarisation de quantum dots

Appendix B

Wrong measurement method

B.1 Bad pattern

B.2 Bad way of measuring

Appendix C

Failed Samples

C.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.

C.2 Failure due to EBL

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